Final

In-Situ Remediation Pilot Study Report
Area of Concern I (AOC I)

Atlantic Fleet Weapons Training Area – Vieques
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico

Contract Task Order 083
October 2013

Prepared for
Department of the Navy
Naval Facilities Engineering Command
Atlantic

Under the
NAVFAC CLEAN Program 1000
Contract No. N62470-08-D-1000

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Virginia Beach, Virginia
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<th>Description</th>
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<tbody>
<tr>
<td>µg/L</td>
<td>micrograms per liter</td>
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<tr>
<td>AOC</td>
<td>area of concern</td>
</tr>
<tr>
<td>AST</td>
<td>aboveground storage tank</td>
</tr>
<tr>
<td>bgS</td>
<td>below ground surface</td>
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<tr>
<td>CERCLA</td>
<td>Comprehensive Environmental Response, Compensation, and Liability Act</td>
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<td>CLEAN</td>
<td>Comprehensive Long-term Environmental Action–Navy</td>
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<tr>
<td>COC</td>
<td>constituent of concern</td>
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<tr>
<td>DO</td>
<td>dissolved oxygen</td>
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<tr>
<td>DOI</td>
<td>Department of Interior</td>
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<tr>
<td>EBS</td>
<td>Environmental Baseline Study</td>
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<tr>
<td>EISB</td>
<td>enhanced in-situ Bioremediation</td>
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<tr>
<td>ELCR</td>
<td>excess lifetime cancer risk</td>
</tr>
<tr>
<td>EQB</td>
<td>Environmental Quality Board</td>
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<tr>
<td>ERA</td>
<td>Ecological Risk Assessment</td>
</tr>
<tr>
<td>ERP</td>
<td>Environmental Restoration Program</td>
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<tr>
<td>FFA</td>
<td>Federal Facilities Agreement</td>
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<tr>
<td>ft/day</td>
<td>feet per day</td>
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<tr>
<td>ft/ft</td>
<td>feet per foot</td>
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<tr>
<td>gpm</td>
<td>gallons per minute</td>
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<tr>
<td>HHRA</td>
<td>Human Health Risk Assessment</td>
</tr>
<tr>
<td>HI</td>
<td>hazard index</td>
</tr>
<tr>
<td>IR</td>
<td>Installation Restoration</td>
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<tr>
<td>ISCO</td>
<td>in-situ chemical oxidation</td>
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<tr>
<td>mg/L</td>
<td>milligram per liter</td>
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<tr>
<td>MOV</td>
<td>Municipality of Vieques</td>
</tr>
<tr>
<td>mV</td>
<td>millivolt</td>
</tr>
<tr>
<td>NASD</td>
<td>Naval Ammunition Support Detachment</td>
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<td>NAVFAC</td>
<td>Naval Facilities Engineering Command</td>
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<tr>
<td>Navy</td>
<td>Department of the Navy</td>
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<tr>
<td>ORC</td>
<td>oxygen releasing compound</td>
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<tr>
<td>ORP</td>
<td>oxygen reduction potential</td>
</tr>
<tr>
<td>PA</td>
<td>Preliminary Assessment</td>
</tr>
<tr>
<td>PRG</td>
<td>preliminary remediation goal</td>
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<tr>
<td>RI</td>
<td>Remedial Investigation</td>
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<tr>
<td>SAP</td>
<td>Sampling and Analysis Plan</td>
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<tr>
<td>SI</td>
<td>Site Inspection</td>
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<tr>
<td>USEPA</td>
<td>United States Environmental Protection Agency</td>
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<tr>
<td>USFWS</td>
<td>United States Fish and Wildlife Service</td>
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<tr>
<td>VOC</td>
<td>volatile organic compound</td>
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</table>
This Pilot Study Report summarizes the activities performed and data obtained during the In-situ Chemical Oxidation (ISCO) and Enhanced In-situ Bioremediation (EISB) Pilot Study conducted at Area of Concern (AOC) I, located at the Former Naval Ammunition Support Detachment (NASD), Vieques, Puerto Rico (Figures 1, 2, and 3). AOC I is approximately 1 acre in size and was a former asphalt plant that operated from the 1960s through 1988. The Municipality of Vieques (MOV) owns the land within which AOC I is located.

This report is prepared under the United States Department of the Navy (Navy), Naval Facilities Engineering Command, Atlantic Division, Comprehensive Long-term Environmental Action–Navy (CLEAN) Contract N62470-08-D-1000, Contract Task Order 083, for submittal to the Naval Facilities Engineering Command (NAVFAC) Atlantic Division, United States Environmental Protection Agency (USEPA) Region 2, and the Commonwealth of Puerto Rico Environmental Quality Board (EQB). The Navy, USEPA, and EQB, together with the United States Fish and Wildlife Service (USFWS) for land owned by the Department of Interior (DOI), work jointly as the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Environmental Restoration Program (ERP) Technical Subcommittee.

A Remedial Investigation (RI) conducted at AOC I identified six constituents of concern (COCs) within groundwater: benzene, bis(2-ethylhexyl)phthalate, 1,2-dichloroethane, 1,2-dichloropropane, 2-methyl-naphthalene, and naphthalene (CH2M HILL, 2008a). No COCs were identified in soil. Prior to the In-situ Remediation Pilot Study, COC concentrations in groundwater were limited to a relatively small area, demonstrated a declining trend over multiple years, and were relatively low. Evaluation of the RI and post-RI groundwater data indicated that although already low, certain COC concentrations could require more than a decade to decrease to acceptable levels. Therefore, a Pilot Study was implemented to determine if accelerated achievement of acceptable COC concentrations was possible.

The Pilot Study was implemented in a two-step systematic approach (ISCO directly followed by EISB) to initially oxidize organics and then increase the intrinsic biodegradation rate to reduce the attenuation time needed to achieve acceptable COC concentrations in groundwater. The baseline monitoring and ISCO injection were initiated in March 2010, followed by a post-injection monitoring event, application of EISB, and then three additional post-injection performance monitoring events, with the last monitoring event completed in November 2012.

The pertinent planning documents that set the framework for the implementation of the Pilot Study comprise the Final In-Situ Remediation Pilot Studies (AOC E and AOC I Sites) Sampling and Analysis Plan, Former Naval Ammunition Support Detachment, Vieques, Puerto Rico (CH2M HILL, 2010a), hereafter referred to as the Pilot Study Sampling and Analysis Plan (SAP), and the Technical Memorandum entitled Proposed Pilot Study of In-Situ Remediation at Vieques AOC I (CH2M HILL, 2008b). Results from the baseline monitoring, ISCO injection event, and the first performance monitoring event were documented in the report entitled: Status Report, Area of Concern I, In-Situ Remediation Pilot Study (CH2M HILL, 2011). Pertinent information from the Status Report is included in this Pilot Study Report.

1.1 Pilot Study Objectives and Goals

The objectives of the Pilot Study were to:

- Determine if the groundwater Pilot Study technologies could reduce the groundwater COC concentrations to acceptable levels.
- Determine if the Pilot Study technologies could reduce the groundwater cleanup timeframe (relative to that predicted by natural attenuation alone).
The Pilot Study approach consisted of an ISCO injection of sodium persulfate (sodium hydroxide alkaline activated Klozur) into four existing 2-inch-diameter monitoring wells (MW-02, MW-03, MW-04, and MW-07, as shown in Figure 4), followed by EISB by placing oxygen releasing compound (ORC) “socks” into the same monitoring wells and into one additional downgradient monitoring well (MW-05). Periodic groundwater monitoring (COC, geochemical, and microbial, as applicable) during March 2010 (pre-injection [a.k.a., baseline]), November 2010, and November 2011 were planned to evaluate the effectiveness of the Pilot Study technologies. Although the data collected during the planned Pilot Study performance monitoring period indicated COC concentrations had decreased to acceptable levels, the ERP Technical Subcommittee concurred during the February 2012 meeting to collect two additional rounds of samples to ensure rebound did not occur and that no further action is warranted (CH2M HILL, 2012). These two sampling events occurred in May and November 2012.

The following Pilot Study Preliminary Remediation Goals (PRGs) were developed based upon the USEPA Maximum Contaminant Levels (MCLs), or other standards for constituents without MCLs.

<table>
<thead>
<tr>
<th>COCs</th>
<th>Pilot Study PRGs</th>
<th>Source of PRGs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>5 µg/L</td>
<td>MCL</td>
</tr>
<tr>
<td>Bis (2-ethylhexyl) phthalate</td>
<td>6 µg/L</td>
<td>MCL</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>3.8 µg/L</td>
<td>PRWQS for Groundwater-SG, lower than MCL of 5 µg/L</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>5 µg/L</td>
<td>MCL</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>27 µg/L</td>
<td>HI of 1: not a potential carcinogen, based on the December 2012 EPA Regional Screening Level</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>6.1 µg/L*</td>
<td>HI based, (using December 2012 RSL for tap water for non-carcinogenic endpoints).</td>
</tr>
</tbody>
</table>

µg/L = micrograms per liter; HI = hazard index; MCL = maximum contaminant level; PRWQS = Puerto Rico Water Quality Standards
* A Pilot Study PRG of 1.4 µg/L was originally selected solely to represent a conservative screening value to evaluate the technical implementability and effectiveness of the proposed Pilot Study technology. The value of 6.1 µg/L is hazard index based, using the December 2012 RSL for tap water for non-carcinogenic endpoints, and is more appropriate to use as a PRG.

The 2011 Edition of the Drinking Water Standards and Health Advisories (issued by the USEPA Office of Water) indicates that the cancer classification of naphthalene is “I – inadequate information to assess carcinogenic potential.” The Lifetime Health Advisory (HA) Level of 100 µg/L for naphthalene is defined as the concentration of naphthalene in drinking water that is not expected to cause any adverse noncarcinogenic effects for a lifetime of exposure. In the updated 2012 Edition of the Drinking Water Standards and Health Advisories, the HA Level of 100 µg/L for naphthalene is unchanged.

The Record of Decision (ROD) entries contained in the USEPA CERCLIS Public Access Database were searched for naphthalene cleanup goals in EPA Region 2. For the nine Superfund Sites where quantitative cleanup goals were available for naphthalene, goals ranged from 10 to 300 µg/L. A PRG of 10 µg/L was selected for three sites in New York, as stipulated in the NYSDEC Groundwater Standards, based on a non-carcinogenic endpoint HI of 1 with an uncertainty factor (UF) of 10 for “Group C” carcinogens to provide sufficient protection from possible carcinogenic effects. Additionally, naphthalene does not have a groundwater standard (SG) in the Puerto Rico Water Quality Standards (PRWQS).

The May 2013 USEPA Regional Screening Level (RSL) Table provides carcinogenic inhalation toxicity values for naphthalene, with a tap water RSL of 0.14 µg/L corresponding to a 1x10-6 excess lifetime cancer risk (ELCR) (or 14 µg/L corresponding to 1x10-4 ELCR). USEPA’s target range for ELCR is 1x10-4 to 1x10-6. The 2013 RSL table also identifies a tap water RSL of 6.1 µg/L for non-carcinogenic endpoints, based on an HI of 1 (for cumulative exposures via ingestion/dermal/inhalation).

Based on the above information, the HI-based PRG of 6.1 µg/L, especially considering it is within the USEPA’s acceptable ELCR range, is used as the PRG for naphthalene.
1.2 Site Background

Vieques is located in the Caribbean Sea approximately 7 miles southeast of the eastern tip of the island of Puerto Rico (Figure 1). Vieques is the largest offshore island of the Commonwealth of Puerto Rico. It is approximately 20 miles long and 4.5 miles wide, and has an area of approximately 33,088 acres (51 square miles).

The Navy purchased large portions of Vieques in the early 1940s to conduct activities related to military training. Site operations within the Former NASD consisted mainly of ammunition loading and storage, vehicle and facility maintenance, and some training. The Navy ceased facility-wide operations on the Former NASD on April 30, 2001, in accordance with Presidential Directive to the Secretary of Defense of January 30, 2000, when the land was transferred to the DOI, MOV, and the Puerto Rico Conservation Trust. The property that contains AOC I was transferred to the MOV.

On February 11, 2005, the Atlantic Fleet Weapons Training Area – Vieques was placed on the National Priorities List (NPL), which required all subsequent environmental restoration activities for Navy Installation Restoration (IR) sites on Vieques to be conducted under CERCLA. On September 7, 2007, the Navy, DOI, USEPA, and PREQB executed a Federal Facility Agreement (FFA) that establishes the procedural framework and schedule for implementing the CERCLA response actions for Vieques.

AOC I is a former asphalt plant, located approximately 900 feet south of Mosquito Pier, adjacent to an active rock quarry within the former NASD and current MOV property. The asphalt plant was in operation from the 1960s through 1988. The former asphalt plant comprised a large concrete pad, asphalt mixing drum, earthen ramp, two concrete-paved containment areas, and an area where two diesel fuel aboveground storage tanks (ASTs) were located (Figure 3).

The AOC I area occupies approximately 1 acre, but the asphalt plant itself occupied a considerably smaller area. The topography of the site is relatively flat; stormwater at and in the immediate vicinity of the former asphalt plant was observed to pond at the site during a rain event rather than run off. At the northern, eastern, and southern margins of the site, the topography slopes downward to Route 200 (to the north), the quarry (to the south), and a drainage ditch for the quarry (to the east). Currently there is no continuous human presence or use of the site other than potentially as a passageway for trucks to/from the rock quarry from Route 200. The area that includes the site is fenced to discourage trespassing. Ecological habitat at the former asphalt plant is minimal, consisting primarily of scrub grass, brush, and small trees growing in and around the former asphalt plant structures and through the gravel-covered terrain. No federally-protected species or preferred habitats were observed at AOC I, nor are any cultural resources present at the site.

1.3 Previous Investigations

Previous environmental investigations conducted at AOC I prior to the implementation of the Pilot Study comprise:

- An Environmental Baseline Survey (EBS) was conducted in 2000 to disclose relevant information regarding the environmental condition of the site prior to property transfer of the former NASD (ERM, 2000). A reconnaissance of AOC I was conducted that identified two concrete-bermed containment areas with sumps. Three surface soil samples were collected. The EBS concluded that AOC I should be further investigated under the IR Program.

- An Expanded Preliminary Assessment (PA)/Site Inspection (SI) was conducted at AOC I in 2000 that consisted of an ecological survey and soil sampling from 26 co-located surface soil and subsurface soil samples to determine whether a release had occurred. The Expanded PA/SI recommended the site be investigated further in an RI to delineate the extent of surface soil impacts at the site and conduct a risk assessment (CH2M HILL, 2002).
RI activities were conducted in 2004, 2005, and 2006 that included surface soil sampling at 18 locations, subsurface soil sampling at 7 locations, and installing and sampling 9 monitoring wells (CH2M HILL, 2005; 2008a). The baseline Human Health Risk Assessment (HHRA) identified six COCs in groundwater. No human health COCs were identified in soil because the potential risks associated with the chemical constituents detected in soil were within acceptable limits (CH2M HILL, 2008a). Additionally, the soil concentrations of the six groundwater COCs were also lower than the concentrations that would likely need to be present to pose a leaching-to-groundwater concern. The Ecological Risk Assessment (ERA) concluded there were no unacceptable risk for ecological receptors at AOC I (CH2M HILL, 2008a).

To help determine the appropriate path forward for the site, a post-RI round of groundwater samples was collected in July 2008 since 2 years had elapsed since the last RI sampling event.

1.4 Conceptual Site Model

The surficial material at the site comprises gravel fill interspersed with silty clay and sand. Beneath the thin veneer of fill, the soil zone at the site is relatively thin (generally 2 to 9 feet thick) and consists of well-graded gravel with sand of the Qa geologic unit (Quaternary or Holocene alluvium). Andesite bedrock lies below the soil, generally weathered at its surface. Figure 5 shows a geologic cross section through the site.

The upper portion of the bedrock is unsaturated. Depth to groundwater typically ranges from 14 to 22 feet below ground surface (bgs), with seasonal fluctuation up to approximately 9 feet. The directions and rates of groundwater movement in the andesite bedrock are confined by the size, frequency, and orientation of fractures and by the hydraulic gradient and, therefore, can be quite variable on the small-scale. However, the general direction of groundwater flow at AOC I for all eight rounds of water level measurements is northwest toward the Vieques Passage.

The hydraulic conductivity measured in 2004 and 2006 ranged from 0.1 foot per day (ft/day) to 8.6 ft/day (CH2M HILL, 2008b). The northern area of the site (represented by well MW-06) has the lowest hydraulic conductivity of 0.1 ft/day, while average hydraulic conductivity in southern and central portion of the site (represented by wells MW01 through MW-05) is 4.1 ft/day. The horizontal hydraulic gradient in the southern and central portion of AOC I ranges from approximately flat (November 2012) to approximately 0.005 feet per foot (ft/ft) (November 2011), but increases to a range of approximately 0.015 ft/ft (November 2012) to 0.032 ft/ft (November 2011) in the northern portion of AOC I. Based on the above information, a relatively low groundwater velocity ranging from 3 to 16 feet per year is suggested, with higher seepage velocity observed in the southern and central portion of the AOC I (CH2M HILL, 2010a).

The conceptual site model of AOC I is presented in Figure 6, which shows the historical features and potential contaminant migration routes. Based on the historical activities and extent of contamination identified during the RI and related investigations, releases occurred during past asphalt plant operations, likely in the form of minor drips and spills. The primary route of contaminant migration was likely vertical leaching through soil and bedrock to groundwater and subsequent transport with groundwater flow through fractures in the bedrock. However, the data show the extent of contamination was generally limited to the immediate vicinity of the former asphalt plant. Further, the pre-Pilot Study contaminant levels present in environmental media were relatively low with respect to human health-based and ecological-based screening values. No unacceptable human health or ecological risks were identified in soil at the site. However, COCs were identified in groundwater; COC concentrations in groundwater prior to the ISCO/EISB Pilot Study (i.e., from 2004 through 2008) are presented in Figure 7. Concentrations exceeding PRGs were limited to the area of MW-04, MW-05, and MW-07, which is the area immediately underlying the main operational activities of the former asphalt plant. However, data from the last sampling event prior to the start of the pilot study show the area of exceedance was limited to MW-07.
SECTION 2

Pilot Study Field Activities

A summary of the Pilot Study field activities completed to date at AOC I is provided in Table 1. Site monitoring/injection wells are shown in Figure 4. The basis for the pilot study field activities can be found in the Pilot Study SAP (CH2M HILL, 2010a). The Vieques Technical Subcommittee, comprising representatives of the Navy, USEPA, and EQB, concurred on the wells to include in the Pilot Study based on historical data and Pilot Study objectives. Wells MW-01, MW-06, MW-08, and MW-09 were excluded from contaminant analysis during the Pilot Study because they were either upgradient of (MW-01) or far downgradient from (MW-06, MW-08, and MW-09) the area of contamination. These wells had been installed during the RI for the purposes of nature and extent determination but were not relevant to the Pilot Study. Due to the small size of the groundwater plume and slow groundwater velocity rates (3 to 16 ft/yr), MW-02, MW-03, MW-04, MW-05, and MW-07 were determined by the Technical Subcommittee as the appropriate wells to be used for monitoring contaminant concentrations during the Pilot Study.

2.1 Baseline Monitoring

Groundwater elevations were measured from nine AOC I monitoring wells on March 15, 2010 (Table 2). Groundwater samples were collected from five monitoring wells (MW-02, MW-03, MW-04, MW-05, and MW-07) between March 18 and 22, 2010. Low-flow sampling techniques were used to purge and collect groundwater samples. Field readings (including turbidity) conducted during sampling were recorded on well purging forms, which are provided in Appendix A. Stabilized field parameters are listed in Table 3. Persulfate concentrations measured with a field test kit at time of sampling are provided in Table 4. Persulfate concentrations were measured to provide a pre-injection baseline. All sampling activities were conducted in accordance with the Pilot Study SAP.

2.2 In-Situ Chemical Oxidation Injection

During the Pilot Study design, the oxidant (persulfate) demand was estimated based on: a) the historical groundwater geochemical data and water quality parameters (showing the anaerobic nature of the subsurface and likelihood of reduced iron and manganese exerting a demand on persulfate), b) the stoichiometric demand based on the historical COC concentrations, and c) professional judgment from numerous persulfate applications. Due to the very low COC concentrations and lack of NAPL at AOC I, the stoichiometric demand, as is common, was negligible.

ISCO injection activities were conducted from March 27 to 31, 2010 by ORIN Remediation Technologies, Inc. of McFarland, Wisconsin in accordance with the Pilot Study SAP. Approximately 835 pounds of sodium persulfate and 800 pounds of sodium hydroxide as an activator were mixed into a 5 percent by weight solution, and injected into MW-02, MW-03, MW-04, and MW-07 (for a total of approximately 2,033 gallons). A summary of the amount of solution injected into each monitoring well and field parameters recorded from the various monitoring wells are provided in Appendix B.

Approximately 500 gallons of mixed persulfate/NaOH solution (approximately 209 pounds persulfate and 19 gallons 25 percent NaOH solution with water to make 500 gallons mixture) were injected into each well (MW-02, MW-03, MW-04, and MW-07). These quantities are approximate as a few injections were done simultaneously and in one instance the flow rate was estimated because it was too low for flow meters to measure accurately (less than 1 gallons per minute [gpm]). The injection pressures ranged from 0 to 30 pounds per square inch (psi), only once briefly reaching the maximum of 30 psi. When the pressure reached 30 psi, the injection rate was lowered to decrease the pressure, which was maintained below 30 psi. The flow rates ranged from approximately 0.8 to 3.9 gpm.

No mounding was observed in the surrounding monitoring wells, nor was persulfate detected in wells that were not used as injection wells. This indicates that the injections influenced the local vicinity around each well as designed, and the oxidant solution did not migrate outside the COC-impacted area. Although
fractures in the bedrock at AOC I may have provided preferential pathways for contaminant migration, the ISCO injections would have followed those same pathways since the injections were intentionally performed at very low pressures to avoid creating additional preferential flow pathways. Monitoring during injection was performed and showed no mounding in surrounding wells.

2.3 Persulfate Monitoring

The AOC I monitoring wells to be sampled during the first post-injection performance monitoring event were tested for residual persulfate approximately 2 months before sampling. The purpose was to confirm the persulfate had been consumed sufficiently so that it would not potentially react with the COCs between the time the samples were collected and the time they were analyzed at the laboratory. The residual sodium persulfate concentrations in AOC I monitoring wells measured on August 24-27, 2010, as shown in Table 4, ranged from non-detect to 105 milligram per liter (mg/L) (MW-07). In a September 20, 2010 Technical Memorandum (CH2M HILL, 2010b), included in Appendix C, the Navy suggested groundwater containing less than 500 mg/L persulfate be sampled using the procedure set forth in the SAP. This proposal was based on a Technical Memorandum by FMC supporting such a limit (FMC, 2010), also included in Appendix C. Per an e-mail from USEPA’s Scott Huling, also included in Appendix C, USEPA preferred to have the samples preserved with ascorbic acid (USEPA, 2010). The Vieques Technical Subcommittee agreed on the sampling approach in an October 4, 2010 conference call (CH2M HILL, 2010c). The procedure agreed upon was that for AOC I groundwater samples containing residual persulfate, the residual persulfate would be neutralized using ascorbic acid (instead of hydrochloric acid) before placing these on ice for shipment to the laboratory for analysis. Persulfate monitoring was conducted in accordance with the SAP (CH2M HILL, 2010a).

2.4 First Post-injection Performance Monitoring Event

The first post-injection performance monitoring event was conducted from November 1 to 4, 2010. Groundwater elevations measured from each monitoring well are summarized in Table 2 and presented in Figure 8. Groundwater flow was discussed in Section 1.4 and is further discussed in Section 3. Low-flow sampling techniques were used to purge and collect groundwater samples. Field readings (including turbidity) conducted during sampling were recorded on well purging forms, which are provided in Appendix A. Stabilized field parameters are listed in Table 3. Persulfate concentrations measured in groundwater using field test kits are presented in Table 4. The persulfate was measured after field parameters stabilized, immediately prior to collecting samples.

For informational purposes, groundwater collected from the five monitoring wells was processed with three different approaches in the field. One set of samples was collected in ascorbic acid-preserved vials, as concurred upon by the Technical Subcommittee (CH2M HILL, 2010c); a second set was collected in unpreserved vials; and a third set was collected in hydrochloric acid-preserved vials (i.e., in accordance with the SAP). Table 4 shows the persulfate concentrations measured in wells at the time of sample collection. Table 5 shows the results of the three analyses (with identification of the preservative method for each) for each well. Of note is that the volatile organic compounds (VOCs) concentrations for each well were essentially the same among the samples preserved with hydrochloric acid, ascorbic acid, and unpreserved. For example, benzene concentrations in samples from well MW-07, which had a measured persulfate concentration between 14 and 21 mg/L, were 9.5 µg/L (unpreserved), 9.5 µg/L (ascorbic acid), and 9.4 µg/L (HCl). Therefore, at the concentrations observed at this site and given the water geochemistry, it does not appear to make a difference for VOC groundwater results how or if the samples were preserved.

2.5 Enhanced In-Situ Bioremediation

Following the post-injection groundwater sampling event in November 2010, ten 2-inch diameter ORC socks (strung together) were placed down each of monitoring wells MW-02, MW-03, MW-04, MW-05 and MW-07 in accordance with the Pilot Study SAP. The ORC sock suspension lines were attached to a fitting on the underside of each well cap, allowing the ORC socks to remain suspended and submerged in groundwater
within the screen zone when the well cap was in place. The ORC socks were removed in July 2011 according to the schedule in the Pilot Study SAP.

2.6 Second Post-injection Performance Monitoring Event

The second post-injection performance monitoring event was conducted from November 9 to 10, 2011. Groundwater elevations measured from each monitoring well are summarized in Table 2 and presented in Figure 9. Low-flow sampling techniques were used to purge and collect groundwater samples. Field readings (including turbidity) conducted during sampling were recorded on well purging forms, which are provided in Appendix A. Stabilized field parameters are listed in Table 3. Persulfate concentrations measured in groundwater using field test kits are presented in Table 4. Persulfate was measured after field parameters stabilized, immediately prior to collecting samples.

The results of the analyses for each well are presented in Table 5 and are discussed in Section 3.

2.7 Third Post-injection Performance Monitoring Event

After the first two post-injection sampling events scheduled in the SAP were conducted, the data were presented to the Vieques Environmental Technical Subcommittee with a recommendation to prepare a no further action proposed plan and record of decision. However, to ensure contaminant rebound was not observed, the Technical Subcommittee agreed to perform two additional sampling events for a subset of the AOC I monitoring wells (i.e., MW-04, MW-05, and MW-07). This agreement was reached in the February 22, 2012 Technical Subcommittee meeting. The third post-injection performance monitoring event was conducted from May 22 to 23, 2012. Groundwater elevations measured from each monitoring well are summarized in Table 2 and presented in Figure 10. Low-flow sampling techniques were used to purge and collect groundwater samples. Field readings (including turbidity) conducted during sampling were recorded on well purging forms, which are provided in Appendix A. Stabilized field parameters are listed in Table 3. Persulfate concentrations measured in groundwater using field test kits are presented in Table 4. Persulfate was measured immediately prior to collecting samples.

2.8 Fourth Post-injection Performance Monitoring Event

The fourth post-injection performance monitoring event was conducted from November 28 to 29, 2012. Groundwater elevations measured from each monitoring well are summarized in Table 2 and presented in Figure 11. Low-flow sampling techniques were used to purge and collect groundwater samples. Field readings (including turbidity) conducted during sampling were recorded on well purging forms provided in Appendix A. Stabilized field parameters are listed in Table 3. Persulfate concentrations measured in groundwater using field test kits are presented in Table 4. Persulfate was measured immediately prior to collecting samples.
Groundwater Monitoring Results

This section summarizes the results of the groundwater monitoring activities during the baseline and post-injection performance monitoring events. The groundwater elevations measured at each of the monitoring wells prior to each sampling event are tabulated in Table 2 and are shown in Figures 8 through 11. While the groundwater elevation fluctuated by as much as 9 feet, the groundwater flow direction stayed consistently to the northwest. The south and central portion of the site, where area of contamination was localized, has a relatively flat gradient. As discussed in section 3.2, there is no correlation between groundwater elevation and COC concentrations in groundwater. Field parameters and detected analytical concentrations are provided in Tables 3 and 5, respectively. The groundwater data were validated in accordance with the Pilot Study SAP. Concentration trends of benzene and naphthalene are shown in Figures 12 through 15, and are discussed below. Concentrations of the other COCs had already decreased to below PRGs prior to the start of the pilot test, as shown in Table 5. Analytical data and data validation reports for all Pilot Study sampling efforts are provided in Appendix D.

3.1 Geochemical Parameters

Groundwater temperature remained consistent during the four sampling events (between 28 and 30 degrees Celsius), which is conducive for both ISCO and EISB (Table 3). The pH remained relatively neutral; however, one monitoring well (MW-07) exhibited elevated pH values from November 2011 to November 2012, potentially due to low residual sodium hydroxide base used to activate the sodium persulfate during ISCO. The pH showed a decreasing trend over the time period from November 2011 to 2012. While elevated pH is not ideal for EISB, the pH range in the other four wells supplied with ORC socks is optimal for EISB.

The specific conductivity increased between the first and second monitoring events at four monitoring wells (MW-02, MW-03, MW-04, and MW-07) before decreasing through the third and fourth events. This trend is likely a result of the residual sodium from the sodium persulfate oxidant and sodium hydroxide catalyst injected into these monitoring wells (sodium persulfate was not injected into well MW-05). The overall low dissolved oxygen (DO) concentrations suggest reducing conditions occur naturally in the aquifer. However, the DO readings of 6.59 mg/L in MW-02 in November 2010 and readings of 11.15 mg/L and 5.44 mg/L in 2011 and 2012, respectively, in MW-07 in 2012 may be the result of localized oxidizing conditions induced during ISCO or residual oxygen released during EISB. Oxygen reduction potential (ORP) concentrations ranged from -70.6 to 113 millivolts (mV) during the baseline sampling in March 2010, showed increasing trends in each individual well over the next few sampling events, and were between -232.8 and 25.4 mV (lower than initial values) in November 2012. The initial increases in ORP are indicative of the oxidant’s effect on groundwater.

Dissolved iron and manganese were analyzed to confirm the presence of an oxidative environment post-injection, which would tend to decrease dissolved iron and manganese. As shown in Table 5, this is what was observed; iron and manganese concentrations declined at the injection wells (MW-02, MW-03, MW-04, and MW-07) following the ISCO injection, indicative of the desired oxidative conditions. Several wells also showed increases of these metals toward the end of the study, indicating a return to normal geochemical conditions.

3.2 COC Concentration Trends

Based on the baseline groundwater monitoring event, benzene and naphthalene were identified as the key COCs (Figure 7). 1,2-dichloroethane, 1,2-dichloropropane, bis(2-ethylhexyl)phthalate, and 2-methylnaphthalene were either not detected or were detected at concentrations below the Pilot Study PRGs during baseline sampling and all subsequent monitoring events.

Benzene was detected above the Pilot Study PRG (Figures 7, 12, and 13) at only one monitoring well (MW-07) following the injection. Although the concentration demonstrate a decrease from 14 micrograms per
liter (µg/L) during baseline sampling to 0.82 µg/L during November 2012 sampling, the change in concentration may be due to natural degradation as well as ISCO influence. Benzene concentrations have decreased steadily since the baseline sampling event, with no evidence of rebound in the 2012 sampling events.

Like benzene, naphthalene (Figures 7, 14, and 15) was detected above the Pilot Study PRG in only one monitoring well (MW-07) following the injection. Also like benzene, naphthalene concentrations in MW-07 decreased to be below the Pilot Study PRG and showed no evidence of rebound.

As stated in Section 1.1, the objectives of the Pilot Study implemented at AOC I were to: (1) determine if the groundwater Pilot Study technologies could reduce COC concentrations to acceptable levels and (2) determine if the Pilot Study technologies could reduce the groundwater cleanup timeframe (relative to that predicted by natural attenuation alone). The associated project quality objective (PQO), as documented in Worksheet 11 of the Pilot Study SAP (CH2M HILL, 2010a), was to collect data sufficient for determining whether unacceptable risk associated with potential potable groundwater use at the site was mitigated (i.e., all COC concentrations below Pilot Study PRGs) and, therefore, no further action was warranted.

As noted previously, the concentrations of all groundwater COCs in all wells (except benzene and naphthalene in well MW07) had declined to below Pilot Study PRGs before the Pilot Study baseline sampling (i.e., between 2004 and 2010). For MW07, Table 6 summarizes the percent reduction of benzene and naphthalene in monitoring well MW-07 prior to and during the Pilot Study implementation. The table also includes 2-methylnaphthalene because it helps demonstrate the potential affect on COC concentration decline by natural processes and the Pilot Study technologies. As shown in the table, the concentrations of these three COCs declined between 74 percent and 79 percent over the 5 ½ years prior to the Pilot Study (i.e., under the influence of natural attenuation processes alone). During the 2 ½-year Pilot Study, the same COCs declined by about 95 percent.

In addition to the above, natural attenuation modeling (see Attachment C of the Pilot Study SAP [CH2M HILL, 2010a]) indicated it would take approximately 7 years for benzene and 14 years for naphthalene to decline from levels measured at AOC I in 2008 to the Pilot Study PRGs under the influence of natural attenuation processes alone. As shown in Figures 12 and 14, the Pilot Study PRGs for both of these two COCs were achieved in about 4 years (i.e., 2008 to 2012).

The information above indicates the decreases in COC concentrations were attributable to both natural processes and Pilot Study technologies, with the Pilot Study technologies likely accelerating the decline to below the PRGs. Regardless of the relative contribution of natural processes and Pilot Study technologies, the monitoring conducted before and during the Pilot Study indicated all COCs at the site declined to below the PRGs without rebound.

### 3.3 Residual Human Health Risk

Although the COC concentrations decreased to below Pilot Study PRGs (in most cases before the pilot study was initiated), human health risk calculations were performed using the most recent COC concentrations (i.e., from May and November 2012) to ensure residual COC concentrations do not pose an unacceptable risk under an unrestricted use scenario. As shown in Table 5, only three of the six COCs were detected in 2012. Based on maximum detected concentrations in 2012 and comparison to the USEPA Regional Screening Levels for tap water (November 2012), the total excess lifetime cancer risk (ELCR) is 3x10⁻⁵ and the maximum target organ-specific hazard index (HI) is 0.5 (Table 7). Both the ELCR and HI are within USEPA-acceptable risk levels.
In summary, the conclusions for the In-situ Pilot Study are as follows:

- The groundwater Pilot Study technologies (potentially coupled with natural processes) achieved the Pilot Study goals by reducing the groundwater COC concentrations to acceptable levels within 26-months (from March 2010 to May 2012), a rate faster than predicted by natural attenuation alone.

- The Pilot Study results are applicable to the site as a whole since the area of contamination was small enough to apply the Pilot Study site-wide.

- Only monitoring well MW-07 showed COC exceedances at the start of the Pilot Study, and only for two COCs: benzene and naphthalene.
  - Benzene concentrations decreased from 59.3 µg/L in September 2004 to 0.82 µg/L in November 2012 (from 14 µg/L to 0.82 µg/L during the Pilot Study). Benzene concentrations declined naturally by 76 percent prior to the Pilot Study and by 94 percent following the ISCO injection and EISB application; overall concentrations declined by 99 percent. Benzene fell below its PRG of 5 µg/L between November 2011 and May 2012 and no rebound was observed.
  - Naphthalene concentrations decreased from 96 µg/L in January 2006 to being undetected in November 2012 (from 21 µg/L to non-detect during the Pilot Study). Naphthalene concentrations declined naturally by 74 percent prior to the Pilot Study and by 95 percent following the ISCO injection and EISB application; overall concentrations declined by 99 percent. Naphthalene fell below its PRG of 6.1 µg/L between November 2011 and May 2012 and no rebound was observed.

- 1,2-Dichloroethane concentrations decreased from 1.6 µg/L (January 2006) to below detection (July 2008). 1,2-Dichloroethane has not been detected since 2006.

- 1,2-Dichloropropane concentrations decreased from 0.33 µg/L (September 2004) to below detection (January 2006). 1,2-Dichloropropane has not been detected since 2004.

- 2-Methylnaphthalene concentrations decreased from 110 µg/L (January 2006) to 1.1 µg/L (November 2012). 2-Methylnaphthalene concentrations fell below the PRG of 27 µg/L between January 2006 and July 2008, prior to the start of the pilot test.

- Bis(2-Ethylhexyl)phthalate concentrations decreased from 9.6 µg/L (September 2004) to below detection (May 2012). Bis(2-Ethylhexyl)phthalate concentrations fell below the PRG of 6 µg/L between September 2004 and July 2008, prior to the start of the pilot test.

Before implementing the Pilot Study, groundwater COC concentrations were trending down due to natural attenuation and a likely overall decrease in residual adsorbed COC mass in the fine-grained matrix. Implementing ISCO and EISB likely accelerated the rate of mass dissolved concentration decreases, and no rebound has been observed. In addition, residual risk under a potable use scenario is acceptable because the ELCR and HI based on the current concentrations of all COCs are within USEPA-acceptable risk levels. Because there are no soil COCs, because the Pilot Study PRGs were achieved site-wide and no rebound was observed, and because residual risks are within acceptable levels under a potable use scenario, no further action is warranted for AOC I. A no further action proposed plan and record of decision will be prepared for AOC I based on information presented in the RI and this Pilot Study report.
SECTION 5

References


CH2M HILL. 2010c. Telephone conversation record-Ascorbic Acid Additive to Post-Injection samples collected at AOC I. October.


Tables
<table>
<thead>
<tr>
<th>Date</th>
<th>Specific Activity</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>March 18-22, 2010</td>
<td>Baseline Groundwater Sampling Event</td>
<td>Purged and sampled 5 monitoring wells (MW-02, MW-03, MW-04, MW-05, and MW-07).</td>
</tr>
<tr>
<td>March 27-31, 2010</td>
<td>ISCO Injection Event</td>
<td>Injected 835 pounds of sodium persulfate with 800 lbs of sodium hydroxide (total of approximately 2,033 gallons) across four existing monitoring wells (MW-02, MW-03, MW-04, and MW-07).</td>
</tr>
<tr>
<td>August 24-27, 2010</td>
<td>Measured persulfate concentrations in wells</td>
<td>Performed in preparation for sampling in accordance with SAP. Residual sodium persulfate was detected in some wells; worked with EPA/EQB between September and October 2010 to modify the sampling approach to account for residual persulfate (use of ascorbic acid as preservative).</td>
</tr>
<tr>
<td>November 4, 2010</td>
<td>EISB (ORC sock placement)</td>
<td>Installed 2-inch diameter ORC socks in the screen zone of monitoring wells MW-02, MW-03, MW-04, MW-05, and MW-07.</td>
</tr>
<tr>
<td>-------------------</td>
<td>-----------------------------------</td>
<td>----------------------------------------</td>
</tr>
<tr>
<td>MW-01</td>
<td>35.27</td>
<td>0.40 to -0.56</td>
</tr>
<tr>
<td>MW-02</td>
<td>34.54</td>
<td>-0.36 to -0.36</td>
</tr>
<tr>
<td>MW-03</td>
<td>34.77</td>
<td>-0.58 to -2.42</td>
</tr>
<tr>
<td>MW-04</td>
<td>34.96</td>
<td>-2.81 to -1.19</td>
</tr>
<tr>
<td>MW-05</td>
<td>34.82</td>
<td>0.22 to -0.78</td>
</tr>
<tr>
<td>MW-07</td>
<td>35.16</td>
<td>-0.27 to -10.27</td>
</tr>
<tr>
<td>MW-08</td>
<td>33.81</td>
<td>0.81 to -0.10</td>
</tr>
</tbody>
</table>

Notes:
- ft BTOC = feet below top of casing
- ft amsl = feet above mean sea level
- Ni: Not yet installed
- MWs-02, 03, 04, and 07 had ISCO applied in them March 27-31, 2010. The previously mentioned wells and MW-05 received EISB treatment November 4, 2010 to July 27, 2011.
<table>
<thead>
<tr>
<th>Field Parameter</th>
<th>MW-02 Date</th>
<th>MW-03 Date</th>
<th>MW-04 Date</th>
<th>MW-05 Date</th>
<th>MW-07 Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (ºC)</td>
<td>29.72</td>
<td>29.34</td>
<td>28.38</td>
<td>29.45</td>
<td>28.80</td>
</tr>
<tr>
<td>Specific Conductance (mS/cm)</td>
<td>1.095</td>
<td>1.620</td>
<td>1.084</td>
<td>0.17</td>
<td>0.51</td>
</tr>
<tr>
<td>Dissolved Oxygen (mg/L)</td>
<td>0.13</td>
<td>6.19</td>
<td>0.26</td>
<td>0.37</td>
<td>0.51</td>
</tr>
<tr>
<td>Oxidation-Reduction Potential (mV)</td>
<td>113.0</td>
<td>172.1</td>
<td>25.4</td>
<td>-8.7</td>
<td>70.6</td>
</tr>
<tr>
<td>pH</td>
<td>6.89</td>
<td>6.08</td>
<td>6.96</td>
<td>7.28</td>
<td>6.09</td>
</tr>
<tr>
<td>Turbidity (NTU)</td>
<td>2.02</td>
<td>1.11</td>
<td>6.31</td>
<td>10.2</td>
<td>3.49</td>
</tr>
</tbody>
</table>

**Notes:**
- ºC: degrees centigrade
- mS/cm: millisiemens per centimeter
- mg/L: milligrams per liter
- mV: millivolts
- NTU: Nephelometric Turbidity Unit
- NM: not measured

MW-02, 03, 04, and 07 had ISCO applied in them (March 27-31, 2010). These wells and MW-05 received EISB treatment November 4, 2010 to July 27, 2011.
**TABLE 4**  
Persulfate Concentration  
AOC / In-Situ Remediation Pilot Study Report  
Former Naval Ammunition Support Detachment  
Vieques, Puerto Rico  

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>MW-01</td>
<td>nm</td>
<td>0</td>
<td>nm</td>
<td>nm</td>
<td>nm</td>
<td>nm</td>
</tr>
<tr>
<td>MW-02</td>
<td>0</td>
<td>14</td>
<td>14</td>
<td>0.7</td>
<td>nm</td>
<td>nm</td>
</tr>
<tr>
<td>MW-03</td>
<td>0</td>
<td>2.1</td>
<td>0-0.7</td>
<td>0</td>
<td>nm</td>
<td>nm</td>
</tr>
<tr>
<td>MW-04</td>
<td>0</td>
<td>1.4</td>
<td>0-0.7</td>
<td>0.7</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>MW-05</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>MW-07</td>
<td>0</td>
<td>105</td>
<td>14-21</td>
<td>1.4</td>
<td>1.4</td>
<td>0</td>
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<tr>
<td>MW-08</td>
<td>nm</td>
<td>0</td>
<td>nm</td>
<td>nm</td>
<td>nm</td>
<td>nm</td>
</tr>
<tr>
<td>MW-09</td>
<td>nm</td>
<td>0</td>
<td>nm</td>
<td>nm</td>
<td>nm</td>
<td>nm</td>
</tr>
</tbody>
</table>

nm- Not measured

Notes:
MWs-02, 03, 04, and 07 had ISCO applied in them March 27-31, 2010. These wells and MW-05 received EiSB treatment November 4, 2010 to July 27, 2011.
Persulfate monitoring was conducted in accordance with the SAP (CH2M HILL, 2010a).
**TABLE 5**

Analytical Results for COCs, Dissolved Iron and Manganese, and Select Wet Chemistry Parameters for AOC I

AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico

<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample ID</th>
<th>Sample Date</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>UGL (Wet)</th>
<th>UGL (Field)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MW01</td>
<td>MW01</td>
<td>07/24/08</td>
<td>MW01</td>
<td>1,2-Dichloroethane</td>
<td>3.8</td>
<td>0.5 U</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1,2-Dichloroethylene</td>
<td>0.5 U</td>
<td>0.5 U</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Methane</td>
<td>0.5 U</td>
<td>0.5 U</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2-Methylnaphthalene</td>
<td>27</td>
<td>5 U</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2-Naphthalene</td>
<td>6.1</td>
<td>5 U</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Dissolved Metals</td>
<td>100 U</td>
<td>100 U</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Wet Chemistry</td>
<td>NA</td>
<td>NA</td>
</tr>
</tbody>
</table>

Notes:
- Bold indicates detections
- Bolded shading indicates detected exceedance.
- B - Analyte also detected in an associated method blank (unvalidated data).
- J - Below reporting limit (unvalidated data).
- U - Nondetect or not detected at significantly greater than that in an associated blank.
- MG/L - Milligrams per liter
- UG/L - Micrograms per liter

---

**Semivolatile Organic Compounds (UG/L)**

<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample ID</th>
<th>Sample Date</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>UGL (Wet)</th>
<th>UGL (Field)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MW01</td>
<td>MW01</td>
<td>07/24/08</td>
<td>MW01</td>
<td>Benzene</td>
<td>5</td>
<td>0.5 U</td>
</tr>
</tbody>
</table>

**Dissolved Metals (UG/L)**

<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample ID</th>
<th>Sample Date</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>UGL (Wet)</th>
<th>UGL (Field)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MW01</td>
<td>MW01</td>
<td>07/24/08</td>
<td>MW01</td>
<td>Iron, Dissolved</td>
<td>83.3 U</td>
<td>100 U</td>
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<tr>
<td>MW01</td>
<td>MW01</td>
<td>07/24/08</td>
<td>MW01</td>
<td>Manganese, Dissolved</td>
<td>37.9 U</td>
<td>10.9 U</td>
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**Wet Chemistry (MG/L)**

<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample ID</th>
<th>Sample Date</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>UGL (Wet)</th>
<th>UGL (Field)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MW01</td>
<td>MW01</td>
<td>07/24/08</td>
<td>MW01</td>
<td>Nitrate</td>
<td>NA</td>
<td>0.66 U</td>
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<tr>
<td>MW01</td>
<td>MW01</td>
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<td>MW01</td>
<td>Persulfate</td>
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<td>0.74 B</td>
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<td>MW01</td>
<td>07/24/08</td>
<td>MW01</td>
<td>Sulfate</td>
<td>NA</td>
<td>22.2 U</td>
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<td>MW01</td>
<td>MW01</td>
<td>07/24/08</td>
<td>MW01</td>
<td>Total Organic Carbon</td>
<td>NA</td>
<td>2.74 J</td>
</tr>
</tbody>
</table>

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Notes:
- Bold indicates detections
- Bolded shading indicates detected exceedance.
- B - Analyte also detected in an associated method blank (unvalidated data).
- J - Below reporting limit (unvalidated data).
- U - Nondetect or not detected at significantly greater than that in an associated blank.
- MG/L - Milligrams per liter
- UG/L - Micrograms per liter
TABLE 5
Analytical Results for COCs, Dissolved Iron and Manganese, and Select Wet Chemistry Parameters for AOC I
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico

<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample Date</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>Volatile Organic Compounds (UG/L)</th>
<th>Semivolatile Organic Compounds (UG/L)</th>
<th>Dissolved Metals (UG/L)</th>
<th>Wet Chemistry (MG/L)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1,2-Dichloroethane</td>
<td>1,2-Dichloropropane</td>
<td>Benzene</td>
<td>2-Methylnaphthalene</td>
</tr>
<tr>
<td>MW03</td>
<td>11/04/10</td>
<td>HCl</td>
<td></td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>NA</td>
</tr>
<tr>
<td></td>
<td>11/04/10</td>
<td>HCl</td>
<td></td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>NA</td>
</tr>
<tr>
<td></td>
<td>11/04/10</td>
<td>HCl</td>
<td></td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>NA</td>
</tr>
<tr>
<td>MW03</td>
<td>11/04/10</td>
<td>HCl</td>
<td></td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>NA</td>
</tr>
</tbody>
</table>

Notes:
- Bold indicates detections
- Bolded shading indicates detected exceedance.
- J indicates detections indicated as detected exceedance
- U indicates detections indicated as unvalidated data
### TABLE 5
Analytical Results for COCs, Dissolved Iron and Manganese, and Select Wet Chemistry Parameters for AOC I

**Former Naval Ammunition Support Detachment Vieques, Puerto Rico**

<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample ID</th>
<th>Sample Date</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>Analytical Unit</th>
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</thead>
<tbody>
<tr>
<td>MW04-02</td>
<td>09/23/04</td>
<td>01/10/06</td>
<td>MW</td>
<td>Volatile Organic Compounds (UG/L)</td>
<td>0.5 U 0.5 U 0.5 U 5 U 0.5 U 0.5 U 0.5 U 0.5 U 0.5 U 0.5 U</td>
</tr>
<tr>
<td>MW04-10</td>
<td>09/23/04</td>
<td>01/10/06</td>
<td>MW</td>
<td>Semivolatile Organic Compounds (UG/L)</td>
<td>41.4 4.3J 4J 4.6J 1.1J 2.6J 2.2J</td>
</tr>
<tr>
<td>MW04-11</td>
<td>09/23/04</td>
<td>01/10/06</td>
<td>MW</td>
<td>Dissolved Metals (UG/L)</td>
<td>10 U 10 U 10 U 5 U 10 U 10 U 10 U 10 U 10 U 10 U</td>
</tr>
<tr>
<td>MW04-12</td>
<td>09/23/04</td>
<td>01/10/06</td>
<td>MW</td>
<td>Wet Chemistry (MG/L)</td>
<td>65.5 J 30 U 50 U</td>
</tr>
</tbody>
</table>

**Chemical Notes**

- Bold indicates detections
- Bolded shading indicates detected exceedance
- 10-0.7 indicates an estimated value of persulfate that was less than 0.7 mg/L.
- NA - Not analyzed
- B - Analyte also detected in an associated method blank (unvalidated data)
- J - Estimated (validated data).
- J - Below reporting limit (unvalidated data).  
- R - Unreliable Result
- U – Nondetect or not detected at significantly greater than that in an associated blank.
- UJ - Nondetect.  Estimated reporting limit.
- MG/L - Milligrams per liter
- UG/L - Micrograms per liter

---

### Notes

- Bold indicates detections
- Bolded shading indicates detected exceedances
- 10-0.7 indicates an estimated value of persulfate that was less than 0.7 mg/L.
- NA - Not analyzed
- B - Analyte also detected in an associated method blank (unvalidated data)
- J - Estimated (validated data).
- J - Below reporting limit (unvalidated data).
- R - Unreliable Result
- U – Nondetect or not detected at significantly greater than that in an associated blank.
- UJ - Nondetect.  Estimated reporting limit.
- MG/L - Milligrams per liter
- UG/L - Micrograms per liter

---

3 of 6
# TABLE 5
Analytical Results for COCs, Dissolved Iron and Manganese, and Select Wet Chemistry Parameters for AOC I

**AOC I In-Situ Remediation Pilot Study Report**
Former Naval Ammunition Support Detachment Vieques, Puerto Rico

<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample ID</th>
<th>Sample Date</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MW05</td>
<td></td>
<td>Acetic Acid</td>
<td>Volatile Organic Compounds (UG/L)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>MW06</td>
<td></td>
<td>Acetic Acid</td>
<td>Semivolatile Organic Compounds (UG/L)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>MW07</td>
<td></td>
<td>Acetic Acid</td>
<td>Dissolved Metals (UG/L)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>MW08</td>
<td></td>
<td>Acetic Acid</td>
<td>Wet Chemistry (MG/L)</td>
<td></td>
</tr>
</tbody>
</table>

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |

|             | MW05      |             | Acetic Acid         |             |       |
|             | MW06      |             | Acetic Acid         |             |       |
|             | MW07      |             | Acetic Acid         |             |       |
|             | MW08      |             | Acetic Acid         |             |       |

|             | MW09      |             | Acetic Acid         |             |       |
|             | MW10      |             | Acetic Acid         |             |       |
|             | MW11      |             | Acetic Acid         |             |       |
|             | MW12      |             | Acetic Acid         |             |       |
## TABLE 5
Analytical Results for COCs, Dissolved Iron and Manganese, and Select Wet Chemistry Parameters for AOC I

<table>
<thead>
<tr>
<th>Chemical Name</th>
<th>Volatile Organic Compounds (UG/L)</th>
<th>Semivolatile Organic Compounds (UG/L)</th>
<th>Dissolved Metals (UG/L)</th>
<th>Wet Chemistry (MG/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1,2-Dichloroethane</td>
<td>1,2-Dichloropropane</td>
<td>Benzene</td>
<td>2-Methylnaphthalene</td>
</tr>
<tr>
<td>Preservative Method</td>
<td>RW</td>
<td>RW</td>
<td>RW</td>
<td>Unpreserved</td>
</tr>
<tr>
<td>Sample Date</td>
<td>11/04/10</td>
<td>11/04/10</td>
<td>11/04/10</td>
<td>05/23/12</td>
</tr>
<tr>
<td>Station ID</td>
<td>MW07</td>
<td>MW07</td>
<td>MW07</td>
<td>MW07</td>
</tr>
</tbody>
</table>

Notes:
- Bold indicates detections
- * indicates below reporting limit
- Unpreserved Ascorbic Acid
- HCl
- Ascorbic Acid
- NA
- B - Analyte also detected in an associated method (unvalidated data)
- J - Estimated (validated data)
- R - Unreliable Result
- U - Nondetect or not detected at significantly greater than that in an associated blank.
- UJ - Nondetect. Estimated reporting limit.

Units:
- MG/L - Milligrams per liter
- UG/L - Micrograms per liter

**Notes on Reporting Limit**
- *U* - Nondetect or not detected at significantly greater than that in an associated blank.
- UJ - Nondetect. Estimated reporting limit.
<table>
<thead>
<tr>
<th>Station ID</th>
<th>Sample ID</th>
<th>Preservative Method</th>
<th>Chemical Name</th>
<th>Volatile Organic Compounds (UG/L)</th>
<th>Semivolatile Organic Compounds (UG/L)</th>
<th>Dissolved Metals (UG/L)</th>
<th>Wet Chemistry (MG/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1,2-Dichloroethane</td>
<td>2-Methylnaphthalene</td>
<td>Iron, Dissolved</td>
<td>Nitrate Persulfate</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.5 µ</td>
<td>0.1 µ</td>
<td>0.5 µ</td>
<td>0.5 µ</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1,2-Dichloropropane</td>
<td>bis(2-Ethylhexyl)phthalate</td>
<td>Manganese, Dissolved</td>
<td>Sulfate</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.5 µ</td>
<td>0.1 µ</td>
<td>0.5 µ</td>
<td>NA</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Benzene</td>
<td>Naphthalene</td>
<td></td>
<td>TOC</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.5 µ</td>
<td>0.1 µ</td>
<td></td>
<td>0.5 µ</td>
</tr>
</tbody>
</table>

Notes:
- Bold indicates detections
- Bolded shading indicates detected exceedance
- Values in parentheses indicate values that vary less than 0.7 µg/L
- * - Not analyzed
- NA - Not analyzed
- JK - Below reporting limit and/or validated data
- J - Estimated (unvalidated data)
- R - Unreliable result
- U - Nondetect or not detected at significantly greater than that in an associated blank
- UJK - Nondetect. Estimated reporting limit
- MG/L - Milligrams per liter
- UG/L - Micrograms per liter

**TABLE 5**
Analytical Results for COCs, Dissolved Iron and Manganese, and Select Wet Chemistry Parameters for AOC I
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico
### TABLE 6
**Pilot Study Data Evaluation at MW-07**
*AOC I In-Situ Remediation Pilot Study Report*
*Former Naval Ammunition Support Detachment Vieques, Puerto Rico*

<table>
<thead>
<tr>
<th>COCs</th>
<th>Pilot Study PRG</th>
<th>Prior to Pilot Study</th>
<th>During Pilot Study</th>
<th>Overall</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>5</td>
<td>59.3</td>
<td>14</td>
<td>76%</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>27</td>
<td>82.1</td>
<td>17.0</td>
<td>79%</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>6.1</td>
<td>81.4</td>
<td>21</td>
<td>74%</td>
</tr>
</tbody>
</table>

*Notes:*

- for non-detects, half the reporting limit was used in the percent COC reduction calculation
# TABLE 7

**Human Health Risk Calculations, 2012 Sampling Events**

*AOC I In-Situ Remediation Pilot Test Report*

*Former Naval Ammunition Support Detachment*

*Vieques, Puerto Rico*

<table>
<thead>
<tr>
<th>CAS Number</th>
<th>Chemical</th>
<th>Maximum Concentration</th>
<th>Qual</th>
<th>Units</th>
<th>Location of Maximum</th>
<th>Data</th>
<th>EPC</th>
<th>Statistic</th>
<th>ELCR</th>
<th>HQ</th>
<th>Target Organs</th>
</tr>
</thead>
<tbody>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>2.9</td>
<td>ug/L</td>
<td>VWAI-MW07</td>
<td>5/23/2012</td>
<td>2.9 Max</td>
<td>7.40E-06</td>
<td>1.00E-01</td>
<td>Blood, Immune</td>
<td></td>
<td></td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>11</td>
<td>ug/L</td>
<td>VWAI-MW05</td>
<td>11/28/2012</td>
<td>11 Max</td>
<td>4.00E-01</td>
<td>1.00E-01</td>
<td>Lungs</td>
<td></td>
<td></td>
</tr>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>3.3</td>
<td>ug/L</td>
<td>VWAI-MW07</td>
<td>5/23/2012</td>
<td>3.3 Max</td>
<td>2.40E-05</td>
<td>5.00E-01</td>
<td>Decreased Body Weight</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Total = 3.00E-05

- Total Blood HI Across All Media = 1.00E-01
- Total Immune System HI Across All Media = 1.00E-01
- Total Lungs HI Across All Media = 4.00E-01
- Total Body Weight HI Across All Media = 5.00E-01
Figures
FIGURE 1
Regional Location Map
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment, Vieques, Puerto Rico
FIGURE 3
1994 Aerial Photograph of AOC I
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment, Vieques, Puerto Rico
LEGEND
- Approximate Area of AOC I
- Wooded Area
- Monitoring Well
- ISCO injection/Monitoring Well

FIGURE 4
Well Location Map
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico
FIGURE 6
AOC I Conceptual Site Model
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico

Legend
- Gravel with Sand, Silt, and Clay
- Fractured Andesite
- Surface water flow direction
- Direction of groundwater flow
- Infiltration and leaching

Not to Scale
<table>
<thead>
<tr>
<th>MW01</th>
<th>MW02</th>
<th>MW03</th>
<th>MW04</th>
<th>MW05</th>
<th>MW06</th>
<th>MW07</th>
<th>MW08</th>
<th>MW09</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>5</td>
<td>9.6</td>
<td>0.56</td>
<td>0.56</td>
<td>0.56</td>
<td>0.56</td>
<td>0.56</td>
<td>0.56</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>9.6</td>
<td>0.56</td>
<td>9.6</td>
<td>0.56</td>
<td>9.6</td>
<td>0.56</td>
<td>9.6</td>
<td>0.56</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
<td>0.1</td>
<td>0.56</td>
</tr>
<tr>
<td>Bis(2-Ethylhexyl)phthalate</td>
<td>6</td>
<td>10U</td>
<td>6</td>
<td>10U</td>
<td>6</td>
<td>10U</td>
<td>6</td>
<td>10U</td>
</tr>
</tbody>
</table>

**Exceeds PRG**

**FIGURE 7**

AOC I Pre-Pilot Study Groundwater Analytical Results for COCs

AOC I In-Situ Remediation Pilot Study Report

Former Naval Ammunition Support Detachment

Vieques, Puerto Rico
FIGURE 8
AOC I Potentiometric Map, November 1, 2010
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico

LEGEND
- Approximate Area of AOC I
- Wooded Area
- Monitoring Well
- Groundwater Elevation ft msl on November 1, 2010
- Groundwater Flow Direction
- Groundwater Elevation Contour, 0.5 ft Interval

North

0 25 50 Feet

ES0000513103834TPA_FB-Potentiometric_Map_Nov1-10-rev1.ai
FIGURE 9
AOC I Potentiometric Map, November 9, 2011
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico
FIGURE 10
AOC I Potentiometric Map, May 22, 2012
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico

LEGEND
- Approximate Area of AOC I
- Wooded Area
- Monitoring Well
- Groundwater Elevation ft msl on May 22, 2012
- Groundwater Flow Direction
- Groundwater Elevation Contour, 0.5 ft Interval
  Dashed where Inferred

North

0 25 50 Feet
FIGURE 11
AOC I Potentiometric Map, November 27, 2012
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico
FIGURE 12
Benzene Concentration Over Time
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment
Vieques, Puerto Rico
FIGURE 13
Details of Benzene Concentration over Time
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment Vieques, Puerto Rico

U values are the limit of detection, actual concentration is less than or equal to that value.
FIGURE 14
Naphthalene Concentration over Time
AOC I In-Situ Remediation Pilot Study Report
Former Naval Ammunition Support Detachment Vieques, Puerto Rico

Note: A pilot study PRG of 1.4 μg/L was originally selected solely to represent a conservative screening value to evaluate the technical implementability and effectiveness of the proposed pilot study technology. The value of 6.1 μg/L is hazard index based, using the December 2012 RSL for tap water for non-carcinogenic endpoints, and is more appropriate to use as a PRG.
U values are the limit of detection, actual concentration is less than or equal to that value.

Note: A pilot study PRG of 1.4 μg/L was originally selected solely to represent a conservative screening value to evaluate the technical implementability and effectiveness of the proposed pilot study technology. The value of 6.1 μg/L is hazard index based, using the December 2012 RSL for tap water for non-carcinogenic endpoints, and is more appropriate to use as a PRG.
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT:** In-Situ Remediation Pilot Study  
**LOCATION:** AOC-4  
**DATE:** 04/19/10

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Depth</td>
<td>45.80 ft (BTOC) Measured</td>
</tr>
<tr>
<td>Depth to Water</td>
<td>24.10 ft (BTOC) Measured</td>
</tr>
<tr>
<td>Water Volume in Well</td>
<td>21.47 ft</td>
</tr>
<tr>
<td>Pump Depth</td>
<td>90.5 ft (BTOC) Measured</td>
</tr>
<tr>
<td>Purge Device/Equipment</td>
<td>SS Monsoon Pump</td>
</tr>
<tr>
<td>Measuring Device/Equipment</td>
<td>Oil/Water Interface Probe</td>
</tr>
</tbody>
</table>

**SAMPLE INFORMATION**

- **Sample ID:** VMAI - MW02 - 0310
- **Parameters Collected for:** Vol, SVOC, FID, Fe(II), Mn, NOx, TOC
- **Sample Date/Time:** 03/18/10 09:40
- **Field Dup:** N/A
- **FD Sample Date/Time:** N/A
- **MS/MSD:** N/A
- **Sample Appearance:** Clear, colorless
- **Field Test Kit Details:** Oxy-Reduct @ 0/F = 1
- **Sample Collection Begins:** 10:40
- **Sample Collection Ends:** 10:14
- **Total Purge Volume:** 5.5 gallons

**FIELD PARAMETERS**

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gas)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp, (°C)</th>
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Signature: [Signature]  
Date: 04/19/10
# GROUNDWATER SAMPLING DATA SHEET

**PROJECT:** In-Situ Remediation Pilot Study

**LOCATION:** AOC-I

**DATE:** 5/18/10

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<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (us/cm)</th>
<th>Salinity (ppm)</th>
<th>DO (mg/L)</th>
<th>pH</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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**Signature:**

**Date:** 5/18/10
**NOTES (CONTINUED)**

SOP(s) used (refer to SOPs in back of this log)?

> YES

Were all requirements of the SAP, PIs and above mentioned SOP(s) met?

> YES

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

> TV 24:19 immediately after dropping pump.

---

**PHOTO LOG**

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Date: 03-18-10
# GROUNDWATER SAMPLING DATA SHEET

**PROJECT**: In-Situ Remediation Pilot Study  
**LOCATION**: AOC  
**DATE**: 3/19/10

**Weather**: Partly cloudy, humid, L-83°, H-93°

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<th>Total Depth:</th>
<th>39 ft. (BTOC) Measured</th>
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<td>Water Column (ft):</td>
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<td>Measuring Device/Equipment:</td>
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**Sample Team**: Dan Montgomery  
**Date and Time On Well**: 3/19/10 07:55  
**Date and Time Off Well**: 3/19/10 08:35

**Sample ID**: VWAI-MW03-0310  
**Sample Date/Time**: 03-19-10 09:10  
**Field ID**: VWAI-MW03-0310  
**Field Sample Date/Time**: 3/19/10 09:10

**Parameters Collected for**: VOCs, SVOCs, Fecal E. coli (E. coli)

**Parameters Collected for**: (FD) VOCs, SVOCs

**Sample Appearance**: Clear colorless

**Field Test Kit Details**: Omp & Qt F=1

## SAMPLE INFORMATION

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<th>Salinity (pH)</th>
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**Signature**:  
**Date**: 3/19/10
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<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (μS/cm) w/in 3%</th>
<th>Salinity (ppt)</th>
<th>DO (%</th>
<th>pH (mg/L) w/in 10%</th>
<th>ORP (mV) w/in 10mV</th>
<th>Turbidity (NTU) w/in 10%</th>
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Signature: [Signature]
Date: 3/9/10
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)?

Were all requirements of the SAP, PIs and above mentioned SOP(s) met?

Explanation of exceptions to SAP, PIs and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

No exceptions. (Map will)

PHOTO LOG

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<th>Photo Number</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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Signature: [Signature]

Date: 3/19/10
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT:** In-Situ Remediation Pilot Study  
**LOCATION:** AO4  
**DATE:** 5/17/00

| Sample Team: | D. Unitas  
|--------------|-------------|

**Weather:** mostly sunny, in 90°F, breezy, humid

| Total Depth: | 41.50 FT. (BTOC) Measured  
|--------------|-----------------------------|
| Depth to Water: | 24.63 FT. (BTOC) Measured  
| Water Column(h): | = 18.37 FT. IN  
| Water Volume in Well: | GAL (3.14159265*(in)^3)*(wells/4))  
| Pump Depth: | 36.7 FT. (BTOC) Measured  
| Measuring Device/Equipment: | Oil/Water Interface Probe  

**Sample Information**

| Sample ID: | VWAI-MW04-C14C  
|------------|------------------|
| Sample Date/Time: | 03/14/00  
| Field Dup: | YES  
| FD Sample Date/Time: | N/A  
| MS/MSD: | YES  
| Were samples filtered? | NO  
| Sample Appearance: | Clear  
| Field Test Kit Details: | 0% Fe = 1  
| If YES, Which samples? | Metals (Frac)  

**Parameters Collected:**

- **VOCs**
- **SVOs**
- **Filtered (For Mn)**
- **SO4, NO3, TOC**
- **(FD)**

**Field Parameters**

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<th>Flow Rate (gpm)</th>
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**Signature:**

| Date: | 5/17/00 |

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**Total Purge Volume:** 31.5 GAL
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<th>Temp. (°C)</th>
<th>SpCond (us/cm)</th>
<th>Salinity (ppt)</th>
<th>DO (gr/L)</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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Date: 3/19/10
### NOTES (CONTINUED)

- **SOP(s) used (refer to SOPs in back of this log)?**: Yes

- **Wore all requirements of the SAP, PI’s and above mentioned SOP(s) met?**: Yes

- **Explanation of exceptions to SAP, PI’s and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:**
  
  No exceptions (belt well)

### PHOTO LOG

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Signature: [Signature]

Date: 5/19/10
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT:** In-Situ Remediation Pilot Study  
**LOCATION:** AOC-1  
**DATE:** 03-16-10

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<td>(BTOC) Measured</td>
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| Water Column(h): (+) 20.29 FT | Date and Time On Well: 03/16/10 10:44 |
| Water Volume in Well:         | Pump Start Date and Time: 03/16/10 12:40 |
| Water Column(h):              | Pump Finish Date and Time: 03/16/10 12:45 |
| Pump Depth: 37.50 FT. (BTOC)  |                          |
| Purge Device/Equipment:       |                          |
| Measuring Device/Equipment:   |                          |

- **LOCATION:** AOC-1  
- **DATE:** 03-16-10

### SAMPLE INFORMATION

- **Sample ID:** VWAI-MW05-0310
- **Parameters Collected for:** TOC, SVOCs, FIP, Metals, Nitrate, Sulfate, TOC
- **Field Dup:** YES
- **FD Sample Date/Time:** N/A
- **MS/MSD:** YES
- **Sample Appearance:** clear, colorless
- **Field Test Kit Details:** prestan test kit, CF = 1
- **Were samples filtered?** Yes
- **If YES, Which samples?** bureaust water

### FIELD PARAMETERS

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**Start collecting GW sample**

**Signature:** D Whittaker  
**Date:** 03-16-10
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<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (uScm) w/in 2%</th>
<th>Salinity (%/ppt)</th>
<th>DO (mg/L) w/in 10%</th>
<th>pH w/in 0.1</th>
<th>ORP (mV) w/in 10mV</th>
<th>Turbidity (NTU) w/in 10%</th>
<th>Color / Odor / Comments</th>
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Signature: [Signature]

Date: [3/8/10]
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)?

Were all requirements of the SAP, PI's and above mentioned SOP(s) met?

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

- TU 24-00 currently other activities going on which will likely affect the outcome. We attempted to sample but sample may be contaminated.
- We had previously noted that unsampled. Our plan is to sample at some point. This should be acceptable.

PHOTO LOG

<table>
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<th>Photo Number</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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Signature: [Signature]  Date: [3/19/10]
**GROUNDWATER SAMPLING DATA SHEET**

**TOTAL DEPTH**
- 45.70 ft (BTDC) Measured
- 25.01 ft (BTDC) Measured

**WATER COLUMN:**
- 20.16 ft

**PUMP DEPTH:**
- 40 ft (BTDC) Measured

**MEASURING DEVICES/ENVIRONMENT:**
- Oil/Water Interface Probe

**SAMPLE INFORMATION**
- **Sample Date/Time:** 03/22/10 0750
- **Parameters Collected:**
  - VOCs, SWOCs, Filt Metals, nitrate
  - sulfates, TOC
- **Reference:** Wa Measured Date and Time Off Well:
  - 03/22/10 1048

**FIELD PARAMETERS**

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<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
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<th>Temp. (°C)</th>
<th>SpCond (uS/cm)</th>
<th>Salinity (ppt)</th>
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<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (μScm) with 3%</th>
<th>DO (%)</th>
<th>DO (mg/L) with 10%</th>
<th>pH with 0.1</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU) with 10%</th>
<th>Color / Odor / Comments</th>
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</thead>
<tbody>
<tr>
<td>0941</td>
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<td>75</td>
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</table>
SOP(s) used (refer to SOPs in back of this log)?

Were all requirements of the SAP, PI's and above mentioned SOP(s) met?

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

Dilution exists 0.2' with 70 m/h flow later.
### Groundwater Sampling Data Sheet

**Project:** In-Situ Remediation Pilot Study  
**Location:** AOC  
**Date:** 11/3/10

**Weather:** Overcast, 70°F, Humid

**Total Depth:** 44.8 ft (BTG)

**Depth to Water:** 20.27 ft (BTG)

**Water Column:** 24.0 ft

**Water Volume in Well:** 0.004329 GAL

**Pump:** SS Monsoon Pump

**Measuring Device:** Oil/Water Interface Probe

**Sample ID:** VWAI-MW02-1110

**Sample Dup/Time:** 11/3, 0745

**Field Date/Time:** 11/3, 0805

**Pump Depth:** 39.8 ft (BTG)

**Purge Device:** SS Monsoon Pump

**Air Monitoring Readings:** 0.0 ppm

**Date and Time On Well:** 11/3, 0845

**Date and Time Off Well:** 11/3, 10:20

**Depth to Water:** 20.27 ft (BTG)

### Sample Information

**Parameters Collected for:**  
- WQC (3 DIFF PRESERVE) SWGC  
- Filt 120u/M, NITRATE/SULFATE/TOC

**Sample Appearance:** Clear

**Field Test Kit Details:** 14 ppm PERSULFATE @ 09:20

**Color / Odor / Comments:** Pale Yellow

### Field Parameters

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (ppm)</th>
<th>Salinity (ppt)</th>
<th>DO (%)</th>
<th>DO (mg/L) with 10%</th>
<th>pH</th>
<th>ORP (mV) with 1000</th>
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**Signature:** Chris Buell  
**Date:** 11/3/10
<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol.</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (μS/cm)</th>
<th>Salinity (ppm)</th>
<th>DO (%)</th>
<th>DO (mg/L)</th>
<th>pH</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
</tr>
</thead>
</table>

Signature: [Signature]
Date: 11/3/10
SOP(s) used (refer to SOPs in back of this log)? B-1

Were all requirements of the SAP, PI's and above mentioned SOP(s) met? YES

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

NO EXCEPTIONS

PHOTO LOG

<table>
<thead>
<tr>
<th>Photo Number</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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</thead>
</table>

Signature: [Signature]

Date: 11/3/10
# Groundwater Sampling Data Sheet

**Project:** In-Situ Remediation Pilot Study  
**Location:** AO-C  
**Date:** 11/4/2010

**Weather:** Mostly Cloudy, a few drizzle or two

**Sample Team:** R. Buthe, C. Reed

<table>
<thead>
<tr>
<th>Total Depth</th>
<th>Date and Time On Well</th>
<th>Water Column</th>
<th>Pump Start Date and Time</th>
<th>Water Volume in Well</th>
<th>Pump Depth</th>
<th>Date and Time Off Well</th>
<th>Measuring Device/Equipment</th>
</tr>
</thead>
</table>

## Sample Information

- **Sample ID:** VWAI-MW03-1110 & 1110A
- **Parameters Collected for:** Vc, S, E, F, Nh, H, J, H, K, L
- **Parameters Collected for:** (FD)
- **Sample Appearance:** Clear
- **Field Test Kit Details:** 0-0.7 ppm Phosphate, 0-2.0 ppm Chloride

## Field Parameters

<table>
<thead>
<tr>
<th>Time</th>
<th>Purge Vol. (gpm)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (gpm/min)</th>
<th>Temp. (°C)</th>
<th>Salinity (ppm)</th>
<th>DO (%)</th>
<th>pH</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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<tr>
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*Note: Sample ID: VWAI-MW03-1110 & 1110A*

**Signature:** [Signature]  
**Date:** 11/4/2010
<table>
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<th>Time</th>
<th>Poured Vol. (gals)</th>
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<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (dS/cm) with 3%</th>
<th>Salinity (ppm)</th>
<th>DO (%)</th>
<th>DO (mg/L) with 10%</th>
<th>pH with 0.1</th>
<th>ORP (mV) with 50mV</th>
<th>Turbidity (NTU) with 10%</th>
<th>Color / Odor / Comments</th>
</tr>
</thead>
</table>

Signature: ______________________ Date: 11/4/2010
**NOTES (CONTINUED)**

SOP(s) used (refer to SOPs in back of this log)?  
B-1  

Were all requirements of the SAP, PIs and above mentioned SOP(s) met?  
Yes  

Explanation of exceptions to SAP, PIs and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:  
No Exception  

**PHOTO LOG**

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<th>Time</th>
<th>Description</th>
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Signature: [Signature]  
Date: 11/4/2010
GROUNDWATER SAMPLING DATA SHEET

PROJECT: In-Situ Remediation Pilot Study
LOCATION: AOC-I
DATE: 11/2/2010

Weather: Sunny, few clouds, wind, aid S8
Sample Team: K. Butler

Total Depth: FT, (BTG) Measured
Depth to Water: 20.72 FT, (BTG) Measured
Water Column (ft): - FT, IN.
Water Volume in Well: 36.5 GAL, (3.411593*h(s)=(wellDia/2))²*π*0.004329
Pump Depth: 36.5 FT, (BTG) Measured
Purge Device/Equip: SS Monsoon Pump
Measuring Device/Equipment: Oil/Water Interface Probe

SAMPLE INFORMATION
Sample ID: VWAI-MW04 - 1110 & 1110A
Parameters Collected for: VEC, SsCs, M765, (Fe, Mo)
Sample Date/Time: 11/2/2010 02:41 AM
FD Sample Date/Time: -
MS/MSD: YES
Sample Appearance: Clear
Field Test Kit Details: Between 0.07 & 0.14 on ProsKit

FIELD PARAMETERS

<table>
<thead>
<tr>
<th>Time</th>
<th>Purge Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (us/cm)</th>
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<th>DO (%)</th>
<th>DO (mg/L)</th>
<th>pH</th>
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<th>Color / Odor / Comments</th>
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<td>6.96</td>
<td>106.9</td>
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</table>

Soda in bottle, visible through kit, slow flush. Between 0.07 & 0.14 on ProsKit.
Any Ascorbic acid will be added to sample VWAI-MW04-1110A.
<table>
<thead>
<tr>
<th>FIELD PARAMETERS</th>
<th>LOCATION: ADC1</th>
</tr>
</thead>
<tbody>
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**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT**

In-Situ Remediation Test Study

**PROJECT NUMBER**

392455 FL FK

**WELL NUMBER**

VWAL-MW04

**SHEET 2 OF 3**

**DATE**

11/2/2010
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)?

Yes

Were all requirements of the SAP, PI's and above mentioned SOP(s) met?

Yes

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

No exceptions made

PHOTO LOG

<table>
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<tr>
<th>Photo Number</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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Signature: [Signature]

Date: 11/2/2010
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT NUMBER**: 392485.FI.FK  
**WELL NUMBER**: VWAI-MW05  
**SHEET 1 OF 3**

**LOCATION**: ADC4  
**DATE**: 11/2/10

**Weather**: Sunny, 85°F, Humid  
**Sample Team**: Christopher Reed, Kenji Butler

**Total Depth**

<table>
<thead>
<tr>
<th>Depth to Water (FT, (BT)OC) Measured</th>
<th>Date and Time On Well</th>
</tr>
</thead>
<tbody>
<tr>
<td>44.6 FT, (BT)OC Measured</td>
<td>11/2, 0815</td>
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</tbody>
</table>

**Depth to Water**: 44.6 FT, (BT)OC Measured  
**Water Column (h)**: 2.065 FT, (BT)OC Measured  
**Water Volume in Well**: GAL (3.141593*h*(in)/12)/2 = 0.034329

**Pump Depth**

<table>
<thead>
<tr>
<th>Pump Depth (FT, (BT)OC Measured)</th>
<th>Date and Time Off Well</th>
</tr>
</thead>
<tbody>
<tr>
<td>39.6 FT, (BT)OC Measured</td>
<td>11/2, 0920</td>
</tr>
</tbody>
</table>

**Purge Device/Equipment**: SS Monsoon Pump  
**Air Monitoring Readings**: 0.0 ppm

**Measuring Device/Equipment**: Oil/Water Interface Probe  
**Total Purge Volume**: 4.0 GAL

**SAMPLE INFORMATION**

**Sample ID**: VWAI-MW05 - 1110  
**Parameters Collected for**: Cl, S, B, EN, D, Fe, SDT, NO3, NO2, NITRATES, TOC

**Sample Date/Time**: 11/2/10, 0920  
**Field Dup**: YES/NO: NO  
**FD Sample Date/Time**:  
**MS/MSD**: YES/NO: NO  
**Sample Appearance**: CLEAR

**Fields**

<table>
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<tr>
<th>Sample</th>
<th>Water Column</th>
<th>Depth to Water</th>
<th>Flow Rate</th>
<th>Temp</th>
<th>SpCond (vcm)</th>
<th>Salinity (ppt)</th>
<th>DO (mg/l)</th>
<th>DO (wt %)</th>
<th>pH</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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<tbody>
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<td>1328</td>
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<td>Slight Petro Oder</td>
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<td>1364</td>
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<td>6.73</td>
<td>34.1</td>
<td>12.2</td>
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</table>
| 0845   | 1.75         | 21.32          | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/
| 0850   | 1.50         | 21.32          | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/ |
| 0855   | 2.00         | 21.32          | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/ |
| 0900   | 2.00         | 21.32          | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/ |
| 0905   | 2.75         | 21.32          | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/ |
| 0910   | 3.25         | 21.32          | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/ |
| 0915   | 3.50         | 21.32          | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/ |
| 0920   | Collect Sample | 21.32    | 250       | 79.94| 1362         | 0.08          | 4.0       | 0.34      | 6.73 | 32.6     | 8.78            | 0.0 ppm Pesticides w/ |

**Signature**: Christopher Reed  
**Date**: 11/2/10
<table>
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<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (uS/cm) with 3%</th>
<th>Salinity (ppt)</th>
<th>DO (%)</th>
<th>DO (mg/L) with 10%</th>
<th>pH with 0.1</th>
<th>ORP (mv) with 10mV</th>
<th>Turbidity (NTU) with 10%</th>
<th>Color / Odor / Comments</th>
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<tbody>
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</tbody>
</table>
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)?  B-1
Were all requirements of the SAP, PIs and above mentioned SOP(s) met?  YES

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

No Exceptions

PHOTO LOG

<table>
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<tr>
<th>Photo Number</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
</tr>
</thead>
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Signature: [Signature]  Date: 11/2/10
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT NUMBER:** 392485.FL.FK  
**WELL NUMBER:** VVWI-MW07  
**DATE:** 11/4/10  
**LOCATION:** AOC-4

**Sample Team:** Chris Reed  
**Instrument:** SS Monsoon Pump  
**Measuring Device:** Oil/Water Interface Probe

---

**SAMPLE INFORMATION**

- **Sample ID:** VVWI-MW07-1110  
- **Parameters Collected for:** VOC (2 DIFF PRESERVE), SVOC, TIC
- **Sample Date/Time:** 11/4, 10:20  
- **Field Date/Time:** 11/4, 10:20  
- **FD Sample Date/Time:** 11/4, 10:25  
- **Sample Appearance:** CLEAR
- **Parameters Collected for:** FF, SVOC, NO
- **Field Test Kit Details:** 14-21 ppm PERSULFATE @ 10.5% MV PERSERVE W/ 2.5% ASCORBIC ACID

---

**FIELD PARAMETERS**

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<th>Salinity (ppt)</th>
<th>DO (mg/L)</th>
<th>pH</th>
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**Total Purge Volume:** 45 GAL

**Signature:** [Signature]  
**Date:** 11/4/10
<table>
<thead>
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<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (μS/cm) w/ 3%</th>
<th>Salinity (ppm)</th>
<th>DO (mg/L w/ 10%)</th>
<th>pH w/ 0.1</th>
<th>ORP (mV) w/ 10mV</th>
<th>Turbidity (NTU) w/ 10%</th>
<th>Color / Odor / Comments</th>
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</table>

Signature: [Signature]
Date: 11/4/10
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)? B-1

Were all requirements of the SAP, PIs and above mentioned SOP(s) met? No

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

"Drawdown was greater than 0.3' initially, but began to recharge after 20 minutes of purging."

PHOTO LOG

<table>
<thead>
<tr>
<th>Photo Number</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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Signature: [Signature]

Date: 11/4/10
# GROUNDWATER SAMPLING DATA SHEET

**PROJECT NUMBER** 392485.FI.FK  
**WELL NUMBER** VWAI-MW02  
**DAT SHEET:** 1 OF 2

**PROJECT:** In-Situ Remediation Pilot Study  
**LOCATION:** AOC-I  
**DATE:** 11/10/11

**Weather:** Rbly, cloudy, with 80's  
**Sample Team:** K.B. No/VBO

**Total Depth:** 45.25 FT (BT) Measured  
**Depth to Water:** 15.75 FT (BT) Measured  
**Water Column(h):** 29.50 FT

**Water Volume in Well:** 10,415.93 (3.141592653) * (wellDia/2)^2 * 0.004329

**Pump Depth:** 40.25 FT (BT) Measured  
**Air Monitoring Readings:** 0 ppm

**Measuring Device/Equip:** YSI Prof Series, LaMotte 2020

## SAMPLE INFORMATION

<table>
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<tr>
<th>Sample ID</th>
<th>Parameters Collected for</th>
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<tbody>
<tr>
<td>Sample Date/Time</td>
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<td>Field Dup</td>
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<td>FD Sample Date/Time</td>
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<td>MS/MSD</td>
<td>Y</td>
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<td>Were samples filtered?</td>
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</table>

If YES, Which samples? Fe/Mn

## FIELD PARAMETERS

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gall)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (l/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (us/cm)</th>
<th>Salinity (ppm)</th>
<th>DO (%)</th>
<th>DO (mg/L)</th>
<th>pH</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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**Signature:**  
**Date:** 11/10/11
SOP(s) used (refer to SOPs in back of this log)? G-1

Were all requirements of the SAP, Pts and above mentioned SOP(s) met? Y/S

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

PHOTO LOG

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<th>Time</th>
<th>Description</th>
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<td>While precipitate coming out of the well</td>
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Signature: [Signature]
Date: 11/2/11
# GROUNDWATER SAMPLING DATA SHEET

**PROJECT:** In-Situ Remediation Pilot Study  
**WEll NUMBER:** VWAI-MW03  
**DATE:** 11/9/11

**WEather:** Overcast, Snow Rain, Humid, High 40°  
**Sample Team:** K. Butler/V80

<table>
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<tr>
<th>Total Depth</th>
<th>39.86 FT (BTGC) Measured</th>
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<tr>
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<td>Water Column(h)</td>
<td>31.74 FT.</td>
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<td>Water Volume in Well</td>
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<td>Pump Depth</td>
<td>32.86 FT (BTGC) Measured</td>
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<td>Mason Pump</td>
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<td>Measuring Device/Equipment</td>
<td>YES Professional Plus/EvMate</td>
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<td>Pump Finish Date and Time</td>
<td>11/9/11 10:52</td>
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<td>Pump Start Date and Time</td>
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<td>Date and Time On Well</td>
<td>11/9/11 10:52</td>
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<td>Air Monitoring Readings</td>
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<td>Total Purge Volume</td>
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## SAMPLE INFORMATION

**Sample ID:** VWAI-MW03-1111/VWAI-MW03-1111  
**Parameters Collected for:** VOCs, SVOCs, TPH(GRO, DOM, ORP)

**Sample Date/Time:** 11/9/11 11:55

**Field Dup:** Yes / No  
**FD Sample Date/Time:** 11/9/11

**MS/MSD:** Yes / No  
**Were samples filtered?** Yes / No

**Sample Appearance:** Clear  
**Field Test Kit Details:** Day/1: Sodium Peridate

## FIELD PARAMETERS

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<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (ml/min)</th>
<th>Temp (°C)</th>
<th>SpCond (us/cm)</th>
<th>Salinity (ppt)</th>
<th>DO (mg/L)</th>
<th>DO (%)</th>
<th>DO (mg/L)</th>
<th>pH</th>
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**Signature:**  
**Date:** 11/9/11
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)?  B-1

Were all requirements of the SAP, PIs and above mentioned SOP(s) met?  Yes

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

PHOTO LOG

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<th>Photo Numbe</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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Signature: [Signature]   Date: 11/9/11
## Groundwater Sampling Data Sheet

### Project Information
- **Project Number:** 392485.FI.FK
- **Well Number:** VWAI-MW04
- **Sheet:** 1 of 1

### Location and Date
- **Location:** AOC-1
- **Date:** 11/10/11

### Weather
- Mostly sunny, warm, dry

### Sample Information
- **Sample ID:** VWAT-MW04-1111
- **Sample Date/Time:** 11/10/11 10:45

### Field Duplication
- **Device/Equipment:** Monoeye pump
- **Date and Time On Well:** 11/10/11 08:00
- **Date and Time Off Well:** 11/10/11 10:10
- **Air Monitoring Readings:** 0:12pm
- **Total Purge Volume:** 0

### Field Parameters

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<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (μS/cm)</th>
<th>Salinity (ppt)</th>
<th>DO (%)</th>
<th>DO (mg/L)</th>
<th>pH (0.01)</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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### Signature
- **Signature:** [Signature]
- **Date:** 11/10/11
GROUNDBWATER SAMPLING DATA SHEET

PROJECT NUMBER 392485.FI.FK  
WELL NUMBER VWAI-MW04  
PROJECT: In-Situ Remediation Pilot Study  
LOCATION: AOC-1  
DATE: 1/10/11

NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)? E-1

Were all requirements of the SAP, PIs and above mentioned SOP(s) met? YES

Explanation of exceptions to SAP, PIs and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

PHOTO LOG

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<tr>
<th>Photo Number</th>
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<th>Time</th>
<th>Description</th>
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</thead>
</table>

Signature:  
Date: 1/10/11
**PROJECT NUMBER**

392485.FK

**WELL NUMBER**

VWAI-MW05

**GROUNDWATER SAMPLING DATA SHEET**

PROJECT: In-Situ Remediation Pilot Study  
LOCATION: ADC4 - NW - 95  
DATE: 11-08-11  
Sample Team: D. Whitaker/C. Vela

**Weather:** Cloudy / Warm

Total Depth: 44.66 ft (BTOC) Measured  
Depth to Water: 2.01 ft  
Water Column (h): (c)  
Water Volume in Well: 16.65 ft (BTOC) Measured  
Water Depth: 28.01 ft  
Pump Depth: 39.16 ft (BTOC) Measured  
Purge Device/Equipment: Monsoon Pump  
Measuring Device/Equipment: YSI Professional Plus

**SAMPLE INFORMATION**

Sample ID: VNAT-MW05 - III  
Sample Date/Time: 11-08-11 /09:35

Field Dup: YES  
FD Sample Date/Time: 11-09-11 /09:40

MS/MSD: YES  
Sample Appearance: clear, colorless

Were samples filtered? YES  
If YES, Which samples? All

**FIELD PARAMETERS**

<table>
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<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (ml/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (us/cm)</th>
<th>Salinity (ppt)</th>
<th>DO (%)</th>
<th>DO (mg/L) with 10%</th>
<th>pH with 0.1</th>
<th>ORP (mV) with 10%</th>
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<td>17.02</td>
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<td>1951</td>
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Signature: [Signature]  
Date: 11-08-11
**NOTES (CONTINUED)**

SOP(s) used (refer to SOPs in back of this log)?  
Yes B-1  

Were all requirements of the SAP, PI's and above mentioned SOP(s) met?  
No  

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

- A second set of VOCs that were for sample ID VWAJ - MW05 - 1141A  
  were prepared using our HCL from 2.40 mL vials and then per in appropriate  
  amount of ascorbic acid not completed because when  
  completed, chemists kit for sodium persulfate got 2.9ppm  
  and thus, thought the 2.40 mL ascorbic acid preserved vials  
  were not reused. Called lab and CH2M Hill chemist and  
  they also believed this, when later consulted PM, found drums to be unknown. Will collect tomorrow after complete re-purge to  
  sample VOCs (HCl preserved) and VOCs (ascorbic acid preserved)  
  and the field duplicate samples as well for VOCs (HCl pres) and  
  VOCs (ascorbic acid pres.).

**PHOTO LOG**

<table>
<thead>
<tr>
<th>Photo Numbe</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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<td></td>
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</tbody>
</table>

Signature: [Signature]  
Date: 11/08/11
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT:** In-Situ Remediation Pilot Study  
**LOCATION:** AO-C4  
**DATE:** 11/9/11

---

**Weather:** Overcast, Some Rain, Humid, Mid 80's  
**Sample Team:** K. Butcher, T. Kruse, C. Veil, T. Pa

---

**Total Depth:** 44.66 ft (BTCC) Measured  
**Depth to Water:** (-) 16.66 ft (BTCC) Measured  
**Water Column:** (m) 24.00 ft  
**Water Volume in Well:** GAL (3.141593*π*[wellDia/2]^2)*0.004329

---

**Pump Depth:** 39.60 ft (BTCC) Measured  
**Purge Device/Equipment:** Meter Pump  
**Measuring Device/Equipment:** YSI Portable pH/DO Date 2002

---

**SAMPLE INFORMATION**

**Sample ID:** VWA1-MW05B-111/0VWA1-MW05B-111  
**Parameters Collected for:** V0Cs, V0s (Acetic Acid)  
**Sample Date/Time:** 11/9/11 0900  
**Field ID:** VWA1-MW05B-111/0VWA1-MW05B-111  
**Parameters Collected for:** (FD) V0Cs, V0s (Acetic Acid)  
**FD Sample Date/Time:** 11/9/11 0905  
**MS/MSD:** YES/NO  
**Were samples filtered?** YES/NO  
**If YES, Which samples?** NA  
**Sample Appearance:** Clear

---

**FIELD PARAMETERS**

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (ml/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (µS/cm) with 3%</th>
<th>Salinity (ppm)</th>
<th>DO (mg/L) with 10%</th>
<th>DO (mg/L) with 10%</th>
<th>pH</th>
<th>ORP (mV) with 10mV</th>
<th>Turbidity (NTU) with 10%</th>
<th>Color / Odor / Comments</th>
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<td>7.1</td>
<td>6.71</td>
<td>-4.0</td>
<td>19.2</td>
<td></td>
<td>Clear, Slightly Positive</td>
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</tbody>
</table>

---

**Signature:**  
**Date:** 11/9/11
SOP(s) used (refer to SOPs in back of this log)? B-1
Were all requirements of the SAP, PIs and above mentioned SOP(s) met? Yes
Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

PHOTO LOG

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<th>Description</th>
</tr>
</thead>
</table>

Signature: [Signature]
Date: 11/4/11
GROUNDWATER SAMPLING DATA SHEET

WEATHER: overcast, drizzling, humid ~ 80 °F

Sample Team: D. Whitaker

Total Depth: 34.50 FT (BTOC) Measured

Depth to Water: 16.92 FT (BTOC) Measured

Water Column(h): 17.58 FT. IN.

Water Volume in Well: 435.04 GAL (3.141593*π*wellDia/2)^2*0.004326 Pump Finish Date and Time: 11-09-11 1050

Pump Depth: 29.50 FT (BTOC) Measured

Date and Time Off Well: 11-09-11 1100

Purge Device/Equip: Monsoon Pump

Air Monitoring Readings: 0.0 ppm

Measuring Device/Equipment: YSI Prof Scr, LaNite 2020

Total Purge Volume: 1,765 GAL (1.25)

SAMPLE INFORMATION

Sample ID: VWAI-MW07-11 II

Parameters Collected for: (FD) H2S, (FD) NO3, (FD) ORP

Sample Date/Time: 11-09-11 0920

Field Dup: YES ID: 11-09-11

FD Sample Date/Time: N/A

MS/MSD: YES NO

Sample Appearance: Color: None, Odor: None

Were samples filtered? YES

If YES, Which samples? Filter

FIELD PARAMETERS

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (gpm)</th>
<th>Temp. (°C)</th>
<th>SpCond (μS/cm)</th>
<th>Salinity (ppt)</th>
<th>DO (%)</th>
<th>DO (mg/L)</th>
<th>pH</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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<td>cut off pump to let recirculate, check pH and FC, later not enough flow in well.</td>
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<td>31.59</td>
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<td>72.1</td>
<td>63.9</td>
<td>13.7</td>
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<tr>
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<td>153.0</td>
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<td>12.30</td>
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<td>34.8</td>
<td>34.8</td>
<td>cut off pump to let recirculate, check pH and FC, later not enough flow in well.</td>
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Signature: D. Whitaker
Date: 11-09-11
SOP(s) used (refer to SOPs in back of this log)?  B-1

Were all requirements of the SAP, PI's and above mentioned SOP(s) met?  Yes

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

PHOTO LOG

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<tr>
<th>Photo Number</th>
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<th>Time</th>
<th>Description</th>
</tr>
</thead>
</table>

Signature: [Signature]  Date: 11/09/11
# GROUNDWATER SAMPLING DATA SHEET

**PROJECT**: In-Situ Remediation Pilot Study  
**LOCATION**: AOC-1  
**DATE**: 05/23/12

**Weather**: 75°F, Sunny

**Total Depth**: 40.58 FT (BTTOC) Measured

**Depth to Water**: 18.22 FT (BTTOC) Measured

**Water Column (h)**: 22.33 FT

**Water Volume in Well**: 3.64 GAL (3.141593*(π)*(well Dia/2)^2) * 0.004325

**Date and Time On Well**: 05/23/12 07:30

**Date and Time Off Well**: 05/23/12 08:50

**Measuring Device/Equipment**: YSI 556 MPS High Accuracy

<table>
<thead>
<tr>
<th>Time</th>
<th>Purge Vol (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (gpm)</th>
<th>Temp, (°C)</th>
<th>SpCond (µS/cm) with 3%</th>
<th>Salinity (ppt)</th>
<th>DO (%)</th>
<th>DO (mg/L) win 10%</th>
<th>pH with 0.1</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU) with 10%</th>
<th>Color / Odor / Comments</th>
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</thead>
<tbody>
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<td>28.12</td>
<td>1351</td>
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**Sample ID**: VVAT - NO04 - 0512  
**Parameters Collected for**: V0Cs, SV0Cs, EMETALs (Fe, Mn)

**Field Sample Date/Time**: 05/23/12 08:25

**MS/MSO**: YES

**Sample Appearance**: Clean, colorless

**Wet samples filtered**: YES

**Per sulfate Test Kit Details**: 0.0 ppm

**Purge Device/Equip**: Air Monitoring Readings: 0.0 ppm

**Total Purge Volume**: 3.50 GAL

**Signature**: __________  
**Date**: 05/23/12

---

**FIELD PARAMETERS**

---

**Sample Team**: D. Whittaker  
**P. Murphy"
<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (pals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (us/cm) win 3%</th>
<th>Salinity (ppt)</th>
<th>DO (%)</th>
<th>DO (mg/L) win 10%</th>
<th>pH win 0.1</th>
<th>ORP (mV) win 10mV</th>
<th>Turbidity (NTU) win 10%</th>
<th>Color / Odor / Comments</th>
</tr>
</thead>
</table>

Signature: __________________________ Date: __________________________
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)? Yes

Were all requirements of the SAP, PI's and above mentioned SOP(s) met? Yes

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

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</tr>
</tbody>
</table>

Signature: [Signature]

Date: 05/23/12
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT:** In-Situ Remediation Pilot Study  
**LOCATION:** AOC-I  
**DATE:** 05/22/12

### Weather
- Sunny, humid, hot

### Sample Team
- Dan Whistler
- Noa Danovs
- Pat Murphy

### Total Depth
- 47.66 FT (BTOC) Measured

### Depth to Water
- 17.04 FT (BTOC) Measured

### Water Column高度
- 26.02 FT

### Water Volume in Well
- 1.24 GAL (3.141593 * radius^2 * height)

### Pump Depth
- 39.00 FT (BTOC) Measured

### Measuring Device/Equipment
- Submersible Pump (17922)

### Total Purge Volume
- 1.85 GAL

### Sample Information
- **Sample ID:** WAVI-MW05-0512
- **Field Dup:** Yes
- **FD Sample Date/Time:** 05/22/12 1055
- **MS/MSD:** Yes
- **Sample Appearance:** Clear, colorless
- **Persulfate Test Kit Details:** 0.0 ppm

### Field Parameters

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol (Gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp (°C)</th>
<th>SpCond (μS/cm) with 3%</th>
<th>Salinity (pF)</th>
<th>DO (%)</th>
<th>DO (mg/L) with 10%</th>
<th>pH</th>
<th>ORP (mV) with 10mV</th>
<th>Turbidity (NTU) with 10%</th>
<th>Color / Odor / Comments</th>
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**Signature:**

**Date:** 05/22/12

---

*Note: The table includes various water quality parameters measured during the sampling process.*
### GROUNDWATER SAMPLING DATA SHEET

**PROJECT**: In-Situ Remediation

**LOCATION**: ADC4

**DATE**

<table>
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<th>Time</th>
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<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (us/cm) with 3%</th>
<th>Salinity (ppm)</th>
<th>DO (mg/L) with 10%</th>
<th>pH with 0.1</th>
<th>ORP (mV) with 10%</th>
<th>Turbidity (NTU) with 10%</th>
<th>Color / Odor / Comments</th>
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**Signature** ____________________________  **Date**: ____________________________
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)?

Yes

Were all requirements of the SAP, PI's and above mentioned SOP(s) met?

Yes

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

PHOTO LOG

<table>
<thead>
<tr>
<th>Photo Numbe</th>
<th>Compass Direction</th>
<th>Time</th>
<th>Description</th>
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Signature: [Signature]

Date: 05/23/12
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT**: In-Situ Remediation Pilot Study  
**LOCATION**: AOC-4  
**DATE**: 05/31/12

- **Sample Team**: Dianna Dancis, Pat Murphy
- **Sample**: Water Column
- **Date and Time On Well**: 05/31/12 08:55
- **Date and Time Off Well**: 05/31/12 14:00

### Measuring Device/Equipment
- **YSI 556 MPS/Hand Turbidimeter**
- **Total Purge Volume**: 185 GAL.

### Sample Information
- **Sample ID**: WWA1-MW07-0512
- **Parameters Collected for**: VOCs, SVOCs, Fe/H2O

### Field Parameters

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gph)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
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<th>Salinity (ppt)</th>
<th>DO (%</th>
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**Signature**:  
**Date**: 05/31/12
# GROUNDWATER SAMPLING DATA SHEET

## FIELD PARAMETERS

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (us/cm)</th>
<th>Salinity (ppt)</th>
<th>DO (ppm)</th>
<th>pH</th>
<th>ORP (mV)</th>
<th>Turbidity (NTU)</th>
<th>Color / Odor / Comments</th>
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Signature: ___________________   Date: ___________________

---
NOTES (CONTINUED)

SOP(s) used (refer to SOPs in back of this log)?

Were all requirements of the SAP, PIs and above mentioned SOP(s) met?

Explanation of exceptions to SAP, PIs and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

While starting the initial purge of MW07 on AOC I, sampling team was unable to keep well down to within 0.3 ft as stated in the SOP. Well is being pumped at less than 125 gpm and recharge on well is insufficient to stop/holding down of water in well. Sampling team is unable to slow flow rate down since it will prompt any flow; will use all pumping pressure. Sampling team notified the PVI, S. Brand, who stated to continue as fast as possible. Will try and resolve flow down issue as fast as possible while adhering to the SOP. NOTE: Team is using or flow restriction and has spent about 45 minutes trying to resolve this issue.

DO is very low which is not sure within 10% at least 3 readings however DO is considered stabilized - the 5 mL/L reading was taken when the water had dropped - pump speed had to be turned up a little to decrease flow. Due to pumping at such low flow, volume/speed at times during sampling the pump had to be turned up a little at a time to keep the water purging.

PHOTO LOG

<table>
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<tr>
<th>Photo Number</th>
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<tr>
<td>2</td>
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<td>Par collecting groundwater sample</td>
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</table>

Signature: [Signature]
Date: 05/23/12
# Groundwater Sampling Data Sheet

**Project Number**: 392485.FL.FK  
**Well Number**: VWAI-MW04  
**Location**: AOI-1  
**Date**: 11/28/12

### Weather
- Sunny, Humid, Hot

### Sample Information
- **Sample ID**: VWAI - MW04 - 1112  
- **Sample Date/Time**: 11/28/12 09:05  
- **Field Dup**: YES/NO ID: NA  
- **FD Sample Date/Time**: NA  
- **MS/MSD**: YES/NO  
- **Persulfate Test Kit Details**: qo min  
- **Parameters Collected**: VOCs, SVOCs, SPE, TCLS, WCHM (SC, NO), WCHM (GC)

### Field Parameters

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (ml/min)</th>
<th>Temp. (°C)</th>
<th>SpCond (μS/cm) w/in 3%</th>
<th>Salinity (%)</th>
<th>DO (%)</th>
<th>DO (μgl/L) w/in 10%</th>
<th>ORP (mV) w/in 10mV</th>
<th>Turbidity (NTU) w/in 10%</th>
<th>Color / Odor / Comments</th>
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</tr>
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</table>

**Signature**:  
**Date**: 11/28/12
### GROUNDWATER SAMPLING DATA SHEET

**Project:** In-Situ Remediation Pilot Study  
**Location:** AOC-1  
**Date:** 11/28/12

**Weather:** Rain, Hot, Humid  
**Sample Team:** J. Horn

---

**Total Depth:** 49.55 ft. (BTOC) Measured  
**Depth to Water:** 25.12 ft. (BTOC) Measured  
**Water Column (h):** 19.00 ft.  
**Water Volume in Well:** 3.0 GAL (3.141593*hi*(wellDIA/2)^2)*0.00432 ft.  
**Pump Depth:** 2260 ft. (BTOC) Measured  
**Purge Device/Equipment:** Pancake pump  
**Measuring Device/Equipment:** YES

**Date and Time On Well:** 11/28/12 1015  
**Pump Start Date and Time:** 11/28/12 1030  
**Pump Finish Date and Time:** 11/28/12 1105  
**Date and Time Off Well:** 11/28/12 1130  
**Air Monitoring Readings:** N/A  
**Total Purge Volume:** ~ 3.5 GAL

---

**SAMPLE INFORMATION**

**Sample ID:** VWBT-MW05-112  
**Parameters Collected for:** VOCs, SUVC, Fredials

**Sample Date/Time:** 11/28/12 1125  
**Field Dup.:** N/A  
**MS/MSD:** N/A  
**Sample Appearance:** Clear  
***Were samples filtered?*** YES/NO  
***If YES, Which samples?*** Fredials

---

**FIELD PARAMETERS**

<table>
<thead>
<tr>
<th>Time</th>
<th>Purged Vol. (gals)</th>
<th>Depth to Water (ft)</th>
<th>Flow Rate (mL/min)</th>
<th>Temp. (°C)</th>
<th>SPCn (ppb/cm) w/3%</th>
<th>Salinity (mg/L)</th>
<th>DO (%)</th>
<th>DO (mg/L) w/10%</th>
<th>pH</th>
<th>ORP (mV) w/10%</th>
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**Date:** 11/28/12
**GROUNDWATER SAMPLING DATA SHEET**

**PROJECT NUMBER**: 392485.FI.FK  
**WELL NUMBER**: VWA1-MW07  
**LOCATION**: ADC-4  
**DATE**: 11/29/12

**Weather**: clear, sunny, real breeze, humid  
**Sample Team**: P.McCary, J.Hern

**Total Depth**: 45.12 FT.(BTG) Measured  
**Depth to Water**: 26.52 FT.(BTG) Measured  
**Water Column**: 14.70 FT.  
**Water Volume in Well**: 3.21 GAL (3.141593 h/in²)wellDia/2*2*0.04326  
**Pump Depth**: 40 FT.(BTG) Measured  
**Purge Device/Equip**: Vise Pump

**Date and Time On Well**: 11/24/12 0730  
**Pump Start Date and Time**: 11/24/12 0745  
**Date and Time Off Well**: 11/24/12 1000  
**Air Monitoring Readings**: N/A

**Total Purge Volume**: 25 GAL

---

**SAMPLE INFORMATION**

**Sample ID**: VWA1-MW07.P7.1112  
**Sample Date/Time**: 11/24/12. 0945  
**Parameters Collected for**: VOCs, Traces, Metals, Chloride  
**Field Dup. ID**: VWA1-MW07.P7.1112  
**Parameters Collected for Field**: VOCs, SVOCs  
**FD Sample Date/Time**: 11/24/12 0920  
**MS/MSD**: Yes  
**Were samples filtered**: Yes  
**Persulfate Test Kit Details**: 0.0 ppm  
**Sample Appearance**: Clear

---

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SOP(s) used (refer to SOPs in back of this log)? Yes

Were all requirements of the SAP, PI's and above mentioned SOP(s) met? Yes but with few exceptions

Explanation of exceptions to SAP, PI's and SOP(s) including why, under what conditions, who authorized exception, anything considered in the decision:

While starting the initial pump of MW07@ AOC 1, sampling team was unable to keep well down down within 0.3 ft as stated in SOPs. Well was pumped between 50l and ~ 110l. Flow rate kept changing due to pumping pressure changes from well water draw down. Well had insufficient exchange to allow steady flow rate and prevent draw down. Sampling team tried their best to prevent and minimize draw down and adhered to SOPs. Sample team supplied consistent with previous round of sampling (May 2013).

PHOTO LOG

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### Vieques AOC I Field Parameters - Summary Sheet

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<th>DO (mg/L)</th>
<th>Conductivity (mS/cm)</th>
<th>pH</th>
<th>Temperature (°C)</th>
<th>Turbidity (NTU) [optional]</th>
<th>Persulfate Concentration (mg/L)</th>
<th>Sulfate Test Kit (mg/L) [optional]</th>
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### DAY 3 - Monitoring during Injection

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<th>DO (mg/L)</th>
<th>Conductivity (mS/cm)</th>
<th>pH</th>
<th>Temperature (°C)</th>
<th>Turbidity (NTU) [optional]</th>
<th>Persulfate Concentration (mg/L)</th>
<th>Sulfate Test Kit (mg/L) [optional]</th>
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### DAY 4 - Monitoring during Injection

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<th>ORP (mV)</th>
<th>DO (mg/L)</th>
<th>Conductivity (mS/cm)</th>
<th>pH</th>
<th>Temperature (°C)</th>
<th>Turbidity (NTU) [optional]</th>
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### ISCO Injection Field Observation Form

**Injection Well: MW02**

**Site:** Vieques AOC-I  
**Project:** In Situ Activated Alkaline Sodium Persulfate Injection  
**Contract:** Navy CLEAN, CTO-83

---

#### Design Summary

- **Screen Interval:** 31 - 41 ft bgs
- **Total Solution Volume:** 514 gallons  
  (includes 20 gallons of chase water)
- **Persulfate Solution per Well:** 475 gal  
  Mass of Persulfate per Well = 209 lbs
- **Mass of NaOH per Well:** 200 lbs (19 gal; 25% solution)
- **Persulfate concentration:** 5%; 50 g/L

---

#### Date | Start Time | Stop Time | Pressure range (psi) | Flowrate (gpm) | Total Time (min) | Total Volume (gallons) | Notes
---|---|---|---|---|---|---|---
3/27/2010 | 0853 | 0910 | 10 | not registering | 17 | 66 | 66 gal batch injected (200 gallon batch simultaneously into MWs 2, 3, and 4). Flow readings not accurate, will switch to injecting into individual wells.
3/29/2010 | 1304 | 1330 | 0 | 2.7 | 43 | -- | 167 gal batch begin injecting; stop to refuel compressor
3/29/2010 | 1349 | 1356 | 0 | 3.0 - 3.2 | 50 | 233 | 167 gal batch injection complete
3/29/2010 | 1445 | -- | 0 | 1.23 | -- | -- | 200 gal batch begin injecting; gravity fed
3/29/2010 | 1510 | 1539 | < 0.5 - 4.5 | 3.6 | 104 | 383 | 150 gal of the 200 gallon batch injected, put other 50 gallons in MW-03
3/30/2010 | 0910 | 0934 | 0 | 3.6 | 128 | 469 | 86 gal batch begin injecting; 86 gal batch injection complete
3/30/2010 | 1016 | 1025 | 0 | 3.2 | 137 | 500+10 | 31 gal batch + 10 gal chase water; injection complete

---

**Notes:** When the pump head was fixed to the well head the flowrate was at less than 0.5 gpm and pressure was at 2 psi. At 1445 when the pump head was unscrewed from the well head and allowed to pour in under gravity feed, the flowrate was over 4 gpm and pressure ranged from 0 - 0.5 gpm.
ISCO Injection Field Observation Form
Injection Well: MW03

Site: Vieques AOC-I
Project: *In Situ* Activated Alkaline Sodium Persulfate Injection
Contract: Navy CLEAN, CTO-83

### Design Summary
- **Screen Interval**: 24 - 34 ft bgs
- **Total Solution Volume**: 514 gal (includes 20 gallons of chase water)
- **Persulfate Solution per Well**: 475 gal
- **Mass of Persulfate per Well**: 209 lbs (19 gal; 25% solution)
- **Mass of NaOH per Well**: 200 lbs
- **Persulfate concentration**: 5 %; 50 g/L

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<th>Date</th>
<th>Start Time</th>
<th>Stop Time</th>
<th>Pressure range (psi)</th>
<th>Flowrate (gpm)</th>
<th>Total Time (min)</th>
<th>Total Volume (gallons)</th>
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Notes:
ISCO Injection Field Observation Form
Injection Well: MW04

Site: Vieques AOC-I
Project: In Situ Activated Alkaline Sodium Persulfate Injection
Contract: Navy CLEAN, CTO-83

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<th>Pressure range (psi)</th>
<th>Flowrate (gpm)</th>
<th>Total Time (min)</th>
<th>Total Volume (gallons)</th>
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<td>200 gal batch begin injecting</td>
</tr>
<tr>
<td>3/29/2010</td>
<td>1027</td>
<td>--</td>
<td>10</td>
<td>2</td>
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<td>--</td>
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</tr>
<tr>
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<td>1111</td>
<td>--</td>
<td>10</td>
<td>2</td>
<td>--</td>
<td>--</td>
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<td>1121</td>
<td>1136</td>
<td>10</td>
<td>2</td>
<td>169</td>
<td>467</td>
<td>200 gal batch injection complete; stop to mix new batch</td>
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<tr>
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<td>1224</td>
<td>1227</td>
<td>10</td>
<td>2</td>
<td>172</td>
<td>473</td>
<td>33 gal batch begin injecting; stop- injection well head blew off due to pressure- reattach</td>
</tr>
<tr>
<td>3/29/2010</td>
<td>1233</td>
<td>--</td>
<td>10</td>
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<td>--</td>
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<td>1242</td>
<td>1246</td>
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<td>2</td>
<td>185</td>
<td>500</td>
<td>Sodium persulfate injection complete</td>
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<td>1250</td>
<td>1255</td>
<td>10</td>
<td>--</td>
<td>190</td>
<td>510</td>
<td>10 gal chase water added</td>
</tr>
</tbody>
</table>

Notes:
ISCO Injection Field Observation Form
Injection Well: MW07

Site: Vieques AOC-I
Project: In Situ Activated Alkaline Sodium Persulfate Injection
Contract: Navy CLEAN, CTO-83

Design Summary
Screen Interval = 33 - 43 ft bgs
Total Solution Volume = 514 gal
(includes 20 gallons of chase water)
Persulfate Solution per Well = 475 gal
Mass of Persulfate per Well = 209 lbs
Mass of NaOH per Well = 200 lbs (19 gal; 25% solution)
Persulfate concentration: 5 %; 50 g/L

<table>
<thead>
<tr>
<th>Date</th>
<th>Start Time</th>
<th>Stop Time</th>
<th>Pressure range (psi)</th>
<th>Flowrate (gpm)</th>
<th>Total Time (min)</th>
<th>Total Volume (gallons)</th>
<th>Notes</th>
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<td>3/30/2010</td>
<td>1034</td>
<td>1035</td>
<td>0</td>
<td>0.4</td>
<td>1</td>
<td>0.4</td>
<td>tested flowrate when gravity fed- very low when put in 5 gal</td>
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<td>--</td>
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<td>--</td>
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<td>1153</td>
<td>18 - 21</td>
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<td>70</td>
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<tr>
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<td>--</td>
<td>24</td>
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<td>--</td>
<td>16</td>
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<td>20</td>
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<td>3/30/2010</td>
<td>1534</td>
<td>--</td>
<td>20</td>
<td>0.6</td>
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</tr>
<tr>
<td>3/30/2010</td>
<td>1545</td>
<td>--</td>
<td>20</td>
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<tr>
<td>3/30/2010</td>
<td>1603</td>
<td>--</td>
<td>22</td>
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<tr>
<td>3/30/2010</td>
<td>1627</td>
<td>--</td>
<td>22</td>
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</tr>
<tr>
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<td>0720</td>
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<td>20</td>
<td>1.7</td>
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</tr>
<tr>
<td>3/31/2010</td>
<td>0750</td>
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<td>24</td>
<td>1.7</td>
<td>--</td>
<td>--</td>
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</tr>
<tr>
<td>3/31/2010</td>
<td>0810</td>
<td>--</td>
<td>24</td>
<td>1.7</td>
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</tr>
<tr>
<td>3/31/2010</td>
<td>0840</td>
<td>0904</td>
<td>24</td>
<td>1.7</td>
<td>414</td>
<td>400</td>
<td>200 gal batch injection complete</td>
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<td>0932</td>
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<td>22</td>
<td>1.7</td>
<td>--</td>
<td>--</td>
<td>100 gal batch begin injecting</td>
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<td>3/31/2010</td>
<td>0950</td>
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<td>30</td>
<td>1.7</td>
<td>--</td>
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<tr>
<td>3/31/2010</td>
<td>1015</td>
<td>--</td>
<td>16</td>
<td>1.7</td>
<td>--</td>
<td>--</td>
<td></td>
</tr>
</tbody>
</table>

Notes:
### ISCO Injection Field Observation Form

**Injection Well:** MW07

**Site:** Vieques AOC-I  
**Project:** In Situ Activated Alkaline Sodium Persulfate Injection  
**Contract:** Navy CLEAN, CTO-83

### Design Summary

- **Screen Interval:** 33 - 43 ft bgs
- **Total Solution Volume:** 514 gal  
  includes 20 gallons of chase water
- **Persulfate Solution per Well:** 475 gal
- **Mass of Persulfate per Well:** 209 lbs
- **Mass of NaOH per Well:** 200 lbs (19 gal; 25% solution)
- **Persulfate concentration:** 5%; 50 g/L

### Date | Start Time | Stop Time | Pressure range (psi) | Flowrate (gpm) | Total Time (min) | Total Volume (gallons) | Notes
--- | --- | --- | --- | --- | --- | --- | ---
3/31/2010 | 1030 | -- | 29 | 0.6 | -- | -- |  
3/31/2010 | 1100 | 1133 | 28 | 0.6 | 505 | 500 | Sodium persulfate injection complete
3/31/2010 | 1150 | 1207 | -- | -- | 522 | 510 | 10 gal chase water added

### Notes:

- AOC I Injection Field Sheets.xls; Injection MW07 (2)
May 24, 2010

Stephen Brand
CH2M Hill
5700 Cleveland Street, Ste. 101
Virginia Beach, VA  23462

Subject:  Summary of Remedial Chemical Injection Activities Performed at the Navy Clean Site in Vieques, Puerto Rico.

Dear Stephen:

The following is a summary of the work completed by ORIN Remediation Technologies, LLC (ORIN) for CH2M Hill at the Navy Clean Site in Vieques, Puerto Rico.

On March 22, 2010 ORIN began preparation for injection activities by discussing site specific health and safety plans with ORIN, CH2M Hill, and JFA personnel. Potential chemical injection, island specific, and Geoprobe related safety hazards were discussed. The group evaluated specific ways to reduce the risks, and the best practices to maintain safety.

On Tuesday March 23, 2010 ORIN began sodium persulfate injection in AOC E. ORIN injected a 20% sodium persulfate solution into monitoring wells 1, 3, 4, and 5. Sodium persulfate injection in AOC E took place March 23-26, and on March 29, 2010.

ORIN injected on monitoring wells 3, 4, and 5 before beginning injection on MW-1. During injection on the first three monitoring wells, ORIN bailed MW-1 to check for sodium persulfate. The goal was to show influence on MW-1 from injection into the surrounding wells. On Thursday March 25, 2010 a field persulfate test indicated sodium persulfate concentrations of 4.2 to 5.6 ppm in MW-1. Injection on MW-1 began later that day.
Each of the four monitoring wells received 20 gallons of chase water following sodium persulfate injection. Injection rates, pressures, and volumes per well are included in table 1.

ORIN commenced calcium nitrate injection activities in AOC E on Monday, March 22, 2010. Immediately after starting the injection through Geoprobe rods, the treatment chemistry surfaced around the bore hole. After discussing how to proceed, CH2M Hill decided to have JFA install temporary injection points. Calcium nitrate injection resumed via installed temporary injection points Thursday March 25, 2010. Treatment chemistry was delivered by gravity feed, under zero PSI. The calcium nitrate injection was completed March 26, 2010. Following calcium nitrate injection, 20 gallons of chase water was injected into each injection point. Injection rates, pressures, and volumes per well are included in table 2.

ORIN began sodium persulfate injection in AOC I on Saturday March 27, 2010. The first 200 gallons of solution ORIN injected into MW- 2, 3, and 4 simultaneously. After the 200 gallon tank was gone, it was clear that ORINs flow meters were not correctly measuring the total gallons. To overcome this problem, ORIN began injecting on only one monitoring well at a time. Over the course of the following injection day, injection pressure in AOC-I monitoring wells steadily increased. After a successful attempt to gravity feed, ORIN began injecting with no seal on the monitoring well. Under this zero PSI, gravity feed system, injection rates increased. Following treatment chemistry injection, each monitoring well received 10 gallons of chase water. Injection in AOC I was completed Wednesday March 31, 2010. Injection rates, pressures, and volumes per well are included in table 3.

If you have any questions regarding this injection or any other project, please give us a call at (608) 838-6699 ext. 305.

Sincerely,

John Dinneen
Field Technician
ORIN Remediation Technologies, LLC.
### Navy Clean AOC E

#### Sodium Persulfate Post Injection Summary

**Table 1**

<table>
<thead>
<tr>
<th>Injection Point</th>
<th>Date</th>
<th>Time On</th>
<th>Time Off</th>
<th>Injection Depth (feet)</th>
<th>Sodium Persulfate Concentration</th>
<th>Injection Pressure (psi)</th>
<th>Flow Rate (gpm)</th>
<th>Gallons Injected</th>
<th>Comments</th>
<th>Total Volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>MW-3</td>
<td>3/23/10</td>
<td>7:30</td>
<td>17:00</td>
<td>40-50</td>
<td>20%</td>
<td>10</td>
<td>.5-1</td>
<td>165</td>
<td></td>
<td></td>
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<tr>
<td>MW-4</td>
<td>3/23/10</td>
<td>7:30</td>
<td>17:00</td>
<td>40-50</td>
<td>20%</td>
<td>8</td>
<td>.5-1</td>
<td>165</td>
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<td></td>
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<tr>
<td>MW-5</td>
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<td>7:30</td>
<td>17:00</td>
<td>40-50</td>
<td>20%</td>
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<td>.5-1</td>
<td>70</td>
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<td>7:35</td>
<td>10:45</td>
<td>40-50</td>
<td>20%</td>
<td>10</td>
<td>0.2</td>
<td>29</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>10:45</td>
<td>17:00</td>
<td>40-50</td>
<td>20%</td>
<td>20-30</td>
<td>0.8</td>
<td>236</td>
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<td>10:45</td>
<td>40-50</td>
<td>20%</td>
<td>12</td>
<td>0.2</td>
<td>29</td>
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</tr>
<tr>
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<td>17:00</td>
<td>40-50</td>
<td>20%</td>
<td>25-30</td>
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<td>284</td>
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<td>11:03</td>
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<td>8</td>
<td>0.2</td>
<td>38</td>
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<td>500</td>
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<td></td>
<td></td>
<td>11:03</td>
<td>13:13</td>
<td>40-50</td>
<td>20%</td>
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<td>3.5</td>
<td>392</td>
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<td>11:08</td>
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<td>20%</td>
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<td>15</td>
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<td>493</td>
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<td>&lt;0.5</td>
<td>135</td>
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</tr>
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<td>3/26/10</td>
<td>7:22</td>
<td>17:13</td>
<td>40-50</td>
<td>20%</td>
<td>18</td>
<td>0.2</td>
<td>200</td>
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<td></td>
</tr>
<tr>
<td>MW-1</td>
<td>3/29/10</td>
<td>7:25</td>
<td>9:05</td>
<td>40-50</td>
<td>20%</td>
<td>18</td>
<td>0.2</td>
<td>33</td>
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</tr>
<tr>
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<td>9:31</td>
<td>16:46</td>
<td>40-50</td>
<td>20%</td>
<td>18</td>
<td>0.25</td>
<td>100</td>
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<td>9:10</td>
<td>40-50</td>
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<td>Injection Pressure (psi)</td>
<td>Flow Rate (gpm)</td>
<td>Gallons Injected</td>
<td>Comments</td>
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<td>------------</td>
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<td>----------</td>
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<td>-----------------</td>
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</tr>
<tr>
<td>IP-7</td>
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<td>172</td>
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<td>16-26</td>
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<td>16-26</td>
<td>5%</td>
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<td>34</td>
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<td>16:24</td>
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<td>102</td>
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<td>16:48</td>
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<td>0.64</td>
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<td>8:07</td>
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<td>8:53</td>
<td>9:40</td>
<td>16-26</td>
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<td>0</td>
<td>1.5</td>
<td>71</td>
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<td>7:53</td>
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<td>4</td>
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<td>8:04</td>
<td>8:07</td>
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<td>5%</td>
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<td>0.29</td>
<td>1</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>9:05</td>
<td>11:45</td>
<td>16-26</td>
<td>5%</td>
<td>0</td>
<td>0.57</td>
<td>97</td>
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</tr>
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</table>
## Sodium Persulfate Post Injection Summary

### Table 3

<table>
<thead>
<tr>
<th>Injection Point</th>
<th>Date</th>
<th>Time On</th>
<th>Time Off</th>
<th>Injection Depth (feet)</th>
<th>Sodium Persulfate Concentration</th>
<th>Injection Pressure (psi)</th>
<th>Flow Rate (gpm)</th>
<th>Gallons Injected</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>MW-2</td>
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<td>8:53</td>
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The purpose of this memorandum is to provide technical justification for conducting the first post-oxidant-injection groundwater sampling event at AOC I, in accordance with the original schedule (i.e., October 2010, approximately 7 months following injection), despite the presence of low levels of residual persulfate in several of the wells.

During the scoping for the In-Situ Remediation Pilot Studies (AOC E and AOC I Sites) Sampling and Analysis Plan, Former Naval Ammunition Support Detachment, Vieques, Puerto Rico (CH2M HILL, 2010), the team concurred that prior to conducting post-injection sampling, field testing for persulfate would be conducted to ensure oxidant is not collected in the samples. The first post-injection sampling event at AOC I is scheduled for October 2010, in accordance with the current Site Management Plan (SMP) schedule. In anticipation of this, on August 24, 2010, CH2M HILL collected groundwater samples for field analysis of persulfate in monitoring wells at AOC I. The following results were obtained:

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<th>Date</th>
<th>Well</th>
<th>Persulfate (ppm)</th>
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</thead>
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<td>MW01</td>
<td>0</td>
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<tr>
<td>8/24/10</td>
<td>MW02</td>
<td>&gt;70</td>
</tr>
<tr>
<td>8/24/10</td>
<td>MW03</td>
<td>49</td>
</tr>
<tr>
<td>8/24/10</td>
<td>MW04</td>
<td>&gt;70</td>
</tr>
<tr>
<td>8/24/10</td>
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<tr>
<td>8/24/10</td>
<td>MW07</td>
<td>&gt;70</td>
</tr>
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</table>
However, because the wells were not purged prior to collecting the samples, the wells showing positive persulfate results were re-sampled on August 27, 2010, following purging of approximately 1.5 to 2 well volumes. The following results were obtained:

<table>
<thead>
<tr>
<th>Date</th>
<th>Well</th>
<th>Gallons Bailed</th>
<th>Well Volumes</th>
<th>Persulfate (ppm)</th>
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<td>14</td>
</tr>
<tr>
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<td>4.25</td>
<td>1.5</td>
<td>2.1</td>
</tr>
<tr>
<td>8/27/2010</td>
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<td>1.5</td>
<td>1.4</td>
</tr>
<tr>
<td>8/27/2010</td>
<td>MW07</td>
<td>8.25</td>
<td>2</td>
<td>105</td>
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</tbody>
</table>

The above information was shared with FMC, Inc., the manufacturer of the sodium persulfate used as the oxidant at AOC I. As shown in the attached correspondence from FMC, concentrations of persulfate under 500 ppm are no longer reactive with contaminants. Thus, the persulfate remaining in groundwater, when below 500 ppm, will not alter the contaminant analytical results for groundwater samples. FMC stated that this finding is based on 10+ years of practice in the field, and that samples shipped to the laboratory should arrive with the same contaminant concentrations as when they left the field.

Furthermore, sodium persulfate was injected at a 20% by weight concentration in March 2010, and in August 2010 was detected at up to only 0.44 % by weight (105 ppm), evidence for considerable consumption or dilution in the subsurface.

Based on the above information, the Navy proposes that the groundwater sampling at AOC I in October 2010 proceed as planned. Following purging and prior to sampling, groundwater from each well will be field tested for persulfate to ensure the residual persulfate concentration is less than 500 ppm.
From: Julio Vazquez [Vazquez.Julio@epamail.epa.gov]
Sent: Tuesday, March 06, 2012 12:01 PM
To: Doerr, Brett/VBO
Cc: Angela Carpenter; Selcoe, Barrie/HOU; Hannah, Bill/VBO; Daniel Rodriguez; daniel.r.hood@navy.mil; dan.waddill@navy.mil; Ballam, Dennis/VBO; Diana Cutt; diane.wehner@noaa.gov; Felix_Lopez@fws.gov; fultoncom@fultoncom.com; jim@uxopro.com; Martin, John/GNV; Swenfurth, John/TPA; Tomik, John/VBO; kevin.cloe@navy.mil; KRutkowski@trcsolutions.com; madeline.rivera@navy.mil; Michael Sivak; Zamboni, Michael/WDC; Mindy Pensak; richard_henry@fws.gov; Sergio Lopez; Brand, Stephen/VBO; Struve, Susana/WDC; THall@TechLawInc.com; Garretson, Timothy/JAX; Wenk, Tim/VBO; Kappleman, William/WDC; wilmarierivera@jca.pr.gov
Subject: Re: Vieques - February 2012 Draft Tech Sub Meeting Minutes; May 2012 Draft Tech Sub Meeting Agenda; Consensus/Action Item Lists

Brett:

One of the items I was assigned to follow up for the subject meeting was the identification of the monitoring wells that should be sampled for the next two rounds for AOC I. After talking to Diana, she suggested we sample MW-04, MW-05 and MW-07, as they are the ones that had benzene concentrations initially exceeding criteria. Call me if you have any questions.

Julio F Vázquez, RPM
U.S. EPA - Region 2
Special Projects Branch/
Federal Facilities Section
New York

United States Environmental Protection Agency
From: Doerr, Brett/VBO  
Sent: Thursday, September 30, 2010 9:16 AM  
To: Swenfurth, John/TPA; Brand, Stephen/VBO; Hannah, Bill/VBO  
Subject: FW: First post-injection sampling event at west Vieques AOC I

From: Cutt.Diana@epamail.epa.gov  
Sent: Thursday, September 30, 2010 10:09 AM  
To: Doerr, Brett/VBO; Rodriguez.Daniel@epamail.epa.gov  
Cc: WilmarieRivera@jca.gobierno.pr; Richard_Henry@fws.gov; Sivak.Michael@epamail.epa.gov; Pensak.Mindy@epamail.epa.gov; Diane.Weher@noaa.gov; kevin.cloe@navy.mil; daniel.r.hood@navy.mil; Tomik, John/VBO; madeline.rivera@navy.mil  
Subject: RE: First post-injection sampling event at west Vieques AOC I

Just spoke to Scott Huling, EPA's in situ oxidation expert in Ada, OK. According to him, residual levels of persulfate even at the concentrations were are seeing at AOC I, can be a problem and continue to effect the contaminant concentrations in the sample. This has been the subject of much recent scrutiny and study by EPA. Although the assertion made by FMC is not necessarily a bad statement, it is leaving out such factors as: UV light, heat from the sun and a bigger issue - heating during analysis in the GS/MS headspace method. All of these factors can activate the persulfate in the sample container and effectively lower the contaminant concentrations (see Scott’s note below).

Scott's suggestions are:

1. wait to sample until no persulfate remains, or
2. add a preservative to the sample. He has successfully used ascorbic acid (4:1 acid:sample ratio).

Scott is available by phone or email to discuss further if need be. Thanks.

-Diana

Diana Cutt, P.G., Geologist  
EPA Region 2  
ERRD/PSB/TST  
290 Broadway  
NY, NY 10007  
212-637-4311

Diana, attached is an abstract from a journal article that was submitted to a journal for publication. I believe Phil Block is generally correct in his letter, but there are conditions in which the persulfate residual in a ground water sample can be activated and can negatively impact the quality of the ground water sample that is not addressed in the memo. Specifically, one condition involves the method of analysis. The headspace method, used to analyze VOCs in different EPA methods, involves a heating step that will activate the persulfate. Even low concentrations of persulfate, i.e., < 500 mg/L, this will significantly impact the quality of the sample. Scott
Scott G. Huling, Ph.D., P.E.
Environmental Engineer
U.S. Environmental Protection Agency
Robert S. Kerr Environmental Research Center
P.O. Box 1198 (or, 919 Kerr Lab Drive)
Ada, OK 74820
Phone: (580) 436-8610; Fax: (580) 436-8614
e-mail: Huling.Scott@epa.gov
website: http://www.epa.gov/ada/research.html
September 10, 2010

RE: Reactivity of Dilute Concentrations of Klozur® Persulfate

It is the experience of FMC over the past ten years that the minimum reactive concentration of sodium persulfate in groundwater is 0.5 g / L (500 ppm). Oxidative reaction rate is proportional to the concentration of the contaminant, the concentration of the oxidant and the concentration of the persulfate activator. At concentrations below this level, the effective reaction rate with contaminants of concern is essentially zero, and for all intents and purposes the oxidative reaction is complete. This is further impacted by the co-incident reduction in persulfate activator concentration.

Transportation of groundwater samples containing less than 500 ppm of persulfate should not occur further significant contaminant reduction in route to the laboratory, assuming the transportation time is not significant (less than a couple of days) and the sample is not exposed to a significant heat source. This can be further mitigated by shipment of the sample on ice.

Philip Block
Technology Manager – Remediation
FMC Corporation
Based on a comment received from EPA regarding the proposal to move forward with the first post-injection sampling at AOC I despite the presence of low levels of residual persulfate in several wells, a conference call was held on Monday October 4, 2010 among the following:

Diana Cutt/EPA – Hydrogeology technical support for Vieques environmental restoration program
Scott Huling/EPA – research lead regarding in-situ chemical oxidation
Susanne Borchert/CH2M HILL – In-situ remediation technology expert
Mike Zamboni/CH2M HILL – Chemist for Vieques environmental restoration program
Brett Doerr/CH2M HILL – Vieques environmental restoration program lead

Based on research done by EPA, samples containing residual persulfate have shown decreases in VOC concentrations in the laboratory when analyzing VOCs using the GC method with the purge and trap process. EPA has found that adding sufficient ascorbic acid to the samples prevents the loss of VOCs because the persulfate preferentially oxidizes the ascorbic acid instead of the VOCs.

Therefore, the group concurred that sampling at AOC I should proceed as planned, with the sampling protocol modified to include the addition of ascorbic acid to the sample containers as a field preservative. Ascorbic acid will be added to the sample containers at a ratio of 4 moles of ascorbic acid (or greater) per mole of persulfate. Scott stated that having more than a 4:1 ratio of ascorbic acid:persulfate (at least up to 40:1 ratio per his research) does not negatively affect the VOC results. Persulfate measurements after purging and prior to sampling will be conducted to ensure sufficient ascorbic acid is added to each sample container for VOCs analysis.

Based on the above, CH2M HILL will proceed with the sampling event during the week of October 25, 2010. If anyone has any concerns or comments on the approach, please let us know by COB Friday October 8, 2010.
Dear Mr. Acaron,

The following Data Validation report is provided as requested for the parameters noted in the table below for SDG # SJ0464. The data validation was performed in accordance with the SW-846 methods utilized by the laboratory, the Region II Standard Operating Procedures for the Validation of Organic Data Acquired Using SW-846 Methods (8260B-Rev 2, January 2006- SOP #HW-24, 8270D-Rev 3 and October 2006-SOP #HW-22), and professional judgment. Region II has not developed a validation checklist SOP for the methods used to assess the inorganic method in this SDG (SW-846 methods 6010B) or the organic methods used to assess the fuels (SW-846 8015G for gasoline and 8015 TPH for diesel range organics). The Region II Standard Operating Procedure for the Evaluation of Metals Data for the CLP was used as applicable for the metals data. For the other fraction alternative worksheets were provided. Region II flagging conventions were used. All areas of concern are discussed in the body of the report and a summary of data qualifications is provided.

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<td>VWAE-MW02-0310MSD</td>
<td>J0464-10MSD</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The following quality control samples were provided with this SDG: samples VWAE-TB01-031610, VWAE-TB01-031710, VWAI-TB01-031810, VWAI-TB01-031910 and VXAI-TB01-032210-trip blanks; samples VWAE-EB01-031610, VWAE-EB01-031710, VWAI-EB01-031810, VWAI-EB01-031910 and VWAI-EB01-032210-equipment blanks; sample VWAE-MW4P-0310-field duplicate of sample VWAE-MW4-0310; and sample VWAI-MW03P-0310-field duplicate of sample VWAI-MW03-0310.

The samples were evaluated based on the following criteria:

- Data Completeness
- Sample Condition
- Technical Holding Times
- GC/MS Tuning
- GC Performance
- Initial/Continuing Calibrations
- ICSA/ICSAB Standards
- CRI Standards
- Blanks
- Internal Standards
- Surrogate Recoveries
- Laboratory Control Samples
- Matrix Spike Recoveries
- Matrix Duplicate RPDs
- Serial Dilutions
- Field Duplicates
- Identification/Quantitation
- Reporting Limits
- Tentatively Identified Compounds

* - indicates that qualifications were not required based on this criteria

**Overall Evaluation of Data/Potential Usability Issues**

A summary of qualifications applied to the sample results are noted below for the fractions validated. Specific details regarding qualification of the data are addressed in the Specific Evaluation section of this narrative. If an issue is not addressed there were no actions required based on unmet quality criteria. When more than one qualifier is associated with a compound/analyte the validator has chosen the qualifier that best indicates possible bias in the results and flagged the data accordingly. However, information regarding all quality control issues is provided in the body of the report and on the qualification summary page. Please note that when a compound or analyte is flagged due to blank contamination the BL qualifier code takes precedence over all other qualifier codes except a code that explains rejected data.
VOA

One sample required a dilution to obtain results within the calibration range.

SVOA

No qualifications to the data were required.

GRO

One of the associated rinse blanks exhibited contamination for GRO. One field sample required qualification.

TPH

No qualifications to the data were required.

Select Metals

The laboratory did not analyze a CRI standard for the analyte manganese as required. The analyte was flagged as estimated for reported concentrations <2X RL.

Specific Evaluation of Data

Data Completeness

The SDG was received complete and intact. Resubmissions were not required. Clarification of gasoline calculation was requested from the laboratory. A copy of the e-mail correspondence is included in the validation worksheets section of this report.

Technical Holding Times

According to chain of custody records, sampling was performed on 3/16-22/10 and samples were received at the laboratory 3/17-23/10. All sample preparation and analysis was performed within Region II and/or method holding time requirements.

CRI Standards

Select Metals

The laboratory did not analyze a CRI standard for the analyte manganese. All positive results were above the action level of 2X the reporting limit. The reported non-detect result for manganese in sample VWAE-W03-0310 was qualified as estimated UJ with a qualifier code of OT.
Blanks

GRO

One of the rinse blanks associated with samples in this SDG exhibited contamination for gasoline range organics. Specific information on the contamination is noted in the following table.

<table>
<thead>
<tr>
<th>Blank ID</th>
<th>Compound</th>
<th>Concentration</th>
<th>Action Level</th>
<th>Q Flag</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAE-EB01-031610</td>
<td>GRO</td>
<td>110 ug/L</td>
<td>blank level</td>
<td>U</td>
</tr>
</tbody>
</table>

Associated samples and required qualifications are noted in the following table.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Q Flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAE-MW03-0310</td>
<td>GRO</td>
<td>U</td>
<td>EBL</td>
</tr>
</tbody>
</table>

Identification/Quantitation

VOA

A dilution was required for sample VWAE-MW05-0310 to obtain results within the calibration range. Therefore, E-flagged compound results were not used in the initial analysis of this sample in favor of the corresponding D-flagged compound result in the dilution, qualifier code: DL.

A summary of qualifications required is provided on the following page. Please do not hesitate to contact DataQual ES with any questions regarding this validation report.

Sincerely,

Jacqueline Cleveland
Vice President
Summary of Data Qualifications

**VOA**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAE-MW05-0310</td>
<td>all E-flagged results</td>
<td>+</td>
<td>R</td>
<td>DL</td>
</tr>
<tr>
<td>VWAE-MW05-0310DL</td>
<td>all compound except D-flagged results</td>
<td>+/-</td>
<td>R</td>
<td>DL</td>
</tr>
</tbody>
</table>

**SVOA**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

No qualifications

**GRO**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAE-MW03-0310</td>
<td>GRO</td>
<td>+</td>
<td>U</td>
<td>EBL</td>
</tr>
</tbody>
</table>

**DRO**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

No qualifications

**Select Metals**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Analyte</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAE-MW03-0310</td>
<td>manganese</td>
<td>-</td>
<td>UJ</td>
<td>OT</td>
</tr>
</tbody>
</table>
Glossary of Qualification Flags and Abbreviations

Qualification Flags (Q-Flags)

- **U**: not detected above the reported sample quantitation limit
- **J**: estimated value
- **UJ**: reported quantitation limit is qualified as estimated
- **N**: analyte has been tentatively identified
- **IN**: analyte has been tentatively identified, estimated value
- **R**: result is rejected; the presence or absence of the analyte cannot be verified

Method/Preparation/Field QC Blank Qualification Flags (Q-Flags)

Organic Methods

- **NA**: The sample result for the blank contaminant is greater than the RL (2X sample RL for common laboratory contaminants) when the blank value is less than the RL. The sample result for the blank contaminant is not qualified with any blank qualifiers.
- **U**: The sample result for the blank contaminant is less than the RL (2X sample RL for common laboratory contaminants) but greater than the MDL when the blank value is less than the RL. The sample result for the blank contaminant is qualified as non-detect U at the reported concentration.
- **RL**: The sample result for the blank contaminant is less than the RL (2X sample RL for common laboratory contaminants) but greater than the MDL when the blank value is less than the RL. The sample result for the blank contaminant is changed to the RL and qualified as non-detect U.

* This guideline is used when the laboratory is reporting non-detects to the MDL. ** This guideline is used when the laboratory is reporting non-detects to the RL.

Inorganic Methods

ICB/CCB/PB Action:

- **No Action**: The sample result is greater than the RL and greater than ten times (10X) the blank value.
- **U**: The sample result is greater than or equal to the MDL but less than or equal to the RL, result is reported as non-detect at the RL* or at the reported concentration**, when the ICB/CCB/PB result is less or greater than the RL.
Glossary of Qualification Flags and Abbreviations, continued

R - Sample result is greater than the RL and less than the ICB/CCB/PB value when the ICB/CCB/PB value is greater than the RL.

J - Sample result is greater than the ICB/CCB/PB value but less than 10X the ICB/CCB/PB value when ICB/CCB/PB value is greater than the RL.

J/UJ - Sample result is less than 10X RL when blank result is below the negative RL.

* This guideline is used when the laboratory is reporting non-detects to the MDL. ** This guideline is used when the laboratory is reporting non-detects to the RL.

Field QC Blank action:

Note – Use field blanks to qualify data only if field blank results are greater than prep blank results.

Do not use rinsate blank associated with soils to qualify water samples and vice versa.

No Action - The sample result is greater than the RL and greater than ten times (10X) the blank value.

U - The sample result is greater than or equal to the MDL but less than or equal to the RL, result is reported as non-detect at the RL* or at the reported concentration**, when the FB result is less or greater than the RL.

R - Sample result is greater than the RL and less than the FB value when the FB value is greater than the RL.

J - Sample result is greater than the FB value but less than 10X the FB value when FB value is greater than the RL.

* This guideline is used when the laboratory is reporting non-detects to the MDL. ** This guideline is used when the laboratory is reporting non-detects to the RL.

General Abbreviations

RL reporting limit
PQL practical quantitation limit
IDL instrument detection limit
MDL method detection limit
CRDL contract required detection limit
CRQL contract required quantitation limit
+ positive result
- non-detect result
<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>TN</td>
<td>Tune</td>
</tr>
<tr>
<td>BSL</td>
<td>Blank Spike/LCS - High Recovery</td>
</tr>
<tr>
<td>BSH</td>
<td>Blank Spike/LCS - Low Recovery</td>
</tr>
<tr>
<td>BD</td>
<td>Blank Spike/Blank Spike Duplicate (LCS/LCSD) Precision</td>
</tr>
<tr>
<td>BRL</td>
<td>Below Reporting Limit</td>
</tr>
<tr>
<td>ISL</td>
<td>Internal Standard - Low Recovery</td>
</tr>
<tr>
<td>ISH</td>
<td>Internal Standard - High Recovery</td>
</tr>
<tr>
<td>MSL</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - Low Recovery</td>
</tr>
<tr>
<td>MSH</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - High Recovery</td>
</tr>
<tr>
<td>MI</td>
<td>Matrix interference obscuring the raw data</td>
</tr>
<tr>
<td>MDP</td>
<td>Matrix Spike/Matrix Spike Duplicate Precision</td>
</tr>
<tr>
<td>2S</td>
<td>Second Source - Bad reproducibility between tandem detectors</td>
</tr>
<tr>
<td>SSL</td>
<td>Spiked Surrogate - Low Recovery</td>
</tr>
<tr>
<td>SSH</td>
<td>Spiked Surrogate - High Recovery</td>
</tr>
<tr>
<td>SD</td>
<td>Serial Dilution Reproducibility</td>
</tr>
<tr>
<td>ICL</td>
<td>Initial Calibration - Low Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICH</td>
<td>Initial Calibration - High Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICB</td>
<td>Initial Calibration - Bad Linearity or Curve Function</td>
</tr>
<tr>
<td>CCL</td>
<td>Continuing Calibration - Low Recovery or %Difference</td>
</tr>
<tr>
<td>CCH</td>
<td>Continuing Calibration - High Recovery or %Difference</td>
</tr>
<tr>
<td>LD</td>
<td>Lab Duplicate Reproducibility</td>
</tr>
<tr>
<td>HT</td>
<td>Holding Time</td>
</tr>
<tr>
<td>PD</td>
<td>Pesticide Degradation</td>
</tr>
<tr>
<td>2C</td>
<td>Second Column - Poor Dual Column Reproducibility</td>
</tr>
<tr>
<td>LR</td>
<td>Concentration Exceeds Linear Range</td>
</tr>
<tr>
<td>BL</td>
<td>Blank Contamination</td>
</tr>
<tr>
<td>RE</td>
<td>Redundant Result - due to Re-analysis or Re-extraction</td>
</tr>
<tr>
<td>DL</td>
<td>Redundant Result - due to Dilution</td>
</tr>
<tr>
<td>FD</td>
<td>Field Duplicate</td>
</tr>
<tr>
<td>OT</td>
<td>Other - explained in data validation report</td>
</tr>
<tr>
<td>%SOL</td>
<td>High moisture content</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>5.0 U</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0 U</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0 U</td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>5.0 U</td>
<td></td>
</tr>
</tbody>
</table>
**Lab Name:** MITKEM LABORATORIES

**Lab Code:** MITKEM  
**Case No.:** J0464  
**Mod. Ref No.:**  
**SDG No.:** SJ0464

**Matrix:** (SOIL/SED/WATER)  
**WATER**  
**Lab Sample ID:** J0464-02A  
**Lab File ID:** V2L4990.D

**Sample wt/vol:** 5.00 (g/mL) ML  
**Level:** (TRACE/LOW/MED)  
**LOW**  
**% Moisture:** not dec.  
**Date Received:** 03/17/2010

**GC Column:** DB-624  
**ID:** 0.25 (mm)  
**Dilution Factor:** 1.0

**Soil Extract Volume:** (uL)  
**Soil Aliquot Volume:** (uL)  
**Purge Volume:** 5.0 (mL)

### Concentration Table

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Contract:
Mod. Ref No.:
SDG No.: SJ0464

Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.

GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J0464  
Mod. Ref No.: 
SDG No.: SJ0464  
Matrix: WATER  
Sample wt/vol: 5.00 (g/mL)  
Level: LOW  
% Moisture: not dec.  
GC Column: DB-624  
ID: 0.25 (mm)  
Dilution Factor: 1.0  
Soil Extract Volume: (uL)  
Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>340 (µg/L)</td>
<td>5</td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0 (µg/L)</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>4.5 (µg/L)</td>
<td>J</td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>5.0 (µg/L)</td>
<td>U</td>
</tr>
</tbody>
</table>
**Lab Name:** MITKEM LABORATORIES
**Lab Code:** MITKEM
**Case No.:** J0464
**Mod. Ref No.:**
**SDG No.:** SJ0464

**Matrix:** (SOIL/SED/WATER) WATER
**Sample wt/vol:** 5.00 (g/mL) ML
**Level:** (TRACE/LOW/MED) LOW
**% Moisture:** not dec.

**GC Column:** DB-624 ID: 0.25 (mm)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (µg/L or µg/Kg)</th>
<th>µg/L</th>
<th>Q</th>
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<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td></td>
<td>520</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td></td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td></td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td></td>
<td>25</td>
<td></td>
</tr>
</tbody>
</table>

**Date Received:** 03/18/2010
**Date Analyzed:** 04/01/2010

**Lab Sample ID:** J0464-04ADL
**Lab File ID:** V2L5281.D

**Soil Extract Volume:** (uL)
**Soil Aliquot Volume:** (uL)
**Purge Volume:** 5.0 (mL)
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Mod. Ref No.: SDG No.: SJ0464
Matrix: (SOIL/SED/WATER) WATER
Lab Sample ID: J0464-05A
Sample wt/vol: 5.00 (g/mL) ML
Lab File ID: V2L5199.D
Level: (TRACE/LOW/MED) LOW
Date Received: 03/18/2010
% Moisture: not dec.
Date Analyzed: 03/30/2010
GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td></td>
<td>130</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
**Lab Name:** MITKEM LABORATORIES  
**Lab Code:** MITKEM  
**Case No.:** J0464  
**Mod. Ref No.:**  
**SDG No.:** SJ0464  
**Matrix:** (SOIL/SED/WATER) WATER  
**Lab Sample ID:** J0464-06A  
**Lab File ID:** V2L4993.D  
**Sample wt/vol:** 5.00 (g/mL) ML  
**Level:** (TRACE/LOW/MED) LOW  
**% Moisture:** not dec.  
**GC Column:** DB-624 ID: 0.25 (mm)  
**Soil Extract Volume:** (uL)  
**Purge Volume:** 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
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</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td></td>
<td>96</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td></td>
<td>5.0</td>
<td>0</td>
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<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td></td>
<td>5.0</td>
<td>0</td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td></td>
<td>5.0</td>
<td>0</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J0464  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Soil Extract Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (µg/L or µg/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µG/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>CAS NO.</td>
<td>COMPOUND</td>
<td>CONCENTRATION UNITS: (µg/L or µg/Kg)</td>
<td>Q</td>
<td></td>
</tr>
<tr>
<td>-----------</td>
<td>------------------------</td>
<td>--------------------------------------</td>
<td>---</td>
<td></td>
</tr>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>120</td>
<td></td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>6.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: SOIL/SED/WATER
Sample wt/vol: 5.00 (g/mL)
Level: LOW
% Moisture: not dec.
GC Column: DB-624
Soil Extract Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (μg/L or μg/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>μg/L 5.0</td>
<td>0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>μg/L 5.0</td>
<td>0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>μg/L 5.0</td>
<td>0</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Lab Sample ID: J0464-11A
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J0464  
Mod. Ref No.:  
SDG No.: SJD464  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Purge Volume: 5.0 (mL)  
Lab Sample ID: J0464-12A  
Lab File ID: V2L4999.D  
Date Received: 03/19/2010  
Date Analyzed: 03/22/2010  
CONCENTRATION UNITS: (ug/L or ug/Kg) µg/L Q

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: WATER
Sample wt/vol: 5.00 (g/mL)
Level: LOW
% Moisture: not dec.
GC Column: DB-624

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS:</th>
<th>Q</th>
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</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0 (ug/L)</td>
<td>0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0 (ug/L)</td>
<td>0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>5.0 (ug/L)</td>
<td>0</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464

Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML

Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.

GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0 uL</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0 uL</td>
<td>U</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>5.0 uL</td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Mod. Ref No.: S J0464
SDG No.: S J0464
Lab Sample ID: J0464-16A
Lab File ID: V2L5002.D
Date Received: 03/20/2010
Date Analyzed: 03/22/2010

% Moisture: not dec.

GC Column: DB-624
ID: 0.25 (mm)
Dilution Factor: 1.0

Soil Extract Volume: (uL)
Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>5.0</td>
<td>U</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM  Case No.: J0464
Matrix: (SOIL/SED/WATER)  WATER
Sample wt/vol:  5.00 (g/mL) ML
Level: (TRACE/LOW/MED)  LOW
% Moisture: not dec.
GC Column: DB-624  ID: 0.25 (mm)
Soil Extract Volume:  (uL)
Purge Volume:  5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>5.0</td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES

Lab Code: MITKEM  Case No.: J0464

Matrix: (SOIL/SED/WATER) WATER  Mod. Ref No.:

Sample wt/vol: 5.00 (g/mL) ML  SDG No.: SJ0464

Level: (TRACE/LOW/MED) LOW  Lab Sample ID: J0464-18A

% Moisture: not dec.  Lab File ID: V2L5003.D

GC Column: DB-624  Dilution Factor: 1.0

Soil Extract Volume:  (uL) Soil Aliquot Volume:  (uL)

Purge Volume:  5.0  (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS:</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0 (ug/L or ug/Kg)</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0 (ug/L or ug/Kg)</td>
<td>U</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>5.0 (ug/L or ug/Kg)</td>
<td>U</td>
</tr>
</tbody>
</table>
### Volatile Organics Analysis Data Sheet

**Lab Name:** MITKEM LABORATORIES  
**Lab Code:** MITKEM  
**Case No.:** J0464  
**Matrix:** WATER  
**Sample wt/vol:** 5.00 (g/mL)  
**Level:** LOW  
**% Moisture:** not dec.  
**GC Column:** DB-624  
**Soil Extract Volume:** (uL)  
**Purge Volume:** 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (µg/L or µg/Kg)</th>
<th>µG/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
</tbody>
</table>

---

**Client Sample No.:** VWAI-EB01-032210
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td></td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
**Lab Name:** MITKEM LABORATORIES  
**Lab Code:** MITKEM  
**Case No.:** J0464  
**Matrix:** WATER  
**Sample wt/vol:** 5.00 (g/mL) ML  
**Level:** LOW  
**% Moisture:** not dec.  
**GC Column:** DB-624 ID: 0.25 (mm)  
**Purge Volume:** 5.0 (mL)

### CAS NO. | COMPOUND | CONCENTRATION UNITS: (μg/L or μg/Kg) | μg/L | Q
--- | --- | --- | --- | ---
107-06-2 | 1,2-Dichloroethane | 5.0 | 0 |
71-43-2 | Benzene | 14 |  |
78-87-5 | 1,2-Dichloropropane | 5.0 | 0 |
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J0464  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624  
ID: 0.25 (mm)  
Dilution Factor: 1.0  
Soil Extract Volume:  
Soil Aliquot Volume:  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (μg/L or μg/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>54 μg/L</td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>53 μg/L</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>58 μg/L</td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>170 μg/L</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J0464  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0  
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (µg/L or µg/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>1634-04-4</td>
<td>Methyl tert-butyl ether</td>
<td>49</td>
<td></td>
<td></td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>47</td>
<td></td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>51</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1330-20-7</td>
<td>Xylene (Total)</td>
<td>150</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Matrix: WATER  
Lab Sample ID: J0464-01E  
Lab File ID: S3G3654.D  
Sample wt/vol: 1000 (g/mL)  
Level: LOW  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL)  
GPC Cleanup: N  
Concentration Units: (ug/L or ug/Kg)  
CAS NO. COMPOUND  
Q  
91-20-3 Naphthalene  
1.0 U  
91-57-6 2-Methylnaphthalene  
1.0 U
<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (µg/L or µg/Kg) µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
</tbody>
</table>
### Lab Information

- **Lab Name:** MITKEM LABORATORIES
- **Lab Code:** MITKEM
- **Case No.:** J0464
- **Contract:**
- **Mod. Ref No.:**
- **SDG No.:** SJ0464

### Sample Information

- **Matrix:** WATER
- **Sample wt/vol:** 1000 (g/mL) ML
- **Level:** LOW
- **% Moisture:**
- **Concentrated Extract Volume:** 1000 (uL)
- **Injection Volume:** 1.0 (uL)
- **GPC Cleanup:** N
- **pH:**
- **Dilution Factor:** 1.0

### Extraction Information

- **Extraction:** SEPF
- **Date Received:** 03/18/2010
- **Date Extracted:** 03/24/2010
- **Date Analyzed:** 03/26/2010

### CAS Numbers and Concentrations

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg) μg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>13</td>
<td></td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>5.8</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J0464  
Mod. Ref No.:  
SDG No.: SJD0464  
Matrix: (SOIL/SED/WATER) WATER  
Lab Sample ID: J0464-05E  
Sample wt/vol: 1000 (g/mL) ML  
Lab File ID: S3G3662.D  
Level: (LOW/MED) LOW  
Extraction: (Type) SEPF  
% Moisture: Decanted: (Y/N)  
Date Received: 03/18/2010  
Concentrated Extract Volume: 1000 (uL)  
Date Extracted: 03/24/2010  
Injection Volume: 1.0 (uL) GPC Factor: 1.00  
Date Analyzed: 03/26/2010  
GPC Cleanup: (Y/N) N  
pH:  
Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>u</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>u</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: __________
Decanted: (Y/N) ______
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL)
GPC Cleanup: (Y/N) N

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
</tbody>
</table>

Date Received: 03/18/2010
Date Extracted: 03/24/2010
Date Analyzed: 03/26/2010

pH: ______  Dilution Factor: 1.0

Contract: __________
Mod. Ref No.: __________
SDG No.: SJ0464
Lab Sample ID: J0464-06B
Lab File ID: S3G3663.D
Extraction: (Type) SEPF
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Mod. Ref No.: 
SDG No.: SJD064

Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: _______ Decanted: (Y/N) _______ Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N pH: _______ Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>g/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td></td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td></td>
<td>1.0</td>
<td>U</td>
</tr>
</tbody>
</table>

Date Tested: 03/24/2010
Date Analyzed: 03/26/2010

CONCENTRATION UNITS: (ug/L or ug/Kg) | pG/L | Q  
1.0 | U  
1.0 | U  

Date Received: 03/18/2010
Date Extracted: 03/24/2010
Date Analyzed: 03/26/2010
### Lab Name: MITKEM LABORATORIES
### Lab Code: MITKEM
### Case No.: J0464
### Lab Sample ID: J0464-09E
### Lab File ID: S3G3665.D
### Matrix: (SOIL/SED/WATER) WATER
### Sample wt/vol: 1000 (g/mL) ML
### Level: (LOW/MED) LOW
### % Moisture: __________ Decanted: (Y/N) __________
### Concentrated Extract Volume: 1000 (uL)
### Injection Volume: 1.0 (uL)
### GPC Cleanup: (Y/N) N
### pH: __________
### Extraction: (Type) SEPF
### Date Received: 03/18/2010
### Date Extracted: 03/24/2010
### Date Analyzed: 03/26/2010

### CAS NO. | COMPOUND            | CONCENTRATION UNITS: (ug/L or ug/Kg) | Q |
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>6.6</td>
<td></td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>8.0</td>
<td></td>
</tr>
<tr>
<td>CAS NO.</td>
<td>COMPOUND</td>
<td>CONCENTRATION UNITS:</td>
<td></td>
</tr>
<tr>
<td>---------</td>
<td>---------------------------------</td>
<td>----------------------</td>
<td></td>
</tr>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0 µg/L</td>
<td></td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0 µg/L</td>
<td></td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0 µg/L</td>
<td></td>
</tr>
<tr>
<td>CAS NO.</td>
<td>COMPOUND</td>
<td>CONCENTRATION UNITS: (ug/L or ug/Kg)</td>
<td>Q</td>
</tr>
<tr>
<td>---------</td>
<td>--------------------------------------</td>
<td>-------------------------------------</td>
<td>---</td>
</tr>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>1.4</td>
<td>J</td>
</tr>
</tbody>
</table>

Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N) Date Received: 03/19/2010
Concentrated Extract Volume: 1000 (uL) Date Extracted: 03/25/2010
Injection Volume: 1.0 (uL) GPC Factor: 1.00 Date Analyzed: 03/27/2010
GPC Cleanup: (Y/N) N pH: Dilution Factor: 1.0

CAS NO. 91-20-3 91-57-6 117-81-7
COMPOUND Naphthalene 2-Methylnaphthalene Bis(2-ethylhexyl)phthalate
CONCENTRATION UNITS: ug/L 1.0 3.0 1.4
Q U U J
### Lab Name: MITKEM LABORATORIES

### Lab Code: MITKEM

### Case No.: J0464

### Contract:

### Mod. Ref No.:

### SDG No.: SJ0464

### Matrix: (SOIL/SED/WATER) WATER

### Sample wt/vol: 1000 (g/mL) ML

### Level: (LOW/MED) LOW

### % Moisture: 

### Decanted: (Y/N)

### Concentrated Extract Volume: 1000 (uL)

### Date Received: 03/19/2010

### Extraction: (Type) SEPF

### Date Extracted: 03/25/2010

### Lab Sample ID: J0464-12B

### Lab File ID: S3G3672.D

### Injection Volume: 1.0 (uL) GPC Factor: 1.00

### Date Analyzed: 03/27/2010

### GPC Cleanup: (Y/N) N

### pH: 

### Dilution Factor: 1.0

### CAS NO. | COMPOUND | CONCENTRATION UNITS: (ug/L or ug/Kg) | Q |
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>117-91-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
### Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL)
GPC Cleanup: (Y/N) N

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS:</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0 µg/L</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0 µg/L</td>
<td>U</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0 µg/L</td>
<td>U</td>
</tr>
</tbody>
</table>

Lab Sample ID: J0464-14E
Lab File ID: S3G3737.D
Date Received: 03/20/2010
Date Extracted: 03/26/2010
Date Analyzed: 03/30/2010

Extraction: (Type) CONT
% Moisture: 
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL)
GPC Cleanup: (Y/N) N

Dilution Factor: 1.0

pH:
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Mod. Ref No.: SDG No.: SJ0464

Matrix: WATER
Sample wt/vol: 1000 (g/mL) ML
Level: LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>U</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  Contract:  
Lab Code: MITKEM  Case No.: J0464  Mod. Ref No.:  
Matrix: (SOIL/SED/WATER) WATER  SDG No.: SJ0464  
Sample wt/vol: 1000 (g/mL) ML  Lab Sample ID: J0464-17B  
Level: (LOW/MED) LOW  Lab File ID: S3G3740.D  
% Moisture:  Decanted: (Y/N)  Date Received: 03/20/2010  
Concentrated Extract Volume: 1000 (uL)  Date Extracted: 03/26/2010  
Injection Volume: 1.0 (uL) GPC Factor: 1.00  Date Analyzed: 03/30/2010  
GPC Cleanup: (Y/N) N  pH:  
Dilution Factor: 1.0  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>µg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td></td>
<td>U</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td></td>
<td>U</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td></td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J0464  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 1000 (g/mL) ML  
Level: (LOW/MED) LOW  
% Moisture: Decanted: (Y/N)  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL) GPC Factor: 1.00  
GPC Cleanup:(Y/N) N  
CAS NO. | COMPOUND                  | CONCENTRATION UNITS: (ug/L or ug/Kg) | Q  
---|---------------------------|--------------------------------------|---  
91-20-3 | Naphthalene               | 1.0                                   | U  
91-57-6 | 2-Methylnaphthalene       | 1.0                                   | U  
117-81-7 | Bis(2-ethylhexyl)phthalate | 1.2                                   | J  

Date Received: 03/23/2010  
Date Extracted: 03/26/2010  
Date Analyzed: 03/30/2010  
Lab File ID: S3G3741.D  
Lab Sample ID: J0464-20B  
SDG No.: SJ0464  

Extra c tion: (Type) CONT  
Dilution Factor: 1.0
<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>21</td>
<td></td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>17</td>
<td></td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>U</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Lab Sample ID: J0464-01EMS
Lab Code: MITKEM
Case No.: J0464
Sample wt/vol: 1000 (g/mL) ML
Lab File ID: S3G3655.D
Level: (LOW/MED) LOW
Extraction: (Type) SEPF
% Moisture: Decanted: (Y/N)
Date Received: 03/17/2010
Concentrated Extract Volume: 1000 (uL)
Date Extracted: 03/23/2010
Injection Volume: 1.0 (uL) GPC Factor: 1.00
Date Analyzed: 03/26/2010
GPC Cleanup: (Y/N) N
pH: Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>μg/L</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td></td>
<td>41</td>
<td></td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylanthalene</td>
<td></td>
<td>39</td>
<td></td>
</tr>
</tbody>
</table>
### Lab Information

- **Lab Name:** MITKEM LABORATORIES
- **Lab Code:** MITKEM
- **Case No.:** J0464
- **Matrix:** WATER
- **Sample wt/vol:** 1000 (g/mL)
- **Level:** LOW
- **% Moisture:**
- **Concentrated Extract Volume:** 1000 (uL)
- **Injection Volume:** 1.0 (uL)
- **GPC Factor:** 1.00
- **GPC Cleanup:** N
- **Date Received:** 03/17/2010
- **Date Extracted:** 03/23/2010
- **Date Analyzed:** 03/26/2010
- **Dilution Factor:** 1.0

### Table: CAS NO. & Concentrations

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>(ug/L or ug/Kg)</td>
<td>31</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>µg/L</td>
<td>30</td>
</tr>
</tbody>
</table>

### Notes

- **Lab Sample ID:** J0464-01EMSD
- **Lab File ID:** S3G3656.D
- **Extraction:** SEPF
- **Mod. Ref No.:** SDG No.: SJ0464
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J0464
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL)
GPC Cleanup: (Y/N) N
pH: Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION UNITS: (ug/L or ug/Kg)</th>
<th>Q</th>
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</thead>
<tbody>
<tr>
<td>91-20-3</td>
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<td></td>
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<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>41</td>
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</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>44</td>
<td></td>
</tr>
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</table>
### Analyses

**SW846 8015 -- Gasoline Range Organic (GRO) by GC-FID**

<table>
<thead>
<tr>
<th>Result</th>
<th>Qual</th>
<th>RL Units</th>
<th>DF</th>
<th>Date Analyzed</th>
<th>Batch ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gasoline Range Organics</td>
<td>55 u</td>
<td>50 μg/L</td>
<td>1 03/24/2010 11:42</td>
<td>1 03/24/2010 11:42</td>
<td>50058</td>
</tr>
<tr>
<td>Surrogate: Bromofluorobenzene</td>
<td>98.1</td>
<td>87-112 %REC</td>
<td>1 03/24/2010 11:42</td>
<td>1 03/24/2010 11:42</td>
<td>50058</td>
</tr>
</tbody>
</table>

**Qualifiers:**
- **ND**: Not Detected at the Reporting Limit
- **J**: Analyte detected below quantitation limits
- **B**: Analyte detected in the associated Method Blank
- **DF**: Dilution Factor
- **S**: Spike Recovery outside accepted recovery limits
- **R**: RPD outside accepted recovery limits
- **E**: Value above quantitation range
- **RL**: Reporting Limit

---

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Mitkem Laboratories

Date: 01-Apr-10

Client: CH2M Hill, Inc.
Client Sample ID: VWAE-EB01-031610
Lab ID: J0464-02
Project: CTO-0083 Vieques AOC E and I
Collection Date: 03/16/10 12:15

<table>
<thead>
<tr>
<th>Analyses</th>
<th>Result</th>
<th>Qual</th>
<th>RL</th>
<th>Units</th>
<th>DF</th>
<th>Date Analyzed</th>
<th>Batch ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>SW846 8015 -- Gasoline Range Organic (GRO) by GC-FID</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Gasoline Range Organics</td>
<td>110</td>
<td></td>
<td>50</td>
<td>ug/L</td>
<td></td>
<td>03/24/2010 13:29</td>
<td>50056</td>
</tr>
<tr>
<td>Surrogate: Bromofluorobenzene</td>
<td>91.3</td>
<td></td>
<td>67-112</td>
<td>%REC</td>
<td></td>
<td>03/24/2010 13:29</td>
<td>50056</td>
</tr>
</tbody>
</table>

Qualifiers: ND - Not Detected at the Reporting Limit
J - Analyte detected below quantitation limits
B - Analyte detected in the associated Method Blank
DF - Dilution Factor
S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
E - Value above quantitation range
RL - Reporting Limit
<table>
<thead>
<tr>
<th>Analyses</th>
<th>Result</th>
<th>Qual</th>
<th>RL Units</th>
<th>DF Date Analyzed</th>
<th>Batch ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>SW846 8015 -- Gasoline Range Organic (GRO) by GC-FID</td>
<td>ND</td>
<td>ND</td>
<td>50 ug/L</td>
<td>03/24/2010 14:38</td>
<td>GRO_W</td>
</tr>
<tr>
<td>Gasoline Range Organics</td>
<td>ND</td>
<td>ND</td>
<td>50 ug/L</td>
<td>03/24/2010 14:38</td>
<td>GRO_W</td>
</tr>
<tr>
<td>Surrogate: Bromofluorobenzene</td>
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<td>87-112</td>
<td>%REC</td>
<td>03/24/2010 14:38</td>
<td>50058</td>
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</tbody>
</table>

**Qualifiers:**
- ND - Not Detected at the Reporting Limit
- J - Analyte detected below quantitation limits
- B - Analyte detected in the associated Method Blank
- DF - Dilution Factor
- S - Spike Recovery outside accepted recovery limits
- R - RPD outside accepted recovery limits
- E - Value above quantitation range
- RL - Reporting Limit
## Analyses

**SW846 8015 -- Gasoline Range Organic (GRO) by GC-FID**

<table>
<thead>
<tr>
<th>SW846 8015</th>
<th>Gasoline Range Organics</th>
<th>250</th>
<th>50 ug/L</th>
<th>1/24/2010 15:14</th>
<th>50058</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surrogate: Bromofluorobenzene</td>
<td>96.0</td>
<td>87-112 %REC</td>
<td>1/24/2010 15:14</td>
<td>50058</td>
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### Qualifiers

- **ND**: Not Detected at the Reporting Limit
- **S**: Spike Recovery outside accepted recovery limits
- **I**: Analyte detected below quantitation limits
- **R**: RPD outside accepted recovery limits
- **B**: Analyte detected in the associated Method Blank
- **E**: Value above quantitation range
- **RL**: Reporting Limit
- **DF**: Dilution Factor
Mitkem Laboratories

Client: CH2M Hill, Inc.
Client Sample ID: VWAE-MW04-0310
Lab ID: J0464-05

Date: 01-Apr-10

Project: CTO-0083 Vieques AOC E and I
Collection Date: 03/17/10 9:10

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<td>SW846 8015 -- Gasoline Range Organic (GRO) by GC-FID</td>
<td>65</td>
<td>50 ug/L</td>
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<td>Surrogate: Bromoform</td>
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Qualifiers:
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<th>Batch ID</th>
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Qualifiers:
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### Analysts

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### Qualifiers:
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Qualifiers:
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- DF - Dilution Factor
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- R - RPD outside accepted recovery limits
- E - Value above quantitation range
- RL - Reporting Limit
MITKEM LABORATORIES

Date: 01-Apr-10

Client: CH2M Hill, Inc.
Client Sample ID: VWAE-MW01-0310
Lab ID: J0464-09
Project: CTO-0083 Vieques AOC E and I
Collection Date: 03/17/10 12:15

<table>
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<tr>
<th>Analyses</th>
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Surrogate: Bromofluorobenzene

Qualifiers:
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- B - Analyte detected in the associated Method Blank
- DF - Dilution Factor
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- R - RPD outside accepted recovery limits
- E - Value above quantitation range
- RL - Reporting Limit
Mitkem Laboratories

Client: CH2M Hill, Inc.
Client Sample ID: VWAE-MW03-0310
Lab ID: J0464-01

Date: 13-Apr-10
Project: CTO-0083 Vieques AOC E and I
Collection Date: 03/16/10 9:40

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<th>Batch ID</th>
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<tr>
<td>SW846 8015B – Total Petroleum Hydrocarbons (TPH) by GC-FID</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Extractable Total Petroleum Hydrocarbon</td>
<td>ND</td>
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<td>0.35 mg/L</td>
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<td>Oil Range Organics</td>
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<td>Surrogate: 5a-Androstane</td>
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<td>30-110 %REC</td>
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Qualifiers: ND - Not Detected at the Reporting Limit
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R - RPD outside accepted recovery limits
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## Analyses

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<tbody>
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<td>Extractable Total Petroleum Hydrocarbon</td>
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<td>0.35 mg/L</td>
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- RL - Reporting Limit
Mitkem Laboratories

Client: CH2M Hill, Inc.

Client Sample ID: VWAE-MW05-0310
Lab ID: J0464-04

Date: 13-Apr-10

Project: CTO-0083 Vieques AOC E and I
Collection Date: 03/17/10 6:50

Analyses | Result | Qual | RL Units | DF | Date Analyzed | Batch ID
---|---|---|---|---|---|---
SW846 80158 -- Total Petroleum Hydrocarbons (TPH) by GC-FID
Extractable Total Petroleum Hydrocarbon | 1.3 | ND | 0.35 mg/L | 1 03/23/2010 23:00 | 49996
Oil Range Organics | ND | 0.35 mg/L | 1 03/23/2010 23:00 | 49996
Surrogate: o-terphenyl | 64.3 | ND | 50-150 %REC | 1 03/23/2010 23:00 | 49996
Surrogate: 5a-Androstan | 33.1 | ND | 30-110 %REC | 1 03/23/2010 23:00 | 49996

Qualifiers: ND - Not Detected at the Reporting Limit
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R - RPD outside accepted recovery limits
B - Analyte detected in the associated Method Blank
E - Value above quantitation range
DF - Dilution Factor
RL - Reporting Limit
## Mitkem Laboratories

**Date:** 13-Apr-10  
**Client:** CH2M Hill, Inc.  
**Client Sample ID:** VWAE-MW04-0310  
**Lab ID:** J0464-05  
**Project:** CTO-0083 Vieques AOC E and I  
**Collection Date:** 03/17/10 9:10

### Analyses

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<th>Result</th>
<th>Qual</th>
<th>RL Units</th>
<th>DF</th>
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<tr>
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### Qualifiers:
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Mitkem Laboratories

Client: CH2M Hill, Inc.
Client Sample ID: VWAE-MW4P-0310
Lab ID: J0464-06

Project: CTO-0083 Vieques AOC E and I
Collection Date: 03/17/10 9:15

Date: 13-Apr-10

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<th>Batch ID</th>
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Mitkem Laboratories  

Date: 13-Apr-10

Client: CH2M Hill, Inc.  
Client Sample ID: VWAE-EB01-031710  
Lab ID: J0464-07  
Project: CTO-0083 Vieques AOC E and I  
Collection Date: 03/17/10 9:35

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- B - Analyte detected in the associated method blank
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- RL - Reporting Limit

---

Mitkem Laboratories

Client: CH2M Hill, Inc.
Client Sample ID: VWAE-MW01-0310
Lab ID: J0464-09
Date: 13-Apr-10

Project: CTO-0083 Vieques AOC E and I
Collection Date: 03/17/10 12:15

---

0586 067
Lab Name: Mitkem Laboratories
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

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<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
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Lab Name: Mitkem Laboratories
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

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<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
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Comments:

ISM_002 FORM IA - IN ILM05.4
Lab Name: Mitkem Laboratories
Lab Code: MITKEM

Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): ug/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
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<tbody>
<tr>
<td>7439-89-6</td>
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Comments:

ISM_002

FORM IA - IN

ILM05.4

0644
**Lab Name:** Mitkem Laboratories
**Lab Code:** MITKEM
**Case No.:**
**Contract:** 933562, N62
**NRAS No.:**
**SDG No.:** SJ0464

**Matrix (soil/water):** WATER
**Lab Sample ID:** J0464-04
**Level (low/med):** MED
**Date Received:** 03/18/2010

% Solids: 0.0

**Concentration Units (ug/L or mg/kg dry weight):** UG/L

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<th>O</th>
<th>M</th>
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Comments:
Lab Name: Mitkem Laboratories
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

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<th>CAS No.</th>
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Comments:

0647
Lab Name: Mitkem Laboratories
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0
Concentration Units (ug/L or mg/kg dry weight): UC/L

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Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

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Comments:

Date Received: 03/20/2010

Lab Name: Mitkem Laboratories
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0
Concentration Units (ug/L or mg/kg dry weight): UG/L

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Comments:

SC 030
Mitkem Laboratories, a Division of Spectrum Analytical, Inc. submits the enclosed data package in response to CH2M Hill’s 1000-CTO-0083, Vieques project. Under this deliverable, analysis results are presented for twenty-two samples that were received at Mitkem from March 17 to March 23, 2010 and logged in under Mitkem Work Order Number J0464. The sample was analyzed per instructions in the chain of custody forms and instruction from client.

The analyses were performed according to EPA SW-846 methods, with this hardcopy report produced in a CLP-type format for Level 4 deliverable with the exception of gasoline range organics and total petroleum hydrocarbons. The analysis results for gasoline range organics and total petroleum hydrocarbons are presented in the standard Mitkem format with supporting raw data.

The following observation and/or deviations are observed for the following analyses:

1. Total Volatile Analysis:

Soil samples were analyzed by Method 8260C for a select list of volatile organic compounds.

Surrogate recovery: recoveries were within the QC limits.

Lab control sample/lab control sample duplicate: spike recoveries were within the QC limits.

Matrix spike/matrix spike duplicate: duplicate matrix spikes were performed on sample VWAE-MW03-0310. Spike recovery and replicate RPD were within the QC limits.

Sample analysis: due to the high concentration of target analytes, sample VWAE-MW05-0310 was re-analyzed at 5x dilution. No other unusual observation was made for the analysis.

2. GRO Analysis:

Samples were analyzed for Gasoline Range Organics (GRO) by the purgable organics option of SW846 Method 8015. GRO includes all resolved and unresolved compounds eluting between the retention times of MTBE and naphthalene inclusive. The instrument is calibrated using an average response factor obtained from injections of a mixture of individual analytes. The lab control sample is spiked with gasoline product.

Surrogate recovery: spike recovery was within the QC limits.
Lab control sample/lab control sample duplicate: spike recovery was within the QC limits.

Matrix spike/matrix spike duplicate: duplicate matrix spikes were performed on sample VWAE-MW03-0310. Spike recovery and replicate RPD were within the QC limits.

Sample analysis: no unusual observation was made for the analysis.

3. Semivolatile Analysis:

The samples analyzed for naphthalene and 2-methylnaphthalene by Method 8270D.

Surrogate recovery: recoveries were within the QC limits.

Lab control sample/lab control sample duplicate: spike recoveries were within the QC limits.

Matrix spike/matrix spike duplicate: duplicate matrix spikes were performed on samples VWAE-MW03-0310 and VWAI-MW02-0310. Spike recoveries and replicate RPDs were within the QC limits for both samples.

Sample analysis: no unusual observation was made for the analysis.

4. TPH Analysis:

The samples were analyzed for extractable Total Petroleum Hydrocarbons (TPH) by the extractable organics option of SW846 Method 8015. TPH includes all resolved and unresolved compounds eluting between the retention times of C9 and C36 inclusive. The instrument is calibrated using an average response factor obtained from injections of a mixture of individual n-alkanes. The lab control sample is spiked with diesel fuel product.

Surrogate recovery: spike recoveries were within the QC limits.

Lab control sample: spike recovery was within the QC limits.

Matrix spike/matrix spike duplicate: duplicate matrix spikes were performed on sample VWAE-MW03-0310. Spike recovery and replicate RPD were within the QC limits.

Sample analysis: no unusual observation was made for the analysis.

5. Metals Analysis:

Samples were analyzed for iron and manganese by SW-846 method 6010C.
Lab control sample: spike recoveries were within the QC limits.

Matrix spike: matrix spike was performed on sample VWAE-MW03-0310. Spike recoveries were within the QC limits.

Duplicate: duplicate analysis was performed on sample VWAE-MW03-0310. Replicate RPDs were within the QC limits.

Sample analysis: serial dilution was performed on sample VWAE-MW03-0310. Percent differences were within the QC limits. No unusual observations were made during sample analysis.

6. Wet Chemistry Analyses:

Samples were analyzed for the anions nitrate, and sulfate by EPA Method 300.0 and total organic carbon by SM5310B.

Laboratory control sample: percent recoveries were within the QC limits.

Matrix spike/matrix spike duplicate: duplicate matrix spikes were performed on samples VWAE-MW03-0310, VWAE-MW03-0310, VWAI-MW02-0310 and VWAI-MW07-0310 for anions. Percent recoveries and percent RPDs were within the QC limits.

Matrix spike/matrix duplicate: matrix spike and matrix duplicate analyses were performed on sample VWAE-MW03-0310 total organic carbon. Spike recovery and percent RPD were within the QC limits.

Sample analysis: the diluted analysis for nitrate in sample VWAE-MW03-0310 was performed outside of the 48-hour hold time. The initial analysis was performed within hold time. Both the initial and diluted analysis have been reported for VWAE-MW03-0310. Nitrate and sulfate were detected in method blanks MB-49893, -49929, -49960, -49979 and -50060 below the reporting limit but above the method detection limit. Samples associated with these blanks also contained nitrate and/or sulfate, either at concentrations below the reporting limit, or more than 10X the method blank concentration, indicating no significant impact of laboratory background levels on sample results. Sample results associated with these blanks are qualified with the "B". No other unusual occurrences were noted during sample analysis.

7. CENSUS and PLFA Analyses:

CENSUS and PLFA analyses were performed by MicorbiialInsights of Rockford, TN. The entire MicorbiialInsights report, including any notes on these analyses is enclosed.
All pages in this report have been numbered consecutively, starting with the title page and ending with a page saying only “Last Page of Data Report”. The Columbia data report is paginated separately, following the “Last” page.

I certify that this data package is in compliance, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the laboratory manager or his designee, as verified by the following signature.

[signature]

Agnes Huntley
CLP Project Manager
04/14/10
**CHAIN OF CUSTODY RECORD**

**Page 1 of 1**

---

**Report To:** Juan Acron / CNV
3011 S.W. Williams Blvd
Gainesville, FL 32607

**Telephone #:** (352) 335-7991

**Project Mgr.:** Stephen Bond / V80

---

**Invoice To:** Denver, CO (Ham Hill)

(See Contract)

---

**Project No.:** 1000 - C70-00023 Vieques

**Site Name:** Vieques AOC E

**Location:** Vieques AOC E

**State:** Pr

**Sample(s):** Dia Whittaker / Michael Zambon

---

<table>
<thead>
<tr>
<th>DW = Drinking Water</th>
<th>GW = Groundwater</th>
<th>WW = Wastewater</th>
<th>O = Oil</th>
<th>SW = Surface Water</th>
<th>SO = Soil</th>
<th>SL = Sludge</th>
<th>A = Air</th>
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<tbody>
<tr>
<td>1 = Na₂SO₃</td>
<td>2 = HCl</td>
<td>3 = H₂SO₄</td>
<td>4 = HNO₃</td>
<td>5 = NaOH</td>
<td>6 = Ascorbic Acid</td>
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<td>8 = NaHSO₄</td>
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**Special Handling:**
- Standard TAT - 7 to 10 business days
- Rush TAT - Date Needed
  - All TATs subject to laboratory approval.
  - Min. 24-hour notification needed for rush.
  - Samples disposed of after 60 days unless otherwise instructed.

---

**List preservative code below:**

2 x 4 x 2 x 9

---

**QA/QC Reporting Notes:**

- Provide MA DEP MCP CAM Report
- Provide CT DPH RCP Report

**QA/QC Reporting Level:**

- Standard
- Other Level IV

---

**State specific reporting standards:**

- Temperature: Ambient
- Container: G = Grab, C = Composite
- Matrices: 
  - G = Grab
  - A = Air
  - S = Soil
  - O = Oil
  - W = Wastewater
  - D = Drinking Water

---

**Lab Id:** WAE-V005-0010
**Sample Id:** WAE-V005-0010
**Date:** 3/16/10
**Time:** 09:40
**Type:** G
**Matrix:** GW
**# of VOA Vials:** 12
**# of Amber Glass:** 6
**# of Plastic:** 6

---

**Analyses:**

- WAE-V005-0010
- WAE-V005-0050
- WAE-V005-0010

---

**Temperature Blank (1/10):** Special Standards on SVOCs. Compound list are reduced and QLS are set by VFP-JAP.

---

**Relinquished by:** Michael Zambon 3/16/10
**Date:** 3/16/10
**Time:** 12:30
**Received by:** Anna Del

---

**E-mail to:** JAcron@cliam.com

---

**Additional Notes:**

- EDD Format: SNEDO
- Ambient:
- Refrigerated:
- Fridge temp °C
- Freezer temp °C

---

11 Almgren Drive • Agawam, MA 01001 • 413-789-9018 • FAX 413-789-4076 • www.spectrum-analytical.com
# CHAIN OF CUSTODY RECORD

**Report To:** Juan Airosa (CNV)
3011 SW Villisca Rd.
Bainbridge, FL 32618

**Telephone #:** (352) 335-7999
**Project Mgr.:** Stephen Brand/N80

**Invoice To:** Denver, CO (M&H Hill)
(5th Contract)

**Location:** West Viques

**Site Name:** Viques AOC E

**Project No.:** 1000 - CTO-0083 Viques

**Sample(s):** Diana Whitaker/Michael Zamboni

### Standard TAT
- Rush TAT - Date Needed:

- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rushes.
- Samples disposed of after 60 days unless otherwise instructed.

---

## Preservative Code

List preservative code below:

- 2: X 4: 2: 9

## Containers

- G: Grab
- C: Composite
- X1: 
- X2: 
- X3: 

## Analyses

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**QA/QC Reporting Notes:**

- Provide MA DEP MCP CAM Report
- Provide CT DPH RCP Report

**QA/QC Reporting Level**

- Standard
- No QC

**State specific reporting standards:**

- **Temperature Blank:** (1/cool). Special standards for SWQA. Company must be reduced and QLS are set.

---

**Relinquished by:** Michael Zamboni 3/17/10 13:00
**Received by:** Ana Arata 3/18/10 09:15

**Date:** 3/18/10
**Time:** 09:15
**Temp°C:** 5.3

- [ ] Ambient
- [ ] Iced
- [ ] Refrigerated
- [ ] Fridge temp. **°C**
- [ ] Freezer temp. **°C**

---

**Special Handling:** 28 Calendar

---

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CHAIN OF CUSTODY RECORD

Page 1 of 1

Special Handling: 28 Calendar Day

- Standard TAT - 2 to 10 business days
- Rush TAT - Date Needed:
  - All TATs subject to laboratory approval.
  - Min. 24-hour notification needed for rushes.
  - Samples disposed of after 60 days unless otherwise instructed.

Report To: Juan Aaron / GVU
3011 SW Williams Rd
Gainesville, FL 32608

Telephone #: (352) 335-799
Project Mgr: Skphin Brand/VOO

Invoice To: Denver, CO (Jim Hill)

(See Contact)

P.O. No.: RQN:

Project No.: 1000-CT0-0083 Virgo

Site Name: Virgo AOC 1

Location: West Virgo

Sampler(s): Dia Whitaker / Michael Zamboni

List preservative code below:

QA/QC Reporting Notes:
(check as needed)

- Provide MA DEP MCP CAM Report
- Provide CT DPH RCP Report
- QA/QC Reporting Level
  - Standard
  - Other

State specific reporting standards:

Containers:

Analyses:

- AQVA vials, run QA/QC
- MS
- MSD

DW = Drinking Water
GW = Groundwater
WW = Wastewater
O = Oil
SW = Surface Water
SO = Soil
SL = Sludge
A = Air
X1 = 
X2 = 
X3 = 

G = Grab
C = Composite

Lab Id: Sample Id: Date: Time: Type: Matrix

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</table>

Temperature Alert: [Color]. Special Standards for SVUs. Compound lists are reduced to CALs.

Relinquished by: 3/19/10 13:00

Received by: 3/18/10 8:52

E-mail to: JAaron@CHM.com

- Ambient
- Refrigerated
- Fridge temp __ °C
- Freezer temp __ °C

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**CHAIN OF CUSTODY RECORD**

Page 1 of 1

<table>
<thead>
<tr>
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<th>Invoice To:</th>
<th>Project No.:</th>
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<tbody>
<tr>
<td>Juan Acuna, CNW</td>
<td>Denver, CO (HimHill)</td>
<td>1000 - CTO - 0023 Viruges</td>
</tr>
<tr>
<td>3011 SW Williamson Road, Gaineville, FL 32608</td>
<td>(See Contact)</td>
<td>Site Name: Viruges APC I</td>
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<tr>
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<td>Location: Viruges APC I</td>
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<td>Sampler(s): Sue Viques/Michael Lambon</td>
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All TATs subject to laboratory approval. Min. 24-hour notification needed for rushes.

Samples disposed of after 60 days unless otherwise instructed.

---

### SPECTRUM ANALYTICAL, INC.

**Telephone #:**

11 Almgren Drive • Agawam, MA 01001 • 413-789-9018 • FAX 413-789-4076 • www.spectrum-analytical.com

---

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<th>Number of Clear Glass</th>
<th>Number of Plastic</th>
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<td>□ Rush TAT - Date Needed:</td>
</tr>
<tr>
<td>- All TATs subject to laboratory approval. Min. 24-hour notification needed for rushes.</td>
</tr>
</tbody>
</table>

State specific reporting standards:

- Standard TAT: 7 to 10 business days
- Rush TAT: Date Needed (subject to laboratory approval)

---

**SPECTRUM ANALYTICAL, INC.**

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**CHAIN OF CUSTODY RECORD**

Report To: **JUAN ACACOROY**  
3011 SW WILSON ROAD  
GAINESVILLE, FL 32608

Invoice To: **Denver, Colorado (CH2M Hill)**  
(see contract)

Telephone #: (352) 335-7991  

P.O. No.: RQN:

Project No.: **1000 - CTO-0085**

Site Name: **Vieques AOC**

Location: **Vieques AOC**  
State: P.R.

Sampler(s): **D. Whimack M. Zamboni**

<table>
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<td>3</td>
<td>( \text{H}_2\text{SO}_4 )</td>
</tr>
<tr>
<td>4</td>
<td>( \text{HNO}_3 )</td>
</tr>
<tr>
<td>5</td>
<td>( \text{NaOH} )</td>
</tr>
<tr>
<td>6</td>
<td>Ascorbic Acid</td>
</tr>
<tr>
<td>7</td>
<td>( \text{CH}_3\text{OH} )</td>
</tr>
<tr>
<td>8</td>
<td>( \text{NaHSO}_4 )</td>
</tr>
<tr>
<td>9</td>
<td>( \text{H,Fb} )</td>
</tr>
</tbody>
</table>

**QA/QC Reporting Notes:**

- **G** = Grab  
- **C** = Composite

**Containers:**

<table>
<thead>
<tr>
<th># of VOA Vials</th>
<th># of Amber Glass</th>
<th># of Clear Glass</th>
<th># of Plastic</th>
</tr>
</thead>
<tbody>
<tr>
<td>X1 = AQ</td>
<td>X2 =</td>
<td>X3 =</td>
<td></td>
</tr>
</tbody>
</table>

**Analyses:**

- **List I 8200-B**
- **List II 7100OC**
- **List III**
- **List IV**
- **List V**
- **List VI**

**Other Reporting Level:**

- **Standard**  
- **No QC**

State specific reporting standards:

**Lab Id:**  

<table>
<thead>
<tr>
<th>Sample Id:</th>
<th>Date:</th>
<th>Time:</th>
<th>Type</th>
<th>Matrix</th>
<th>Temp°C</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>VNAI-MH07-0310</strong></td>
<td>03/22/10</td>
<td>0950</td>
<td>G</td>
<td>GW</td>
<td>4</td>
</tr>
<tr>
<td><strong>VNAI-MH07-0310</strong></td>
<td>1050</td>
<td>6</td>
<td>AQ</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td><strong>VNAI-MH07-0310</strong></td>
<td>1055</td>
<td>6</td>
<td>AQ</td>
<td>2</td>
<td></td>
</tr>
</tbody>
</table>

**QA/QC Reporting Level:**

- **Standard**  
- **No QC**

**Special Handling:**

- **Standard TAT** - 7-10 business days  
- **Rush TAT** - Date Needed  
- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rushes.
- Samples disposed of after 60 days unless otherwise instructed.
# Sample Condition Form

<table>
<thead>
<tr>
<th>Received By:</th>
<th>Reviewed By:</th>
<th>Date:</th>
<th>Work Order #:</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Name]</td>
<td>[Name]</td>
<td>3/17/19</td>
<td>J0466</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Client Project:</th>
<th>Client:</th>
<th>CH2M</th>
</tr>
</thead>
<tbody>
<tr>
<td>UIQUEES</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Preservative Name/Lot No.:</th>
<th>VOA Matrix Key:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>US = Unpreserved Soil</td>
</tr>
<tr>
<td></td>
<td>UA = Unpreserved Aqueous</td>
</tr>
<tr>
<td></td>
<td>M = MeOH</td>
</tr>
<tr>
<td></td>
<td>N = NaHSO4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Lab Sample ID</th>
<th>Preservation (pH)</th>
<th>VOA Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>J0464 01</td>
<td>HNO₃ H₂SO₄ HCl NaOH H₃PO₄</td>
<td>1/4&quot;</td>
</tr>
<tr>
<td>J0464 02</td>
<td></td>
<td></td>
</tr>
<tr>
<td>J0464 03</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>1) Cooler Sealed</th>
<th>Yes / No</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Yes</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>2) Custody Seal(s)</th>
<th>Present / Absent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Custody Seal(s)</td>
<td>Intact / Broken</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>3) Custody Seal Number(s)</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>4) Chain-of-Custody</th>
<th>Present / Absent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Absent</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>5) Cooler Temperature</th>
<th>3°C, 3°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>IR Temp Gun ID</td>
<td>MT-1</td>
</tr>
<tr>
<td>Coolant Condition</td>
<td>ICED</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>6) Airbill(s)</th>
<th>Present / Absent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Airbill Number(s)</td>
<td>FEDEX</td>
</tr>
<tr>
<td></td>
<td>865861519120</td>
</tr>
<tr>
<td></td>
<td>86029419779</td>
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</table>

<table>
<thead>
<tr>
<th>7) Samples Bottles</th>
<th>Intact / Broken / Leaking</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Broken</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>8) Date Received</th>
<th>3/17/19</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>9) Time Received</th>
<th>2:05</th>
</tr>
</thead>
</table>

See Sample Condition Notification/Corrective Action Form: yes / no | Rad OK: yes / no |
### Sample Condition Form

**Received By:** AED  
**Reviewed By:** SW  
**Date:**  
**Mittem Work Order #:** J0464

**Client Project:** CTO - 607 V1:QUE:5

**Client:** CH2M

---

<table>
<thead>
<tr>
<th>No</th>
<th>Item</th>
<th>Description</th>
<th>Status</th>
<th>Lab Sample ID</th>
<th>Preservation (pH)</th>
<th>VOA Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1) Cooler Sealed</td>
<td>Yes / No</td>
<td></td>
<td>J0464</td>
<td>04 22</td>
<td>H</td>
</tr>
<tr>
<td>2</td>
<td>2) Custody Seal(s)</td>
<td>Present / Absent</td>
<td></td>
<td></td>
<td>06 22</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Coolers / Bottles</td>
<td>Intact / Broken</td>
<td></td>
<td></td>
<td>07 22</td>
<td></td>
</tr>
<tr>
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<td>3) Custody Seal Number(s)</td>
<td>NA</td>
<td></td>
<td>J0464</td>
<td>04 22</td>
<td></td>
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<tr>
<td>4</td>
<td>4) Chain-of-Custody</td>
<td>Present / Absent</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>5) Cooler Temperature</td>
<td>2°C, 3°C, 5°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>IR Temp Gun ID</td>
<td>MT-1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Coolant Condition</td>
<td>ICED</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>6) Airbill(s)</td>
<td>Present / Absent</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Airbill Number(s)</td>
<td>FEDE&lt; 8640 9096 4029 8640 9096 4030 8640 9096 4018</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>7) Samples Bottles</td>
<td>Intact / Broken / Leaking</td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>8</td>
<td>8) Date Received</td>
<td>3/18/10</td>
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<tr>
<td>9</td>
<td>9) Time Received</td>
<td>9:15</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Preservative Name/Lot No.:**

**VOA Matrix Key:**
- **US** = Unpreserved Soil
- **A** = Air
- **UA** = Unpreserved Aqueous
- **H** = HCl
- **M** = MeOH
- **E** = Encore
- **N** = NaH2SO4
- **F** = Freeze

**See Sample Condition Notification/Corrective Action Form** yes / no

**Rad OK** yes / no
**Sample Condition Form**

<table>
<thead>
<tr>
<th>Received By:</th>
<th>AED</th>
<th>Reviewed By:</th>
<th>Date: 3/19/10</th>
<th>Mittem Work Order #: J0464</th>
</tr>
</thead>
<tbody>
<tr>
<td>Client Project:</td>
<td>CTD - 053</td>
<td>Unique</td>
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</table>

### Lab Sample ID

<table>
<thead>
<tr>
<th>Preservation (pH)</th>
<th>Lab Sample ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>HNO₃</td>
<td>J0464 10</td>
</tr>
<tr>
<td>H₂SO₄</td>
<td>11 1.2 2.2</td>
</tr>
<tr>
<td>HCl</td>
<td>12 1.3</td>
</tr>
<tr>
<td>NaOH</td>
<td></td>
</tr>
<tr>
<td>H₃PO₄</td>
<td>2</td>
</tr>
<tr>
<td>VOA Matrix</td>
<td>H</td>
</tr>
<tr>
<td>Soil Headspace or Air Bubble ≥ 1/4&quot;</td>
<td></td>
</tr>
</tbody>
</table>

### 1) Cooler Sealed

- Yes/No: Yes

### 2) Custody Seal(s)

- Present/Absent: Absent
- Coolers/Bottles: Intact/Broken

### 3) Custody Seal Number(s)

- N/A

### 4) Chain-of-Custody

- Present/Absent: Present/Absent

### 5) Cooler Temperature

- 4°C, ≤ 5°C
- IR Temp Gun ID: MT-1
- Coolant Condition: ICED

### 6) Airbill(s)

- Present/Absent: Present
- Airbill Number(s): FEDEX
  - 865861519141
  - 865861519152

### 7) Samples Bottles

- Intact/Broken/Leaking: Leaking

### 8) Date Received

- 3/19/10

### 9) Time Received

- 8:52

Preservative Name/Lot No.: 

---

VOA Matrix Key:

- US = Unpreserved Soil
- A = Air
- UA = Unpreserved Aqueous
- H = HCl
- M = MeOH
- E = Encore
- N = NaHSO₄
- F = Freeze

---

See Sample Condition Notification/Corrective Action Form yes/no

Form ID: OAIF.0006

[Files] Documents And Settings\sopeace\WARWICK\Local Settings\Temporary Internet Files\OUI M A I R Sample Condition form.xls

Rej OK yea no

---

0019
<table>
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<th>Sample Condition Form</th>
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<tbody>
<tr>
<td><strong>Received By:</strong> AEO</td>
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<tr>
<td><strong>Client Project:</strong> CTO 007</td>
</tr>
<tr>
<td><strong>Client:</strong> CHZM</td>
</tr>
<tr>
<td><strong>Lab Sample ID:</strong></td>
</tr>
<tr>
<td>J0464</td>
</tr>
<tr>
<td>J0463</td>
</tr>
</tbody>
</table>

| **Soil Preservation (pH):** |
| **VOA Matrix:** |
| H | |

| **Cooler Sealed:** Yes/No |
| **Headspace or Air Bubble ≥ 1/4”:** |
| No |

| **Custody Seal(s):** Present/Absent |
| **Coolers / Bottles Intact / Broken:** |
| Absent |

| **Custody Seal Number(s):** |

| **Chain-of-Custody:** Present/Absent |

| **Cooler Temperature:** |
| **IR Temp Gun ID:** |
| 4°C | M5-1 |

| **Coolant Condition:** |
| **Airbill(s):** Present/Absent |
| **Airbill Number(s):** |
| FEDEX | B6584519/85 |

| **Samples Bottles:** Intact / Broken / Leaking |

| **Date Received:** |
| **Time Received:** |
| 3/23/10 | 16:58 |

| **Preservative Name/Lot No.:** |

| **VOA Matrix Key:** |
| US = Unpreserved Soil | A = Air |
| UA = Unpreserved Aqueous | H = HCl |
| M = MeOH | E = Encore |
| N = NaHSO4 | F = Freeze |

See Sample Condition Notification/Corrective Action Form: yes/no
I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: SJ0464  LAB: Mitrkon Labs
SITE NAME: Vieque AOC E C10-83

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent? [ ] Y [ ] N [ ] N/A

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, and/or cover letter signed release present? [ ] Y [ ] N [ ] N/A

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? [ ] Y [ ] N [ ] N/A

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, and/or Chain of Custodies from the field samplers present for all samples sign release present? [ ] Y [ ] N [ ] N/A

ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)? [ ] Y [ ] N [ ] N/A

1.3 Sample Conditions/Problems
1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?  

ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects non"UJ".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

The maximum holding time for aqueous samples is 14 days.

The maximum holding time for soils non aqueous samples is 14 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with HCL acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. For non-aqueous samples for volatile components that are frozen (less than 7°C) or are properly cooled (4°C ± 2°C) and perserved with NaHSO₄, the maximum holding time is 14 days from sample collection. If
uncertain about preservation, contact the laboratory
/sampling team to determine whether or not samples were
preserved.

ACTION: Qualify sample results according to Table 1:

Table 1. Holding Time Actions for Trace Volatile Analysis

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Preserved</th>
<th>Criteria</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Detected</td>
<td>Non-Detected</td>
<td>Associated</td>
</tr>
<tr>
<td></td>
<td>Compounds</td>
<td>Compounds</td>
<td></td>
</tr>
<tr>
<td>Aqueous</td>
<td>No</td>
<td>≤7 days</td>
<td>No qualifications</td>
</tr>
<tr>
<td></td>
<td>No</td>
<td>&gt;7 days</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>&gt;14 days</td>
<td>J</td>
</tr>
<tr>
<td>Non Aqueous</td>
<td>No</td>
<td>≤14 days</td>
<td>R</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
</tr>
<tr>
<td></td>
<td>Yes/No</td>
<td>&gt;14 days</td>
<td>J</td>
</tr>
</tbody>
</table>

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Water
b. Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:

a. Water
b. Soil

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data
3.3 Were the surrogate recovery limits followed per Table 2. If Table 2 criteria were not followed, the laboratory may use in-house performance criteria (per SW-846, Method 8000C, section 9.7). Other compounds may be used as surrogates, depending upon the analysis requirements.

Table 2. Surrogate Spike Recovery Limits for Water and Soil/Sediments

<table>
<thead>
<tr>
<th>DMC</th>
<th>Recovery Limits (%)Water</th>
<th>Recovery Limits Soil/Sediment</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Bromofluorobenzene</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dibromofluoromethane</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Toluene-d₈</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dichloroethane-d₈</td>
<td>80-120</td>
<td>70-130</td>
</tr>
</tbody>
</table>

Note: Use above table if laboratory did not provide in-house recovery criteria.

Note: Other compounds may be used as surrogates depending upon the analysis requirements.

3.4 Were outliers marked correctly with an asterisk?

ACTION: Circle all outliers with a red pencil.

3.5 Were one or more volatile surrogate recoveries out of specification for any sample or method blank. Table 2.

If yes, were samples reanalyzed?

Were method blanks reanalyzed?
ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects, but qualify positive results as estimated "J".

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. If one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose the blank problem to be an isolated occurrence.

3.6 Are there any transcription/calculation errors between raw data and reported data? [ ] [X] [ ] [N/A]

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Sample (Form III/Equivalent)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG?

[ ] [X] [ ] [N/A]
Note: LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation/resubmittals. Make note in the data assessment.

4.2 Were the Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

A. Water

B. Soil

C. Med Soil

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

4.3 Have in house LCS recovery limits been developed (Method 8000C, Sect 9.7).

4.4 If in house limits are not developed, are LCS acceptance recovery limits between 70 - 130% (Method 8000c Sect 9.5)?

4.5 Were one or more of the volatile LCS recoveries outside the in house laboratory recovery criteria for spiked analytes? If in house limits are not present use 70 - 130% recovery limits.
Table 3. LCS Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Action</th>
<th>Detected Spiked Compounds</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td>J</td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td>UJ</td>
<td>No Qualifications</td>
</tr>
<tr>
<td>Lower Acceptance Limit %R</td>
<td>No Qualifications</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

5.0 Matrix Spikes (Form III or equivalent)

5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix?

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, Method 8260B, Sect 8.4.2).

5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III?

ACTION: If any data are missing take action as specified in section 3.2 above.

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples
of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month (page 8000C, section 9.5.)

a. Water

b. Waste

c. Soil/Solid

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The concentration of the LCS should be determined as described SW-Method 8000C Section 9.5.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

5.4 Have in house MS recovery limits been developed (Method 8000C, Sect 9.7) for each matrix.

5.5 Were one or more of the volatile MS/MSD recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000C, Sect. 9.5.4.

ACTION: Circle all outliers with a red pencil.

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.
NOTE: No qualification of data is necessary on MS and MSD data alone. However, using informed professional judgement, the data reviewer may use MS and MSD results in conjunction with other QC criteria to determine the need for some qualifications.

Note: The data reviewer should first try to determine to what extent the results of the MS and MSD affect the associated data. This determination should be made with regard to the MS and MSD sample itself, as well as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affect all associated samples, and the reviewer must use professional judgement to qualify the data from all associated samples.

Note: The reviewer must use professional judgement to determine the need for qualification of non-spiked compounds.

ACTION: Follow criteria in Table 4 when professional judgement deems qualification of sample.

Table 4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Detected Spiked Compounds</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td>UJ</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td>No Qualifications</td>
</tr>
</tbody>
</table>
6.0 Blank (CLP Form IV Equivalent)

6.1 Is the Method Blank Summary form present?  

6.2 Frequency of Analysis: Has a method blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch?  

6.3 Has a method blank been analyzed for each GC/MS system used?  

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable for volatile organic compounds?  

7.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.
7.2 Do any field/rinse blanks have positive volatile organic compound results?

**YES** ☑ **NO** ☐ **N/A**

**ACTION:** Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

**NOTE:** All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

**ACTION:** Follow the directions in Table 5 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

- VVWA-EI-B01-03-1610
- VVWA-I-B01-03-1810
- VVWA-EI-B01-03-1610
- VVWA-EI-B01-03-1710
- VVWA-EI-B01-03-1710
- VVWA-I-B01-03-1810
- VVWA-I-B01-03-1910
- VVWA-I-B01-03-2210
- VVWA-EI-B01-03-1910
- VVWA-EI-B01-03-2210
**Table 5. Volatile Organic Analysis Blank Contamination Criteria**

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td>No qualification</td>
<td></td>
</tr>
<tr>
<td>&lt; CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>&gt; CRQL*</td>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report the concentration for the sample with a U, or quantity the data as unusable R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and ≥ blank contamination</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>= CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>Gross contamination</td>
<td>Detects</td>
<td>Qualify results as unusable R</td>
<td></td>
</tr>
</tbody>
</table>

---

**NOTE:** 2x the CRQL for methylene chloride, 2-butanone, and acetone.

Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 ug/L.

If gross blank contamination exists (e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected volatile organic target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
7.3 Are there field/rinse/equipment blanks associated with every sample?  

**ACTION:** For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.

**NOTE:** For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

**ACTION:** If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?

9.3 Has an instrument performance check solution (BFB)
been analyzed for every twelve hours of sample analysis per instrument? (see Table 4, SW-846, page 8260B-36)

**YES** **NO** **N/A**

**ACTION:** List date, time, instrument ID, and sample analyses for which no associated GC/MS GC/MS tuning data are available.

**ACTION:** If the laboratory/project officer cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

**ACTION:** If mass assignment is in error, flag all associated sample data as unusable, "R".

9.4 Have the ion abundances been normalized to m/z 95?

9.5 Have the ion abundance criteria been met for each instrument used?

**ACTION:** List all data which do not meet ion abundance criteria (attach a separate sheet).

**ACTION:** If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.)

9.7 Have the appropriate number of significant figures (two) been reported?

**ACTION:** If large errors exist, take action as specified in section 3.2.

9.8 Are the spectra of the mass calibration compounds acceptable.

**ACTION:** Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.
10.0 Target Analytes (CLP Form I Equivalent)

10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:
   a. Samples and/or fractions as appropriate [ ] [ ] [ ]
   b. Matrix spikes and matrix spike duplicates [ ] [ ] [ ]
   c. Blanks [ ] [ ] [ ]
   d. Laboratory Control Samples [ ] [ ] [ ]

10.2 Are the reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?
   a. Samples and/or fractions as appropriate [ ] [ ] [ ]
   b. Matrix spikes and matrix spike duplicates (Mass spectra not required) [ ] [ ] [ ]
   c. Blanks [ ] [ ] [ ]
   d. Laboratory Control Samples [ ] [ ] [ ]

ACTION: If any data are missing, take action specified in 3.2 above.

10.3 Is chromatographic performance acceptable with respect to:
   Baseline stability? [ ] [ ] [ ]
<table>
<thead>
<tr>
<th>Resolution?</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak shape?</td>
<td>YES</td>
<td>NO</td>
<td>N/A</td>
</tr>
<tr>
<td>Full-scale graph (attenuation)?</td>
<td>YES</td>
<td>NO</td>
<td>N/A</td>
</tr>
</tbody>
</table>

**Other:** ____________________________

**ACTION:** Use professional judgement to determine the acceptability of the data.

10.4 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample? **YES**

**ACTION:** If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, contact the lab.

10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? **YES**

10.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? **YES**

10.7 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum? **YES**

**ACTION:** Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be
positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

11.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

11.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier? [ ] [ ]

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

11.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate [ ] [ ]

b. Blanks [ ] [ ]

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a CAS#.

NOTE: If TICs are present in the associated blanks take action as specified in section 3.2 above.
11.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)?

**ACTION:**
1. Flag with "R" any target compound listed as a TIC.
2. Make sure all rejected compounds are properly reported if they are target compounds.

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

11.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

**ACTION:** Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO2 (M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found?

**NOTE:** Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be
reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL's adjusted to reflect sample dilutions and, for soils, sample moisture?  

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration?  

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above.

14.0 GC/MS Initial Calibration (CLP Form VI Equivalent)
14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction?

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

ACTION: If the percent relative standard deviation (% RSD) is > 20%, (8000C-39) qualify positive results for that analyte "J". When % RSD > 90%, qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.2 Are all average RRFs > 0.050?

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

Chloromethane 0.10
1,1-Dichloroethane 0.10
Bromoform 0.10
Chlorobenzene 0.30
1,1,2,2-Tetrachloroethane 0.30

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, or for the requirements for the 5 compounds in 14.2 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration.

NOTE: (Method Requirement) For the following CCC compounds, the %RSD values must be ≤ 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment.
USEPA Region II
Date: January 2006
SW846 Method 8260B VOA
SOP: HW-24, Rev. 2

YES NO N/A

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 20.0%, or > 30% for the 6 compounds in 14.3 above, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF? [ ] [ ] [ ]

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.) [ ] [ ] [ ]

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

15.0 GC/MS Calibration Verification (CLP Form VII Equivalent)
15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest?  

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?  

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.  

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").  

15.3 Was the % D determined from the calibration verification determined using RRF or CF?  

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.  

15.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2).  

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be ≤ 20.0%. If %D values reported are > 20.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride
ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

15.5 Do any volatile compounds have a RRF < 0.05? [ ] [ ]

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

Chloromethane 0.10
1,1-Dichloroethane 0.10
Bromoform 0.10
Chlorobenzene 0.30
1,1,2,2-Tetrachloroethane 0.30

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, or < the the requirements for the 5 compounds is section 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard (IS) areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to +100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)? [ ] [ ]
USEPA Region II
SW846 Method 8260B VOA

Date: January 2006
SOP: HW-24, Rev. 2

YES NO N/A

ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area Lower Limit</th>
<th>Area Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when the associated IS are counts area > + 100%.

3. If the IS area is below the lower limit (< -50%), qualify all associated non-detects (U-values) "J".

4. If extremely low area counts are reported (< -25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.
17.0 Field Duplicates

17.1 Were any field duplicates submitted for volatile analysis?

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.

VWAE-MW4P-03-10 > Dillution  all attached sheet, no equal
VWAE-MW4P-03-10

WVVWI -M05-0310
WVVWI -M003P-0310 > MoC

Dilution — VWAE-MW05-0310
**DataQual**

**Initial Calibration Date:** 3/29/2010

**RRF and %RSD Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>MTBE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>0.752</td>
</tr>
<tr>
<td>Area of Compound</td>
<td>1606395</td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>534322</td>
</tr>
<tr>
<td>Conc. of Internal STD</td>
<td>50</td>
</tr>
<tr>
<td>Conc. of Compound</td>
<td>200</td>
</tr>
<tr>
<td>Calculated RRF</td>
<td>0.752</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>benzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>6.5</td>
</tr>
<tr>
<td>RRF of STD 1</td>
<td>0.979</td>
</tr>
<tr>
<td>RRF of STD 2</td>
<td>1.040</td>
</tr>
<tr>
<td>RRF of STD 3</td>
<td>1.065</td>
</tr>
<tr>
<td>RRF of STD 4</td>
<td>0.925</td>
</tr>
<tr>
<td>RRF of STD 5</td>
<td>1.087</td>
</tr>
<tr>
<td>Calculated % RSD</td>
<td>6.5</td>
</tr>
</tbody>
</table>

**Continuing Calibration File ID:** 4/2/2010

**RRF and %D Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>1,2-dichloroethane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>0.329</td>
</tr>
<tr>
<td>Area of Compound</td>
<td>180904</td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>549516</td>
</tr>
<tr>
<td>Conc. of Internal STD</td>
<td>50</td>
</tr>
<tr>
<td>Conc. of Compound</td>
<td>50</td>
</tr>
<tr>
<td>Calculated RRF</td>
<td>0.329</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>xylene (total)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>2.9</td>
</tr>
<tr>
<td>Average RRF</td>
<td>0.599</td>
</tr>
<tr>
<td>Calibration Check RRF</td>
<td>0.582</td>
</tr>
<tr>
<td>Calculated % D</td>
<td>2.8</td>
</tr>
</tbody>
</table>
**FIELD DUPLICATE SAMPLE SUMMARY**

**Sample ID:** VWAE-MW04-0310  
**Duplicate Sample ID:** VWAE-MW04P-0310

Water: RPD>75%  
Soil: RPD>100%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>MTBE</td>
<td>130</td>
<td>96</td>
<td>30</td>
</tr>
</tbody>
</table>

**COMMENTS:** No qualifications

* result below the CRQL
E - The concentration of this analyte exceeds the calibration range of the instrument.

A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z - Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: SJO464
LAB: Mitkem Labs

SITE NAME: Vieques AOC E CTO-83

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format? [✓] [ ]

ACTION: If not, note the effect on review of the data in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present? [✓] [ ]

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? [✓] [ ]
II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples?

\[\text{\checkmark}\]

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

\[\text{\checkmark}\]

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all non-detects data are qualified as unusable (R), and detects are flagged "J".

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ". Sampled 3/16-22/10 Extr 3/23-26/10

2.0 Holding Times

Rec 3/17-23/10 Anal 3/26-30/10

2.1 Have any semivolatile technical holding times, determined from date of collection to date of extraction, been exceeded?

\[\text{\checkmark}\]

Continuous extraction of water samples for semivolatile analysis must be started within 7 days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within
40 days of the date of extraction.

**Table of Holding Time Violations**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Matrix</th>
<th>Date Sampled</th>
<th>Date Lab Received</th>
<th>Date Extracted</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
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</tbody>
</table>

(See Traffic Report)

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).
3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water  [ ] ___ ___

b. Low/Med Soil [ ] ___ ___

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water  [ ] ___ ___

b. Low/Med Soil [ ] ___ ___

ACTION: If CLP deliverables are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk?  [ ] ___ ___

ACTION: Circle all outliers in red.

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank (Reviewer should use lab in house recovery limits. Use surrogate recovery limits from USEPA National Functional Guidlines January 2005 page 130, if in house limits are not available. See Method 8000B-43 or 80000C-24).  [ ] ___ ___

Note: Examine lab in house limits for reasonableness.

If yes, were samples re-analyzed?  [ ] ___ ___
Were method blanks re-analyzed? [ ] [ ] [ ]

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

1. Flag all positive results as estimated ("J").

2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.

3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".

2. Non-detects for that fraction should be qualified as unusable (R).

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II? [ ] [ ] [ ]

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document
effect in data assessments.

4.0 Matrix Spikes (Form III/Equivalent)

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate/or duplicate unspiked Sample recoveries been listed on the Recovery Form (Form III)?

NOTE: Method 3500B/page 4 states the spiking compounds:

- Base/neutrals
  - 1,2,4-Trichlorobenzene
  - Acenaphthene
  - 2,4-Dinitrotoluene
  - Pyrene
  - N-Nitroso-di-n-propylamine
  - 1,4-Dichlorobenzene

- Acids
  - Pentachlorophenol
  - Phenol
  - 2-Chlorophenol
  - 4-Chloro-3-methylphenol
  - 4-Nitrophenol

Note: Some projects may require the spiking of specific compounds of interest.

Note: See Method 8270D-sec 8.4.2 for deciding on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate. If samples are expected to contain target analytes, then laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratory should use a matrix spike and matrix spike duplicate pair.

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water

b. Low Solid

c. Med Solid

- 11 -
ACTION: If any matrix spike data are missing, take
the action specified in 3.2 above. It may be
necessary to contact the lab to obtain the
required data.

NOTE: If the data has not been reported on CLP
equivalent form, then the laboratory must
provide the information necessary to evaluate
the spike recoveries in the MS and MSD. The
required data which should have been provided
by the lab include the analytes and
concentrations used for spiking, background
concentrations of the spiked analytes (i.e.,
concentrations in unspiked sample), methods
and equations used to calculate the QC
acceptance criteria for the spiked analytes,
percent recovery data for all spiked
analytes.

The data reviewer must verify that all
reported equations and percent recoveries are
correct before proceeding to the next
section.

4.3 Were matrix spikes performed at concentration
equal to 100ug/L for acid compounds, and 200ug/l
for base compounds (Method 3500B-4), or those
specified in project plan.

4.4 How many semivolatile spike recoveries are outside
Laboratory in house MS/MSD recovery limits (use recovery limits
values in Method 8270D-43644 Table 6 if in house values not
available).

Water

\( \frac{0}{2} \)

Solids

\( \frac{0}{0} \) out of \( \frac{0}{n} \)
4.5 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

\[
\begin{align*}
\text{Water} & \quad \emptyset \quad \text{out of } \_ \\
\text{Solids} & \quad \_ \quad \text{out of } \_ \\
\end{align*}
\]

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a Laboratory Control Sample (LCS) analyzed with each analytical batch? \(\checkmark\) ___ ___

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 **Blanks (Form IV/Equivalent)**

5.1 Is the Method Blank Summary (Form IV) present? \(\checkmark\) ___ ___

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch? \(\checkmark\) ___ ___

5.3 Has a method blank been analyzed either after
the calibration standard or at any other time
during the analytical shift for each GC/MS system
used?

ACTION: If any method blank data are missing, call
lab for explanation/resubmittal. If not
available, use professional judgement to
determine if the associated sample data
should be qualified.

5.4 Chromatography: review the blank raw data -
chromatograms (RICs), quant reports or data system
printouts and spectra.

Is the chromatographic performance (baseline
stability) for each instrument acceptable for
the semivolatiles?

ACTION: Use professional judgement to determine the
effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled
water blanks" are validated like any other
sample and are not used to qualify the data.
Do not confuse them with the other QC blanks
discussed below.

6.1 Do any method/instrument/reagent blanks have
positive results for target analytes and/or TICs?
When applied as described below, the contaminant
concentration in these blanks are multiplied by
the sample dilution factor and corrected for
percent moisture where necessary.

6.2 Do any field/rinse/blanks have positive results
for target analytes and/or TICs (if required,
see section 10 below)?
**USEPA Region II**
**SW846 Method 8270D (Rev.4, January 1998)**

**Date:** October, 2006
**SOP HW-22 Rev.3**

**YES** | **NO** | **N/A**
--- | --- | ---

**ACTION:** Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

**NOTE:** All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

**ACTION:** Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

```
VWAI - EB01 - 032210

\text{Br}(2 \text{eh}) \text{ ppm} 1.25 > \text{No qual}
```

```
all other QC tests exhibited no qual
```
Blank Action for Semivolatile Analyses

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Method, Field</td>
<td>Detects</td>
<td>Not detected</td>
<td>No qualification required</td>
</tr>
<tr>
<td>&lt; CRQL *</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>No qualification required</td>
<td></td>
</tr>
<tr>
<td>= CRQL *</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>No qualification required</td>
<td></td>
</tr>
<tr>
<td>&gt; CRQL *</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report concentration of sample with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and ≥ blank contamination</td>
<td>No qualification required</td>
<td></td>
</tr>
</tbody>
</table>

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

NOTE: If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required.

6.3 Are there field/rinse/equipment blanks associated with every sample? ☑

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

6.4 Was a instrument blank analyzed after each sample/dilution which contained a target compound
that exceeded the initial calibration range. [ ] [ ] [ ]

6.5 Does the instrument blank have positive results for target analytes and/or TICs?

Note: Use professional judgement to determine if carryover occurred and qualify analytes accordingly.

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270D? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column. [ ] [ ] [ ]

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (Form V/Equivalent)

8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)? [ ] [ ] [ ]

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5
page 8270D-12).

8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift?

YES NO N/A

8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?

YES NO N/A

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

<table>
<thead>
<tr>
<th>DATE</th>
<th>TIME</th>
<th>INSTRUMENT</th>
<th>SAMPLE NUMBERS</th>
</tr>
</thead>
<tbody>
<tr>
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</table>

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

8.4 Have the ion abundances been normalized to m/z 198?

YES NO N/A

8.5 Have the ion abundance criteria been met for each instrument used?

YES NO N/A

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

- 18 -
ACTION: If ion abundance criteria are not met, take action specified in section 3.2

8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) __ _ __

8.7 Have the appropriate number of significant figures (two) been reported? __ _ __

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

8.8 Are the spectra of the mass calibration compound acceptable? __ _ __

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

9.0 Target Analytes

9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate __ _ __

b. Matrix spikes and matrix spike duplicates __ _ __

c. Blanks __ _ __

9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270D-14)? __ _ __
ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

a. Samples and/or fractions as appropriate [ ] [ ]

b. Matrix spikes and matrix spike duplicates (Mass spectra not required) [ ] [ ]

c. Blanks [ ] [ ]

ACTION: If any data are missing, take action specified in 3.2 above.

9.4 Are the response factors shown in the Quant Report? [ ] [ ]

9.5 Is chromatographic performance acceptable with respect to:

Baseline stability? [ ] [ ]

Resolution? [ ] [ ]

Peak shape? [ ] [ ]

Full-scale graph (attenuation)? [ ] [ ]

Other: ___________________________ [ ] [ ]

ACTION: Use professional judgement to determine the acceptability of the data.

9.6 Are the lab-generated standard mass spectra of identified semivolatile compounds present for
each sample?

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not
generate their own standard spectra, make a
note in the data assessment narrative. If
spectra are missing, reject all positive
data.

9.7 Is the RRT of each reported compound within 0.06
RRT units of the standard RRT in the continuing
calibration?

\[ \checkmark \]  

9.8 Are all ions present in the standard mass spectrum
at a relative intensity greater than 10% (of the
most abundant ion) also present in the sample mass
spectrum?

\[ \checkmark \]  

9.9 Do the relative intensities of the characteristic
ions in the sample agree within ± 30% of the
 corresponding relative intensities in the
reference spectrum?

\[ \checkmark \]  

ACTION: Use professional judgement to determine
acceptability of data. If it is determined
that incorrect identifications were made, all
such data should be rejected (R), flagged "N"
(Presumptive evidence of the presence of the
compound) or changed to not detected (U) at
the calculated detection limit. In order to
be positively identified, the data must
comply with the criteria listed in 9.7, 9.8,
and 9.9.

ACTION: When sample carry-over is a possibility,
professional judgement should be used to
determine if instrument cross-contamination
has affected any positive compound
identification.
10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form Is, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? \[\text{No/\checkmark}\]

NOTE: Review sampling reports to determine if the lab was required to identify non target analytes (refer to section 7.6.2, page 8270D-21).

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate \[\text{[] \[\checkmark\]}\]

b. Blanks \[\text{[] \[\checkmark\]}\]

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)? \[\text{[]} \[\checkmark\] \]

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the
sample mass spectrum?

10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R."

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and, for soils, sample moisture?
ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration? ✓

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI/Equivalent)

13.1 Is the Initial Calibration Form (Form VI/ Equivalent) present and complete for the semivolatile fraction? ✓

ACTION: If any calibration forms or standard row data are missing, take action specified in 3.2 above.

13.2 Are all base neutral or acid RRFs > 0.050? ✓
Check the **average RRFs** of the four System Performance Check Compounds (SPCCs): N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. These compounds must have **average RRFs** greater than or equal to 0.05 before running samples and should not show any peak tailing.

**ACTION:** Circle all outliers in red.

**ACTION:** For any target analyte with **average RRF < 0.05**

1. "R" all non-detects;

2. "J" all positive results.

13.3 Are response factors for base neutral or acid target analytes stable over the concentration range of the calibration (% Relative standard deviation [%RSD] < 15.0%)?

**NOTE:** The % RSD for each individual Calibration Check Compound (CCC, Method 8270D-40 see Table 4) must be less than 30% before analysis can begin. If greater 30%, the lab must clean and recalibrate the instrument.

**CALIBRATION CHECK COMPOUNDS**

<table>
<thead>
<tr>
<th>Base/Neutral Fraction</th>
<th>Acid Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acenaphthene</td>
<td>4-Chloro-3-methylphenol</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>2,4-Dichlorophenol</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>2-Nitrophenol</td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>Phenol</td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>2,4,6-Trichlorophenol</td>
</tr>
</tbody>
</table>
Benzo(a)pyrene

ACTION: If the %RSD for any CCC >30% and no corrective action taken, then "J" qualify all positive hits and "UJ" qualify all non-detects.

ACTION: Circle all outliers in red.

ACTION: If the % RSD is ≥ 15.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detect results for that analyte "R," unusable. Alternatively, the lab should calculate first or second order regression fit of the calibration curve and select the fit which introduces the least amount of error.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

13.4 Did the laboratory calculate the calibration curve by the least squares regression fit?  

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.)

ACTION: Circle Errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.5 Do the target compounds for this SDG include Pesticides?
13.6 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%?

ACTION: If DDT percent breakdown exceeds 20%:

i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

14.0 GC/MS Calibration Verification (Form VII/Equivalent)

14.1 Are the Calibration Verification Forms (Form VII) present and complete for all compounds of interest?

14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

________________________

________________________

________________________

ACTION: If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis,
call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

14.3 Do any of the SPCCs have an RRF < 0.05? __ 1 __

If YES, make a note in data assessment if the lab did not take corrective action specified in section 7.4.4, page 8270D-18. __ 1 __

14.4 Do any of the CCCs have a %D between the initial and continuing RRF which exceeds 20.0%?

ACTION: If yes, make a note in data assessment.

14.5 Do any semivolatile compounds have a % Difference (% D) between the initial and continuing RRF which exceeds 20.0%? __ 1 __

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is above 90%, qualify all non-detects for that analyte as "R", unusable.

14.6 Do any semivolatile compounds have a RRF < 0.05? __ 1 __

ACTION: Circle all outliers in red.

ACTION: If RRF < 0.05, qualify as unusable ("R") associated non-detects and "J" associated positive values.

14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more). __ 1 __
ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area</th>
<th>Lower Limit</th>
<th>Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

Note: Check Table 5, 8270D-41 for associated analytes.

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.
iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Laboratory Control Samples (LCS)

16.1 Were any LCS samples run in order to verify analytes which failed criteria for spike recovery?

16.2 Did the lab spike LCS sample spiked with the same analytes and the same concentrations as the matrix spike?

16.3 Were the mean and standard deviation of all analytes within the QC acceptance ranges as shown in Table 6, 8270D-43?

ACTION: If the recovery of any analyte falls out of the designated range, the analytical results for that compound is suspect and should be qualified "J" in the unspiked samples.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for semivolatile analysis?
ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

VWAE-MW04-0310 > moQ
VWAE-MW04P-0310

WVVWAI-MW03-0310 > moQ
WVVWAI-MW03P-0310
DataQual

Worksheets – GRO BY 8015

This SDG contains Gasoline results SW-846 method 8015. Region II validation guidelines were used as applicable, however, the Region has not developed an SOP for this method so these worksheets are used as an alternative.

Holding Times

Sampling Date: 3/16-22/10  14-day soil sample holding time and 7 day water sample holding time was
Received Date: 3/17-3/23  applied based on SW-846 recommendations
Preparation Date: 3/24
Analysis Dates: 3/24

COC documentation was present and in order. All sample extraction and analysis holding time requirements were met for these water and field QC blank samples.

Calibrations

A seven-point calibration curve was analyzed for both the target compound and the surrogate compound. %RSDs were calculated for the target fuel ranges as well as for individual hydrocarbons over the range of retention times of interest and the surrogate compounds. Continuing calibration standards were analyzed per the method. All %D values were within QC limits with the exception of one hydrocarbon. All average %Ds were within 20%. No qualifications were required. These samples were analyzed on one sequence.

Blank Summary

Blank qualification guidelines:

- No action is taken if a compound is found in the blank but not in the sample.
- **Sample weight, volume or dilution factor must be taken into consideration when applying criteria.**
- Apply the same data validation guidelines to any associated method, trip, rinse and field blanks and all associated samples.
- Qualification/Action codes:
  - **U** - The blank contamination concentration is ≤ RL or > RL and sample result is < RL. Result is qualified as U at the RL.
  - **U** - The blank contamination concentration is > RL and sample result is either is > RL but < blank contamination concentration. Result is qualified as U at reported concentration.
  - **J** - The blank contamination concentration is > RL and sample result is < 10X blank contamination level.
  - **NA** - The sample is greater than the RL when the blank contamination concentration is < RL or the sample result is greater than 10X blank contamination concentration when the blank contamination concentration is > RL.

Blank Contamination and Qualification Summaries

<table>
<thead>
<tr>
<th>Blank ID</th>
<th>Compound</th>
<th>Concentration</th>
<th>Action Level</th>
<th>Q Flag</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAE-EB01-031610</td>
<td>GRO</td>
<td>110 ug/L</td>
<td>blank level</td>
<td>U</td>
</tr>
</tbody>
</table>

Associated samples and required qualifications are noted in the following table.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Q Flag</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAE-MW03-0310</td>
<td>GRO</td>
<td>U</td>
</tr>
</tbody>
</table>

Vieques PR, AOC E
CTO-83
SDG SJ0464
Page 1 of 2
Surrogate Recoveries Summary

All surrogate recoveries were acceptable. No qualifications were required.

Matrix Spike/Matrix Spike Duplicate Summary

The MS/MSD pair in this SDG exhibited acceptable recoveries and RPDs. The submitted LCS samples were acceptable. No qualifications were required.

Field Duplicate Sample Summary

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Duplicate Conc.</th>
<th>RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>GRO</td>
<td>65</td>
<td>60</td>
<td>8%</td>
</tr>
</tbody>
</table>

Comments: 30% RPD criteria, No qualifications were required.

Sample Result Verification

Specific Comments:
Raw data was verified.

Reviewer: JACleveland
Date: 6/4/10

Vieques PR, AOC E
CTO-83
SDG SJ0464
Page 2 of 2
**DataQual**

**Initial Calibration Date:** 11/18/2009

**RF and %RSD Calculations:**

| Compound Name: | GRO, level I (2.5) |
| Lab Value: | 8.449 X 10^6 |
| Area of Compound | 212328 |
| Cone of Compd | 3 |
| Calculated RRF | 844931 |

| Compound Name: | GRO |
| Lab Value: | 4.4 |
| RRF of STD 1 | 84490 |
| RRF of STD 2 | 76470 |
| RRF of STD 3 | 77450 |
| RRF of STD 4 | 76640 |
| RRF of STD 5 | 75220 |
| RRF of STD 6' | 80320 |
| Calculated % RSD | 4.37 |

**Continuing Calibration File ID:** CCV 3/23/10, 22:04

**RF and %D Calculations:**

| Compound Name: | GRO |
| Lab Value CF: | 68319 |
| Lab Value %D: | 12.9 |
| Area of compound | 34159318 |
| Concentration | 500 |
| Calculated CF | 68318.6 |

| Average CF | 78430 |
| Calibration Check CF | 68319 |
| Calculated % D | 12.9 |
SAMPLE CALCULATION

Sample ID: VWA-E-MW01-0310
Standard ID: ICAL, 11/18/09
Compound: GRO
Concentration: 150 µg/L

<table>
<thead>
<tr>
<th></th>
<th>Water (mg/L)</th>
<th>Soil (mg/Kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>13291102</td>
<td></td>
</tr>
<tr>
<td>CF of Compound</td>
<td>78430</td>
<td></td>
</tr>
<tr>
<td>Final Volume</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>Dilution Factor</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>GPC Factor</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Injection Volume</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Weight of Sample</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Initial Volume of Sample</td>
<td>5</td>
<td>NA</td>
</tr>
<tr>
<td>% Solids Factor</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Concentration</td>
<td>169.46</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>

Final Conc = GRO Conc. - Surrogate Conc. = 169.46 - 17.51 = 151.95 µg/L.
Thanks so much Ed. I thought that might be it so I subtracted out the surrogate area then calculated the GRO result but I didn’t check it for the after calculation concentrations! Have a good day!

Hi Jackie—

It is because of the surrogate. For GRO, the surrogate elutes within the retention time range for GRO, so we have to subtract the concentration of the surrogate from the GRO before we calculate the final value. In this case (J0464-09) it is 169.464 – 17.513 = 151.95, which rounds to 150.

Please let me know if you need more information, or have any additional questions.

Thanks.

--Ed

Good Afternoon Ed,

I have what I hope is a very quick question! For Mitkem Work Order Number J0464 the GRO results on the Form 1s do not match my calculations or the raw data quant pages. For example: sample VWAE-MW01-0310 (J0464-09) Form 1 result is 150 ug/L but quant page (page 400 of data package) & my recalculation of the result say 169 ug/L. Please clarify the discrepancy with resubmissions and/or an explanation, as necessary! I planned to ship this DV report to the client today so if you are able to respond this afternoon that would be fantastic!

Jackie

Jacqueline Cleveland
Vice-President
DataQual, ES, LLC.
636-352-9391
cleve137@charter.net
This SDG contains Diesel results SW-846 method 8015M. Region II validation guidelines were used as applicable, however, the Region has not developed an SOP for this method so these worksheets are used as an alternative.

**Holding Times**

- **Sampling Date:** 3/16-22/10
- **Received Date:** 3/17-3/23
- **Preparation Date:** 3/22
- **Analysis Dates:** 3/23-3/24

14-day soil sample holding time and 7 day water sample holding time was applied based on SW-846 recommendations.

COC documentation was present and in order. All sample extraction and analysis holding time requirements were met for these water and field QC blank samples.

**Calibrations**

A seven-point calibration curve was analyzed for both the target compound and the surrogate compound. %RSDs were calculated for the target fuel ranges as well as for individual hydrocarbons over the range of retention times of interest and the surrogate compounds. Continuing calibration standards were analyzed per the method. All %D values were within QC limits with the exception of one hydrocarbon. All average %D's were within 20%. No qualifications were required. These samples were analyzed on one sequence.

**Blank Summary**

- **Blank qualification guidelines:**
  - No action is taken if a compound is found in the blank but not in the sample.
  - **Sample weight, volume or dilution factor must be taken into consideration when applying criteria.**
  - Apply the same data validation guidelines to any associated method, trip, rinse and field blanks and all associated samples.
  - **Qualification/Action codes:**
    - **U** - The blank contamination concentration is ≤ RL or > RL and sample result is < RL. Result is qualified as U at the RL.
    - **U** - The blank contamination concentration is > RL and sample result is either is > RL but < blank contamination concentration. Result is qualified as U at reported concentration.
    - **J** - The blank contamination concentration is > RL and sample result is < 10X blank contamination level.
    - **NA** - The sample is greater than the RL when the blank contamination concentration is < RL or the sample result is greater than 10X blank contamination concentration when the blank contamination concentration is > RL.

**Blank Contamination and Qualification Summaries**

<table>
<thead>
<tr>
<th>Blank ID</th>
<th>Compound</th>
<th>Concentration</th>
<th>Action Level</th>
<th>Q Flag</th>
</tr>
</thead>
<tbody>
<tr>
<td>no contamination</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Associated samples and required qualifications are noted in the following table.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Q Flag</th>
</tr>
</thead>
</table>

Vieques PR, AOC E
CTO-83
SDG SJ0464
Page 1 of 2
Surrogate Recoveries Summary

All surrogate recoveries were acceptable. No qualifications were required.

Matrix Spike/Matrix Spike Duplicate Summary

The MS/MSD pairs in this SDG exhibited acceptable recoveries and RPDs. The submitted LCS samples were acceptable. No qualifications were required.

Field Duplicate Sample Summary

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Duplicate Conc.</th>
<th>RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>ETPH</td>
<td>1.6</td>
<td>1.5</td>
<td>6%</td>
</tr>
<tr>
<td>ORO</td>
<td>0.54</td>
<td>0.63</td>
<td>15%</td>
</tr>
</tbody>
</table>

Comments: 30% RPD criteria, No qualifications were required.

Sample Result Verification

Specific Comments:

Raw data was verified.
**DataQual**

**Initial Calibration Date:**

1/20/2010

**RRF and %RSD Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Triacontane</th>
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</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>1.1200</td>
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</table>

<table>
<thead>
<tr>
<th>Area of Compound</th>
<th>5090095</th>
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<tbody>
<tr>
<td>Conc. of Compound</td>
<td>2.5</td>
</tr>
<tr>
<td>Area of Internal Standard</td>
<td>72747684</td>
</tr>
<tr>
<td>Conc. of Internal Standard</td>
<td>40</td>
</tr>
<tr>
<td>Calculated CF</td>
<td>1.1195</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>TPH (C9..C28)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>6.52</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>CF of STD 1</th>
<th>1.1060</th>
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</thead>
<tbody>
<tr>
<td>CF of STD 2</td>
<td>1.0570</td>
</tr>
<tr>
<td>CF of STD 3</td>
<td>1.1230</td>
</tr>
<tr>
<td>CF of STD 4</td>
<td>1.0850</td>
</tr>
<tr>
<td>CF of STD 5</td>
<td>0.9480</td>
</tr>
<tr>
<td>CF of STD 6</td>
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</tr>
<tr>
<td>CF of STD 7</td>
<td></td>
</tr>
<tr>
<td>Calculated % RSD</td>
<td>6.51</td>
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</table>

**Continuing Calibration File ID:**

3/24/10, 0454

**RRF and %D Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Octane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Area of Compound</th>
<th>38314083</th>
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</thead>
<tbody>
<tr>
<td>Conc. of Compound</td>
<td>50</td>
</tr>
<tr>
<td>Area of Internal Standard</td>
<td>60758444</td>
</tr>
<tr>
<td>Conc. of Internal Standard</td>
<td>40</td>
</tr>
<tr>
<td>Calculated CF</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>TPH (C9..C28)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>3.4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Average CF</th>
<th>1.0640</th>
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</thead>
<tbody>
<tr>
<td>Calibration Check CF</td>
<td>1.1000</td>
</tr>
<tr>
<td>Calculated % D</td>
<td>-3.4</td>
</tr>
</tbody>
</table>
SAMPLE CALCULATION

Sample ID: VWAE-MW05-0310
Standard ID: ICAL, 1/20/10
Compound: DRO
Concentration: 1.3 mg/L

<table>
<thead>
<tr>
<th></th>
<th>Water (mg/L)</th>
<th>Soil (mg/Kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>2315375528</td>
<td></td>
</tr>
<tr>
<td>CF of Compound</td>
<td>1.064</td>
<td></td>
</tr>
<tr>
<td>Area of Internal Standard</td>
<td>68179633</td>
<td></td>
</tr>
<tr>
<td>Concentration of Internal Standard</td>
<td>40</td>
<td></td>
</tr>
<tr>
<td>Final Volume</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Dilution Factor</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>GPC Factor</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Injection Volume</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Weight of Sample</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Initial Volume of Sample</td>
<td>1000</td>
<td>NA</td>
</tr>
<tr>
<td>% Solids Factor</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Concentration</td>
<td>1.28</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>
Site: Vieques PR
Case #: 
SDG #: JØ464
Samples: Soil Water
A.1.1 **Contract Compliance Screening Report**

Present?

**ACTION:** If no, contact RSCC/PO.

A.1.2 **Record of Communication (from RSCC)**

Present?

**ACTION:** If no, request from the RSCC.

A.1.3 **Sampling Trip Report**

Present and complete?

**ACTION:** If no, contact RSCC/PO.

A.1.4 **Chain of Custody/Sample Traffic Report**

Present?

Legible?

Signature of sample custodian present?

**ACTION:** If no, contact RSCC/WAM/PO.

A.1.5 **Cover Page**

Present?

Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?

Do the sample identification numbers on the Cover Page agree with sample identification numbers on:

(a) Traffic Report Sheet?
(b) Form I's?

Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?

ACTION:
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 SDG Narrative, DC-1 & DC-2 Form

Is the SDG Narrative present?

Is Sample Log-in Sheet (Form DC-1) present and complete?

Is Complete SDG Inventory Sheet (Form DC-2) present and complete?

ACTION:
If no, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.7 Form I to XV

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name?
Laboratory Code?
RAS/Non-RAS Case No.?
SDG No.?
**Standard Operating Procedure**

**USEPA Region 2**

**Evaluation of Metals Data for the Contract Laboratory Program**

**Data Assessment and Contract Compliance Review**

**SOP: HW-2 Revision 13**

**Appendix A.1**

**Sept. 2006**

**Contract No.?**

<table>
<thead>
<tr>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>[✓]</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**

If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

**A.1.7.2**

After comparing values on Forms I-IX against the raw data, do any computation/transcription errors exceed 10% of the reported values on the Forms for:

- (a) all analytes analyzed by ICP-AES?  
  - [✓]

- (b) all analytes analyzed by ICP-MS?  
  - [✓]

- (c) Mercury?  
  - [✓]

- (d) Cyanide?  
  - [✓]

**ACTION:**

If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

**A.1.8 Raw Data**

Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

**A.1.8.1 Digestion/Distillation Log**

- Digestion Log for ICP-AES (Form XII) present?  
  - [✓]

- Digestion Log for ICP-MS (Form XII) present?  
  - [✓]

- Digestion Log for mercury (Form XII) present?  
  - [✓]

- Distillation Log for cyanide (Form XII) present?  
  - [✓]

Are pH values for metals and
**Standard Operating Procedure**

USEPA Region 2
Evaluation of Metals Data for the Contract Laboratory Program
Data Assessment and Contract Compliance Review

<table>
<thead>
<tr>
<th>SOP: HW-2</th>
<th>Revision 13</th>
<th>Appendix A.1</th>
<th>Sept. 2006</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>cyanide reported for each aqueous sample?</td>
<td></td>
<td></td>
<td></td>
<td>✓</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Are percent solids calculations present for soils/sediments?</td>
<td></td>
<td></td>
<td></td>
<td>✓</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Are preparation dates present on the sample preparation logs/bench sheets?</td>
<td></td>
<td></td>
<td></td>
<td>✓</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**NOTE:**
Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.

A.1.8.2 Is the analytical instrument real-time printouts present for:

ICP-AES? |   |   |   |
ICP-MS? |   |   | ✓ |
Mercury? |   | ✓ |   |
Cyanide? |   |   | ✓ |

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:

Legible? | ✓ |   |
Properly labeled? | ✓ |   |

Are all field samples, QC samples and field QC samples present on:

Digestion/Distillation log? | ✓ |   |
Instrument Printouts? | ✓ |   |

**ACTION:**
If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.
A.1.9 **Technical Holding Times:** (Aqueous and soil samples)
(Examine sample Traffic Reports and digestion/distillation logs to
determine the holding time from the sample collection date to the sample
preparation date.)

A.1.9.1 Cyanide distillation (14 days) exceeded?  
Mercury analysis (28 days) exceeded?  
Other Metals analysis (180 days) exceeded?

ACTION:
If yes, reject (R) and red-line non-detects and flag as estimated (J) results ≥ MDL even
if sample(s) was preserved properly.

NOTE:
In addition to qualifying the data, a list of all samples and analytes which exceeded the holding times must
be prepared. Report for each sample the number of days that were exceeded.
(Submit the sample collection date from the sample preparation date).
Attach this list to the data review narrative.

A.1.9.2 Is pH of aqueous samples for:
Metals Analysis ≤ 2?
Cyanide Analysis ≥ 12?

ACTION:
If no for any of the above, flag non-detects as "R" and detects as "J".

A.1.9.3 Is the cooler temperature ≤ 10 C°?

ACTION:
If cooler temperature is > 10 C°, flag non-detects as "UJ" and detects as "J".

A.1.10 **Final Data Correctness - Form I**
A.1.10.1 Are Form I's for all samples
present and complete?

**ACTION:**
If no, prepare Telephone Record Log and contact CLP PO/TOPO for submittal from the laboratory.

A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

Is the calculation error less than 10% of the correct result? [✓] ___ ___

Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)? [✓] ___ ___

Are results on Form I'S reported by correct significant figures? [✓] ___ ___

Are soil sample results on Form I's corrected for percent solids? [___] ___ [✓]

Are all "less than MDL" values reported by the CRQLs and coded with "U"? [✓] ___ ___

Are values less than the CRQLs but greater than or equal to the MDLs flagged with "J"? [✓] ___ ___

Are appropriate contractual quality control and Method qualifiers used? [✓] ___ ___

**ACTION:**
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3 Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data? [✓] ___ ___

Was a brief physical description
of the samples before and after digestion given on the Form I's?

[ ] ___ ___

Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I?

[ ] ___ ___

**ACTION:**
If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11 **Initial Calibration**

A.1.11.1 Is a record of at least 2 point (A blank and a standard) calibration present for ICP-AES analysis?

[ ] ___ ___

Is a record of at least 2 point (a blank and a standard) calibration present for ICP-MS analysis?

[ ] ___ ___

Is a record of at least 5 point calibration (a blank & 4 standards) present for Hg analysis?

[ ] ___ ___

Is a record of at least 4 point calibration (a blank & 4 standards) present for cyanide?

[ ] ___ ___

**ACTION:**
If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).

Is one initial calibration standard at the CRQL level for cyanide and mercury?

[ ] ___ ___

**ACTION:**
If no, write in the Contract Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11.2 Is the curve correlation coefficient ≥ 0.995 for:
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Mercury Analysis?  YES  NO  N/A  ✓

Cyanide Analysis?  YES  NO  N/A

ICP-AES (more than 2 point Calib.)?  YES  NO  N/A

ICP-MS (more than 2 point calib.)?  YES  NO  N/A

**ACTION:**
If no, qualify the associated sample results ≥ MDL as estimated “J” and non-detects as “UJ”.

**NOTE:**
The correlation coefficient shall be calculated by the data validator using standard concentrations and the corresponding instrument response (e.g., absorbance, peak area, peak height, etc.).

A.1.12 Initial and Continuing Calibration Verification - Form IIA

A.1.12.1 Present and complete for every metal and cyanide?  YES  NO  N/A

Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte?  YES  NO  N/A

**ACTION:**
If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.

A.1.12.2 Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent?  YES  NO  N/A

**ACTION:**
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.12.3 Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples?  YES  NO  N/A

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ACTION:
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative and qualify results ≥ MDL as estimated (J).

A.1.12.2 Circle on each Form IIA all percent recoveries that are outside the contract windows.

Are ICV/CCVs within control limits for:

- Metals - 90-110%R? 
  - [ ]

- Hg - 80-120%R? 
  - [ ]

- Cyanide - 85-115%R? 
  - [ ]

ACTION:
If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89%(65-79% for Hg; 70-84% for CN).
Qualify only positive results(≥ MDL) as "J" if the ICV/CCV %R is between 111-125%(121-135% for Hg;116-130% for CN). Reject (R) and red-line only detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detects) if the recovery is less than 75% (65% for Hg; 70% for CN).

NOTE:
For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

A.1.12.3 Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)?

- [ ]

ACTION:
If no, Qualify all cyanide results ≥ MDL as "J".

A.1.13 CRQL Standard Analysis - Form IIB

A.1.13.1 For each ICP-AES run, was a CR1
(CRQL or MDL when MDL > CRQL) standard analyzed?
(Note: CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)

For each ICP-MS run, was a CRI
(CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis?

For each mercury run, was a CRQL standard analyzed?

For each cyanide run, was a CRQL standard analyzed?

**ACTION:**
If no for any of the above, write this deficiency in the Contract Problems/Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL) as J and non-detects UJ.

The affected ranges are:
ICP-AES Analysis - *True Value ± CRQL
ICP-MS Analysis - *True Value ± CRQL
Mercury Analysis - *True Value ± CRQL
Cyanide Analysis - *True Value ± CRQL

A.1.13.2 Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis?

**ACTION:**
If no, write in the Contract Problem/Non-Compliance Section of the "Data Review Narrative".

A.1.13.3 Circle on each Form IIB all percent recoveries that are outside the acceptance windows.
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<table>
<thead>
<tr>
<th>Is the CRQL standard within control limits for:</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metals (ICP-AES/ICP-MS) - 70 - 130%?</td>
<td></td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>Mercury - 70 - 130%?</td>
<td></td>
<td>✓</td>
<td></td>
</tr>
<tr>
<td>Cyanide - 70 - 130%?</td>
<td></td>
<td>✓</td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**
If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag(J) only detects <2xCRQL if the recovery is between 131% and <180%. If the recovery is less than 150%, reject (R) and red-line non-detects and detects < 2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detects <2xCRQL and flag (J)detects > 2xCRQL but < ICV/CCV if the recovery is > 180%.

**NOTE:**
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard.
2. Flag (J) or reject (R) only the final sample results on Form I's when Sample raw data are within the affected ranges and the CRQL standard is outside the acceptance window.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

#### A.1.14 Initial and Continuing Calibration Blanks - Form III

**A.1.14.1** Present and complete for all the instruments used for the metals and cyanide analyses?

Was an initial Calibration Blank analyzed after ICV?

Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?

Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by
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using MDLs from direct analysis (Preparation Method "NP1")?
(Check Form III against the raw data)

ACTION:
If no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".

A.1.14.2 Circle with red pencil on each Form III all Calib. Blank values that are:

> MDL but < CRQL

> CRQL

A.1.14.2.1 When MDL < CRQL, is any Calib. Blank value > MDL but < CRQL?

ACTION:
If yes, change sample results > MDL but < CRQL to the CRQL with a "U". Do not qualify non-detects.

A.1.14.2.2 When MDL < CRQL, is any Calib. Blank value > CRQL?

ACTION:
If yes, reject (R) and red line the associated sample results > CRQL but < ICB/CCB Blank Result. Flag as "J" detects > ICB/CCB blank value but < 10xICB/CCB value. Change the sample results > MDL but ≤ the CRQL to CRQL with a "U".

A.1.14.2.3 Is any Calibration Blank value below the negative CRQL?

ACTION:
If yes, flag (J) as estimated all associated sample results ≥ CRQL but <10xCRQL.

NOTE:
1. For ICB that does not meet the technical QC Criteria, apply the action to all samples
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1. For CCBs that do not meet the technical QC criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

A.1.15 Preparation Blank - FORM III

NOTE: The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1 Was one Preparation Blank prepared with and analyzed for:

Each Sample Delivery Group (SDG)?

[ ] [X] [ ]

Each batch of the SDG samples digested/distilled?

[ ] [X] [ ]

Each matrix type?

[ ] [X] [ ]

All instruments used for metals and cyanide analyses?

[ ] [X] [ ]

ACTION:
If no for any of the above, flag as estimated (J) all the associated data \(<10\times MDL\) for which the Preparation Blank was not analyzed.

NOTE:
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated (J), but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

\[ \geq \text{MDL but } \leq \text{CRQL}, \text{ and} \]

\[ > \text{CRQL} \]

A.1.15.2.1 When MDL \(<\text{CRQL}\), is any preparation blank value \( \geq \text{MDL but } \leq \text{CRQL}\)?

[ ] [X] [ ]

ACTION:
If yes, change sample result \( \geq \text{MDL} \)
but \( \leq \text{CRQL} \) to \( \text{CRQL} \) with a “U”.

A.1.15.2.2 When the MDL \( \leq \text{CRQL} \), is any Preparation Blank value greater than its CRQL? ___ [ ] ___

If yes, is the Prep. Blank value greater than the value of the associated Field Blank collected and analyzed with the SDG samples? ___ [ ] ___

If yes, is the lowest concentration of that analyte in the associated samples less than 10 times the Preparation Blank value? ___ [ ] ___

**ACTION:**
If yes, reject (R) and red-line all associated sample results greater than the CRQL but less than the Prep. Blank value. Flag as “J” detects > Prep. Blank value but \(<10\times\text{Prep. Blank} \). If the sample result \( \geq \text{MDL} \) but \( \leq \text{CRQL} \), replace it with CRQL-U.

If the Prep. Blank value is less than the same analyte value in the Field Blank, do not qualify the sample results due to the Prep. Blank criteria.

**NOTE:**
Convert soil sample result to mg/Kg on wet weight basis to compare with the soil Prep. Blank result \( \leq \text{Form III.} \)

A.1.15.2.3 Is the Prep. Blank concentration below the negative CRQL? ___ [ ] ___

**ACTION:**
If yes, flag (J) all associated sample results less than \( 10\times\text{CRQL} \). Qualify non-detects as estimated (UJ).

A.1.15.2.4 When the MDL is greater than the CRQL, is the preparation blank concentration on Form III greater than two times the MDL? ___ [ ] ___

**ACTION:**
If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.

A.1.16 **ICP-AES/ICP-MS Interference Check Sample (ICS) - Form IV**

**NOTE:** Not required for CN, Hg, Al, Ca, Fe and Mg.

A.1.16.1 Present and complete? [✓] ___ ___

Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples? [✓] ___ ___

Was ICS analyzed at the beginning of the ICP-MS analytical run? [✓] ___ ___

**ACTION:**
If no, flag as estimated (J) all Sample results.

A.1.16.2 **ICP-AES Method**

A.1.16.2.1 **ICSA Solution:**

For ICP-AES, are the ICSA "Found" analyte values within the control limits ± of CRQL of the true/established mean value? [✓] ___ ___

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV? [✓] ___ ___

**ACTION:**
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results ≥MDL
for which the ICSA “Found” value is greater than
(True value+CRQL). Do not qualify non-detects.
If the ICSA “Found” value is less than
(True value-CRQL), flag non-detects as “UJ” and
detects as “J”.

A.1.16.2.3 ICSAB Solution

For ICP-AES, are all analyte results in
ICSAB within the control limits of 80-120
of the true/established mean value? [ ]

If no for any of the above, is the
sample concentration of Al, Ca, Fe,
or Mg in the same units (μg/L or mg/kg)
greater than or equal to its respective
concentration in the ICSAB Solution on
Form IV? [ ]

ACTION:
If yes, apply the following action to
all samples analyzed between a previous
technically acceptable analysis of the
ICS and a subsequent technically acceptable
analysis of the ICS in the analytical run:

Flag (J) as estimated those associated
sample results ≥ MDL for which the ICSAB
analyte recovery is greater than 120% but
< 150%. If the ICSAB recovery falls within
50-79%, qualify sample results ≥ MDL as “J”
and non-detects as “UJ”. Reject (R) and red-line
all sample results (detects & non-detects) for
which the ICSAB analyte recovery is less than
50%. If the recovery is above 150%, reject (R)
and red-line only positive results.

A.1.16.3 ICP-MS Method

A.1.16.3.1 ICSA Solution:

For ICP-MS, are the ICSA “Found” analyte
values within the control limits of ±CRQL
of the true/established mean value? [ ]

ACTION:
If no, apply the following action to all
samples reported from the analytical run:

Flag (J) as estimated only sample results ≥ MDL
if the ICSA “Found” value is greater than
(True value+CRQL). Do not qualify non-detects.
If the ICSA “Found” value is less than
(True value-CRQL), flag the associated sample
detects as “J” and non-detects as “UJ”.

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A.1.16.3.3 **ICSAB Solution**

For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater? __ [ ] __ __

**ACTION:**

If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only detects (≥ MDL).

A.1.17 **Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A**

*Note:* Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only)

A.1.17.1 **Was Matrix Spike analysis performed:**

For each matrix type? [ ] __ __

For each SDG? [ ] __ __

On one of the SDG samples? [ ] __ __

For each concentration range (i.e., low, med., high)? [ ] __ __

For each analytical Method (ICP-AES, ICP-MS, Hg, CN) used? [ ] __ __

Was a spiked sample prepared and analyzed with the SDG samples? [ ] __ __

**ACTION:**

If no for any of the above, flag as  estimated(J) all the positive data for which a spiked sample was not analyzed.

**NOTE:**

If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.
A.1.17.2  Was a field blank or PE sample used for the spiked sample analysis?  

**ACTION:**
If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.

A.1.17.3  Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.

Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?

**NOTE:**
Disregard the out of control spike recoveries for analyses whose concentrations are greater than or equal to four times the spike added.

Are results outside the control limits (75-125%) flagged with Lab Qualifier "N" on Form I's and Form VA?

**ACTION:**
If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.

A.1.17.4  Aqueous

Are any spike recoveries:

(a) less than 30%?  

(b) between 30-74%?  

(c) between 126-150%?  

(d) greater than 150%?  

**ACTION:**
If the matrix spike recovery is less than 30%, reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as “J” and non-detects
as "UJ". If between 126-150%, flag (J)
all data ≥ MDL as "J". If greater than 150%,
reject (R) and red-line all associated data ≥ MDL.

(Note: Replace "N" with "J", "R" as appropriate.)

A.1.17.5 Soil/Sediment

Are any spike recoveries:
(a) less than 10%? [___]   [__]  [✓]
(b) between 10-74%? [___]   [__]  [✓]
(c) between 126-200%? [___]   [__]  [✓]
(d) greater than 200%? [___]   [__]  [✓]

Action:
If yes for any of the above, proceed
as follows:

If the matrix spike recovery is less
than 10%, reject (R) and red-line all
associated data (detects & non-detects);
if between 10-74%, qualify all associated
data ≥ MDL as "J" and non-detects as "UJ";
if between 126-200%, flag (J) all associated
data ≥ MDL as "J" If greater than 200%, reject
(R) and red-line all associated data ≥ MDL.
(Note: Replace "N" with "J" or "R" as appropriate.)

A.1.18 Lab Duplicates - Form VI

A.1.18.1 Was the lab duplicate analysis performed:

For each SDG? [✓]  [___]  [___]
On one of the SDG samples? [✓]  [___]  [___]
For each matrix type? [✓]  [___]  [___]
For each concentration range (low or med.)? [✓]  [___]  [___]
For each analytical Method
( ICP-AES/ICP-MS, Hg, CN) Used? [___]  [___]  [___]
Was a lab duplicate prepared and
analyzed with the SDG samples? [✓]  [___]  [___]
**ACTION:**
If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.

**NOTE:**
If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

A.1.18.2 Was a Field Blank or PE sample used for the Lab Duplicate analysis?  
[ ]

**ACTION:**
If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.

A.1.18.3 Circle on each Form VI all values that are:

RPD > 20%, or

Absolute Difference > CRQL

Are all values within control limits (RPD ≤ 20% or absolute difference ≤ ±CRQL)?  
[ ]

If no, are all results outside the control limits flagged with an "*" (Lab Qualifier) on Form VI and on all Form I's?  
[ ]

**ACTION:**
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

**NOTE:**
The laboratory is not required to report on Form VI the RPD when both values are non-detects.

A.1.18.4 **Aqueous**

A.1.18.4.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),
is any RPD > 20% but < 100%?  

is any RPD ≥ 100%?  

**ACTION:**
If the RPD is > 20% but < 100%, flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

*(NOTE: Replace "*" with "J" or "R" as appropriate.)*

A.1.18.4.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate values:

> ± CRQL?  

> ± 2xCRQL?  

**ACTION:**
If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 2xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but < 5xCRQL.

*(NOTE: Replace "*" with "J", "UJ" or "R" as appropriate.)  
2. If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.*

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?  

is any RPD ≥ 120%?  

**ACTION:**
If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample
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YES NO N/A

data ≥ CRQL. If the RPD is ≥ 120%, reject (R) and red-line the associated sample
data ≥ CRQL.

A.1.18.5.2 When the sample and/or duplicate value
<5xCRQL (substitute MDL for CRQL when MDL > CRQL),
is the absolute difference between sample
and duplicate:

> ± 2 x CRQL?

[____] ✓

> ± 4 x CRQL

[____] ✓

ACTION:
If the absolute difference is > 2 x CRQL,
flag all the associated sample results ≥ MDL
but < 5xCRQL as "J" and non-detects as "UJ".
If the absolute difference is > 4xCRQL, reject
(R) and red-line all the associated non-detects
and detects ≥ MDL but < 5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.
2. If one value is > CRQL and the other value is non-detect,
calculate the absolute difference between the value > CRQL
and the MDL, and use this difference to qualify sample results.

A.1.19 Field Duplicates

A.1.19.1 Aqueous Field Duplicates
Was an aqueous Field Duplicate pair
collected and analyzed?
(Check Sampling Trip Report)

[____] ✓ [____]

ACTION:
If yes, prepare a Form (Appendix A.4) for each
aqueous Field Duplicate pair. Report the sample
and Field Duplicate results on Appendix A.4 from
their respective Form I’s. Calculate and report RPD
on Appendix A.4 when sample and its Field Duplicate
values are both > 5xCRQL. Calculate and report the
absolute difference on Appendix A.4 when at least one
value (sample or duplicate) is < 5xCRQL. Evaluate the
aqueous Field Duplicate analysis in accordance with the
QC criteria stated in Sections A.1.19.2 and A.1.19.3.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is > CRQL and the other value is
   non-detect, calculate the absolute difference
   between the value > CRQL and the MDL, and use
   this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form {Appendix A.4} for Field Duplicates that have:

\[ \text{RPD} \geq 20\% \quad \text{or} \quad \text{Difference} > \pm \text{CRQL} \]

When sample and duplicate values are both \( \geq 5 \times \text{CRQL} \) (substitute MDL for CRQL when MDL > CRQL),

is any RPD \( \geq 20\% \)?

\[ \square \quad [\_] \quad \checkmark \]

is any RPD \( \geq 100\% \)?

\[ \square \quad [\_] \quad \checkmark \]

ACTION:
If the RPD is \( > 20\% \) but \( < 100\% \), flag (J) only
the associated sample and its Field Duplicate results \( \geq \text{CRQL} \). If the RPD is \( \geq 100\% \), reject (R)
and red-line only the associated sample and its
Field Duplicate result \( \geq \text{CRQL} \).

A.1.19.3 When the sample and/or duplicate value(s)
\( < 5 \times \text{CRQL} \) (substitute MDL for CRQL when MDL > CRQL),
is the absolute difference between sample
and duplicate:

\[ > \pm \text{CRQL}? \]

\[ \square \quad [\_] \quad \checkmark \]

\[ > \pm 2 \times \text{CRQL}? \]

\[ \square \quad [\_] \quad \checkmark \]

ACTION:
If the absolute difference is \( > \text{CRQL} \),
flag detects \( \geq \text{MDL} \) but \( < 5 \times \text{CRQL} \) as "J"
and non-detects as "UJ". If the difference
is \( > 2 \times \text{CRQL} \), reject (R) and red-line non-detects
and results ≥ MDL but < 5xCRQL of the sample and its Field Duplicate.

Soil/Sediment Field Duplicates

A.1.19.4 Was a soil field duplicate pair collected and analyzed? [___] [___] [✓]

(Check Sampling Trip Report)

ACTION:
If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than 5xCRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is < 5xCRQL. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and apply the criteria to qualify the results.

A.1.19.5 Circle on each Appendix A.4 all values that have:

RPD ≥ 35%, or Difference > ± 2xCRQL
When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%? [___] [___] [✓]

is any RPD ≥ 120%? [___] [___] [✓]

ACTION:
If the RPD is ≥ 35% but < 120%,
flag only the associated sample and its Field Duplicate results \( \geq CRQL \) as "J". If the RPD is \( \geq 120\% \), reject (R) and red-line only the sample and its Field Duplicate results \( \geq CRQL \).

A.1.19.5 When the sample and/or duplicate value(s) \(<5xCRQL \) (substitute MDL for CRQL when MDL \( > CRQL \)), is the absolute difference between sample and Field Duplicate:

\[
\begin{align*}
> \pm 2 \times CRQL? & \quad \text{[___]} \quad \checkmark \\
> \pm 4 \times CRQL? & \quad \text{[___]} \quad \checkmark
\end{align*}
\]

**ACTION:**
If the absolute difference is \( > 2xCRQL \), flag Sample and its Field Duplicate results \( \geq MDL \) but \( <5xCRQL \) as "J" and non-detects as "UJ". If the difference is \( >4xCRQL \), reject (R) and red-line non-detects and detects \( \geq MDL \) but \( <5xCRQL \) of the sample and its Field Duplicate.

A.1.20 **Laboratory Control Sample (LCS) - Form VII**

A.1.20.1 Was one LCS prepared and analyzed for:

Each SDG? \[\checkmark\] __ __

Each matrix type? \[\checkmark\] __ __

Each batch samples digested/distilled? For each Method(ICP-ABS, ICP-MS, Hg, CN) used? \[\checkmark\] __ __

Was an LCS prepared and analyzed with the samples? \[\checkmark\] __ __

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact CLP PO or TOPO for submittal of the LCS results. Flag (J) as estimated all the data for which an LCS was not analyzed.

**NOTE:**
If only one LCS was analyzed for
more than 20 samples, then the first
20 samples analyzed are not flagged (J),
but all additional samples must be
qualified (J).

A.1.20.2 **Aqueous LCS**

Circle on each Form VII the LCS percent
recoveries outside control limits 80-120%.

**NOTE:**
1. Use digested ICV as LCS for aqueous mercury
2. Use distilled ICV as LCS for aqueous cyanide

Is any LCS recovery:

Less than 50%?

[ ]

Between 50% and 79%?

[ ]

Between 121% and 150%?

[ ]

Greater than 150%?

[ ]

**ACTION:**
If the LCS recovery is less than 50%,
reject (R) and red-line all associated
sample data (detects & non-detects); for
a recovery between 50-79%, flag detects
as “J” all non-detects as “UJ”. If the LCS
recovery is between 121-150%, flag only
detects as “J”, if the recovery is greater
than 150%, reject (R) and red-line all detects.

A.1.20.3 **Solid LCS**

If an analyte's MDL is equal to or
greater than the true value of LCS,
disregard the "Action" below for that
analyte even though the LCS is out of
control limits.

Is the LCS "Found" value greater
than the Upper Control Limit
reported on Form VII?

[ ]

**ACTION:**
If yes, flag (J) all the associated detects \( \geq \) MDL as estimated (J).

Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII? 

ACTION: 
If yes, flag detects as "J" and non-dectes as "UJ".

A.1.21 ICP-AES/ICP-MS Serial Dilution - Form VIII

NOTE: Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 \( \times \) MDL.

A.1.21.1 Was a Serial Dilution analysis performed:

For each SDG? 

[✓] [ ] [ ]

On one of the SDG samples? 

[✓] [ ] [ ]

For each matrix type? 

[✓] [ ] [ ]

For each concentration range (low or med.)? 

[✓] [ ] [ ]

Was a Serial Dilution sample analyzed with the SDG samples? 

[ ] [ ] [✓]

ACTION: 
If no for any of the above, flag as estimated (J) detects \( \geq \) MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.

A.1.21.2 Was a Field Blank or PE sample used for the Serial Dilution Analysis?

[✓] [ ]

ACTION: 
If yes, flag as estimated (J) detects \( \geq \) MDL of all the SDG samples

A.1.21.3 Circle on Form VIII the Percent Differences (\%) between sample results and its dilution results that are outside the control limits \( \pm 10\% \)
when initial concentrations ≥ 50 x MDLs.

Are results outside the control limits flagged with an "E" (Lab Qualifier) on Form VIII and all Form I's? [____] [____] [✓]

ACTION:
If no, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.21.4 Are any %D values:

> 10%? [✓] [____]

≥ 100%? [✓] [____]

ACTION:
If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose raw data ≥ MDL;
if the %D is ≥ 100%, reject (R) and red-line all associated samples with raw data ≥ MDL.

(NOTE: Replace "E" with "J" or "R" as appropriate.)

A.1.22 Total/Dissolved or Inorganic/Total Analytes

A.1.22.1 Were any analyses performed for dissolved as well as total analytes on the same sample(s)? [___] [✓]

Were any analyses performed for inorganic as well as total analytes on the same sample(s)? [___] [✓]

ACTION:
If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic) and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:

(1) The dissolved (or inorganic) concentration is greater than total concentration, and
(2) greater than or equal to 5xMDL.

A.1.22.2 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%? [___] [✓]
A.1.22.3  Is any dissolved (or inorganic) concentration greater than its total concentration by more than 50%?  

ACTION:  
If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23  Field Blank - Form I  
NOTE: Designate "Field Blank" as such on Form I

A.1.23.1 Was a Field/Rinsate Bank collected and analyzed with the SDG samples?  

If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL (or 2xMDL when MDL>CRQL)?  

If yes, circle the Field Blank value on Form I that is greater than the CRQL, (or 2 x MDL when MDL > CRQL).  

Is any Field Blank value greater than CRQL also greater than the Preparation Blank value?  

If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria?  

ACTION:  
If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results) greater than the CRQL but less than the Field Blank value. Reject on Form I's soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.

If the Field Blank value is less than the
Prep. Blank value, do not qualify the sample results due to the Field Blank criteria.

NOTE:
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

A.1.24 Verification of Instrumental Parameters - Form IX, XA, XB, XI

A.1.24.1 Is verification report present for:

Method Detection Limits (Form IX - Annually)? [✓] [ ] [ ]

ICP-AES Interelement Correction Factors (Form XA & XB - Quarterly)? [ ] [ ] [ ]

ICP-AES & ICP-MS Linear Ranges (Form XI - Quarterly)? [✓] [ ] [ ]

ACTION:
If no, contact CLP PO/TOPO for submittal from the laboratory.

A.1.24.2 Method Detection Limits - Form IX

A.1.24.2.1 Are MDLs present on Form IX for:

All the analytes? [✓] [ ] [ ]

All the instruments used? [ ] [ ] [ ]

Digested and undigested samples and Calib.Blanks? [ ] [ ] [✓]

ICP-AES and ICP-MS when both instruments are used for the same analyte? [ ] [ ] [✓]

ACTION:
If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than ¼ CRQL.
A.1.24.2.2 Is MDL greater than the CRQL for any analyte?

If yes, is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?

ACTION:
If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.

A.1.24.3 Linear Ranges - Form XI

A.1.24.3.1 Was any sample result higher than the high linear range for ICP-AES or ICP-MS?

Was any sample result higher than the highest calibration standard for mercury or cyanide?

If yes for any of the above, was the sample diluted to obtain the result reported on Form I?

ACTION:
If no, flag (J) as estimated the affected detects (≥ MDL) reported on Form I.

A.1.25 ICP-MS Tune Analysis - Form XIV

A.1.25.1 Was the ICP-MS instrument tuned prior to calibration?

ACTION:
If no, reject (R) and red-line all sample data for which tuning was not performed.

A.1.25.2 Was the tuning solution analyzed or scanned at least five times consecutively?

Were all the required isotopes spanning the analytical range present in the tuning solution?

Was the mass resolution within
0.1 amu for each isotope in the tuning solution? [ ] [ ] [ ]

Was %RSD less than 5% for each isotope of each analyte in the tuning solution? [ ] [ ] [ ]

**ACTION:**
If no for any of the above, qualify all results ≥ MDL associated with that Tune as estimated “J”, and all non-detects associated with that Tune as “UJ”.

A.1.26 **ICP-MS Internal Standards - Form XV**

A.1.26.1 Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)? [ ] [ ] [ ]

Were all the target analyte masses bracketed by the masses of the five internal standards? [ ] [ ] [ ]

**ACTION:**
If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.

A.1.26.2 Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank? [ ] [ ] [ ]

If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed? [ ] [ ] [ ]

Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)? [ ] [ ] [ ]

**ACTION:**
If no for any of the above, flag detects as “J” and non-detects “UJ” of all the analytes with atomic masses between the atomic mass of the internal standard lighter
**SAMPLE CALCULATION**

EPA SAMPLE ID: VWAE-MW01-0310  
COMPOUND: Manganese  
CONCENTRATION: 2130 ug/L  
%Solids – NA  

Raw Data result: 2.1347 mg/L

2.1347 mg/L (1000ug/1mg) = 2134.7 ug/L

**FIELD DUPLICATE SAMPLE SUMMARY**

Note: All reported results are noted in the table below because the client requested that the MDL be used as reporting limit instead of the RL for this project. RPDs or absolute differences were calculated based on Region II guidelines: if results are ≥5X RL RPD is calculated, if results are <5X RL the absolute difference is calculated. Flags are applied to field duplicate pair only as follows: For RPD values - RPD ≥ 35% but <120% results are J, RPD ≥ 120%, results are R. For absolute difference values >+/− 2X RL results are J, >+/− 4X RL results are R.

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>Duplicate Sample ID:</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Analyte</strong></td>
<td><strong>Sample Conc.</strong></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments: No qualifications required.

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>Duplicate Sample ID:</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Analyte</strong></td>
<td><strong>Sample Conc.</strong></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments: No qualifications required.

Reviewer: [Signature]  
Date: 6/4/10

Vieques CTO-83  
SDG SJ0464  
Select Metals  
Page 1 of 1
December 27, 2010
SDG# SJ2254, Mitkem Laboratories
Vieques Island, Puerto Rico

Dear Mr. Acaron,

The following Data Validation report is provided as requested for the parameters noted in the table below for SDG # SJ2254. The data validation was performed in accordance with the SW-846 methods utilized by the laboratory, the Region II Standard Operating Procedures for the Validation of Organic Data Acquired Using SW-846 Methods (8260B-Rev 2, August 2008- SOP #HW-24 and 8270D-Rev 4, August 2008-SOP #HW-22), and professional judgment. Region II has not developed a validation checklist SOP for the methods used to assess the metals in this SDG (SW-846 methods 6010C). The Region II Standard Operating Procedure for the Evaluation of Metals Data for the CLP was used as applicable for the metals data. Region II flagging conventions were used. All areas of concern are discussed in the body of the report and a summary of data qualifications is provided.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Lab ID</th>
<th>Matrix</th>
<th>VOA</th>
<th>SVOA</th>
<th>Fe, Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW04-1110H</td>
<td>J2254-01A</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW04-1110</td>
<td>J2254-01F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW05-1110A</td>
<td>J2254-02A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-MW05-1110H</td>
<td>J2254-03A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-MW05-1110</td>
<td>J2254-03F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW05-1110A</td>
<td>J2254-04A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-EB01-110210</td>
<td>J2254-05F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VWAI-EB01-110210A</td>
<td>J2254-06A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-TB01-110210</td>
<td>J2254-07A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-MW02-1110H</td>
<td>J2254-08A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-MW02-1110</td>
<td>J2254-08F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW02-1110A</td>
<td>J2254-09A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-EB01-110310</td>
<td>J2254-10B</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VWAI-EB01-110310A</td>
<td>J2254-11A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-TB01-110310</td>
<td>J2254-12A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-MW03-1110H</td>
<td>J2254-13A</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW03-1110</td>
<td>J2254-13F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW03-1110A</td>
<td>J2254-14A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-MW07-1110H</td>
<td>J2254-15A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-MW07-1110</td>
<td>J2254-15F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW07-1110A</td>
<td>J2254-16A</td>
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<td>X</td>
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<td></td>
</tr>
<tr>
<td>VWAI-MW07P-1110</td>
<td>J2254-17F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VWAI-MW07P-1110A</td>
<td>J2254-18A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-EB01-110410</td>
<td>J2254-19F</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VWAI-EB01-1110A</td>
<td>J2254-20A</td>
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<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VWAI-TB01-110410</td>
<td>J2254-21A</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The following quality control samples were provided with this SDG: samples VWAI-TB01-110210, VWAI-TB01-110310 and VWAI-TB01-110410-trip blanks; samples VWAI-EB01-110210, VWAI-EB01-110210A, VWAI-EB01-110310, VWAI-EB01-110310A, VWAI-EB01-110410 and VWAI-EB01-1110A-equipment blanks; and sample VWAI-MW07P-1110-field duplicate of sample VWAI-MW07-1110.

The samples were evaluated based on the following criteria:

- Data Completeness
- Sample Condition
- Technical Holding Times
- GC/MS Tuning
- GC Performance
- ICP MS Tuning
- Initial/Continuing Calibrations
- ICSA/ICSAB Standards
- RL Standards
- Blanks
- Internal Standards
- Surrogate Recoveries
- Laboratory Control Samples
- Matrix Spike Recoveries
- Matrix Duplicate RPDs
- Serial Dilutions
- Field Duplicates
- Identification/Quantitation
- Reporting Limits
- Tentatively Identified Compounds

* indicates that qualifications were not required based on this criteria

**Overall Evaluation of Data/Potential Usability Issues**

A summary of qualifications applied to the sample results are noted below for the fractions validated. Specific details regarding qualification of the data are addressed in the Specific Evaluation section of this narrative. If an issue is not addressed there were no actions required based on unmet quality criteria. When more than one qualifier is associated with a compound/analyte the validator has chosen the qualifier that best indicates possible bias in the results and flagged the data accordingly. However,
information regarding all quality control issues is provided in the body of the report and on the qualification summary page. Please note that when a compound or analyte is flagged due to blank contamination the BL qualifier code takes precedence over all other qualifier codes except a code that explains rejected data.

**VOA**

No qualifications to the data were required.

**SVOA**

No qualifications to the data were required.

**Select Filtered Metals**

Blank contamination was noted in one of the associated CCB samples. Qualifications were required.

The laboratory did not perform a matrix spike or a serial dilution in this SDG. These QC samples are required by Region II. Qualifications were required.

**Specific Evaluation of Data**

**Data Completeness**

The SDG was received complete and intact. Resubmissions were not required.

**Technical Holding Times**

According to chain of custody records, sampling was performed on 11/2-4/10 and samples were received at the laboratory 11/3-5/10. All sample preparation and analysis was performed within Region II and/or method holding time requirements.

**Blanks**

**Select Filtered Metals**

One associated blank exhibited contamination as noted in the following table. Please see the Glossary of Qualification Flags and Abbreviations for details.

<table>
<thead>
<tr>
<th>Blank ID</th>
<th>Analyte</th>
<th>Concentration</th>
<th>Action Level</th>
<th>Q Flag</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCB 2</td>
<td>iron</td>
<td>443B ug/L</td>
<td>LOD</td>
<td>U at LOD</td>
</tr>
</tbody>
</table>

Associated samples and required qualifications are noted in the following table.
Matrix Spike

Select Filtered Metals

The laboratory did not perform a matrix spike sample on a sample from this SDG. Region II required that all positive results be qualified as estimated J because of this. Therefore, the reported positive results for iron and manganese were qualified as estimated J with a qualifier code of OT.

Serial Dilution

Select Filtered Metals

The laboratory did not perform a serial dilution sample on a sample from this SDG. Region II required that all positive results be qualified as estimated J because of this. Therefore, the reported positive results for iron and manganese were qualified as estimated J with a qualifier code of OT.

A summary of qualifications required is provided on the following page. Please do not hesitate to contact DataQual ES with any questions regarding this validation report.

Sincerely,

Laura Maschhoff
President

Jacqueline Cleveland
Vice President
Summary of Data Qualifications

**VOA**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>No qualifications</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**SVOA**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>No qualifications</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Select Filtered Metals**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Analyte</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW03-1110, VWAI-MW04-1110</td>
<td>iron</td>
<td>+B</td>
<td>U at LOD</td>
<td>MBL</td>
</tr>
<tr>
<td>all samples</td>
<td>iron, manganese</td>
<td>+</td>
<td>J</td>
<td>OT</td>
</tr>
</tbody>
</table>
Glossary of Qualification Flags and Abbreviations

Qualification Flags (Q-Flags)

U  not detected above the reported sample quantitation limit
J  estimated value
UJ reported quantitation limit is qualified as estimated
N  analyte has been tentatively identified
JN analyte has been tentatively identified, estimated value
R  result is rejected; the presence or absence of the analyte cannot be verified

Method/Preparation/Field QC Blank Qualification Flags (Q-Flags)

Organic Methods

NA  The sample result for the blank contaminant is greater than the RL (2X sample RL for common laboratory contaminants) when the blank value is less than the RL. The sample result for the blank contaminant is not qualified with any blank qualifiers.
U* The sample result for the blank contaminant is less than the RL (2X sample RL for common laboratory contaminants) but greater than the MDL when the blank value is less than the RL. The sample result for the blank contaminant is qualified as non-detect U at the reported concentration.
RL** The sample result for the blank contaminant is less than the RL (2X sample RL for common laboratory contaminants) but greater than the MDL when the blank value is less than the RL. The sample result for the blank contaminant is changed to the RL and qualified as non-detect U.

Inorganic Methods

ICB/CCB/PB Action:

No Action - The sample result is greater than the RL and greater than ten times (10X) the blank value.
U - The sample result is greater than or equal to the MDL but less than or equal to the RL , result is reported as non-detect at the RL* or at the reported concentration**, when the ICB/CCB/PB result is less or greater than the RL.

* This guideline is used when the laboratory is reporting non-detects to the MDL. ** This guideline is used when the laboratory is reporting non-detects to the RL.
Glossary of Qualification Flags and Abbreviations, continued

R - Sample result is greater than the RL and less than the ICB/CCB/PB value when the ICB/CCB/PB value is greater than the RL.

J - Sample result is greater than the ICB/CCB/PB value but less than 10X the ICB/CCB/PB value when ICB/CCB/PB value is greater than the RL.

J/UJ - Sample result is less than 10X RL when blank result is below the negative RL.

* This guideline is used when the laboratory is reporting non-detects to the MDL. ** This guideline is used when the laboratory is reporting non-detects to the RL.

Field QC Blank action:

Note – Use field blanks to qualify data only if field blank results are greater than prep blank results.

Do not use rinsate blank associated with soils to qualify water samples and vice versa.

No Action - The sample result is greater than the RL and greater than ten times (10X) the blank value.

U - The sample result is greater than or equal to the MDL but less than or equal to the RL, result is reported as non-detect at the RL* or at the reported concentration**, when the FB result is less or greater than the RL.

R - Sample result is greater than the RL and less than the FB value when the FB value is greater than the RL.

J - Sample result is greater than the FB value but less than 10X the FB value when FB value is greater than the RL.

* This guideline is used when the laboratory is reporting non-detects to the MDL. ** This guideline is used when the laboratory is reporting non-detects to the RL.

General Abbreviations

LOD level of detection
RL reporting limit (equivalent to the LOD)
PQL practical quantitation limit
IDL instrument detection limit
MDL method detection limit
+ positive result
- non-detect result
<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>TN</td>
<td>Tune</td>
</tr>
<tr>
<td>BSL</td>
<td>Blank Spike/LCS - High Recovery</td>
</tr>
<tr>
<td>BSH</td>
<td>Blank Spike/LCS - Low Recovery</td>
</tr>
<tr>
<td>BD</td>
<td>Blank Spike/Blank Spike Duplicate (LCS/LCSD) Precision</td>
</tr>
<tr>
<td>BRL</td>
<td>Below Reporting Limit</td>
</tr>
<tr>
<td>ISL</td>
<td>Internal Standard - Low Recovery</td>
</tr>
<tr>
<td>ISH</td>
<td>Internal Standard - High Recovery</td>
</tr>
<tr>
<td>MSL</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - Low Recovery</td>
</tr>
<tr>
<td>MSH</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - High Recovery</td>
</tr>
<tr>
<td>MI</td>
<td>Matrix interference obscuring the raw data</td>
</tr>
<tr>
<td>MDP</td>
<td>Matrix Spike/Matrix Spike Duplicate Precision</td>
</tr>
<tr>
<td>2S</td>
<td>Second Source - Bad reproducibility between tandem detectors</td>
</tr>
<tr>
<td>SSL</td>
<td>Spiked Surrogate - Low Recovery</td>
</tr>
<tr>
<td>SSH</td>
<td>Spiked Surrogate - High Recovery</td>
</tr>
<tr>
<td>SD</td>
<td>Serial Dilution Reproducibility</td>
</tr>
<tr>
<td>ICL</td>
<td>Initial Calibration - Low Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICH</td>
<td>Initial Calibration - High Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICB</td>
<td>Initial Calibration - Bad Linearity or Curve Function</td>
</tr>
<tr>
<td>CCL</td>
<td>Continuing Calibration - Low Recovery or %Difference</td>
</tr>
<tr>
<td>CCH</td>
<td>Continuing Calibration - High Recovery or %Difference</td>
</tr>
<tr>
<td>LD</td>
<td>Lab Duplicate Reproducibility</td>
</tr>
<tr>
<td>HT</td>
<td>Holding Time</td>
</tr>
<tr>
<td>PD</td>
<td>Pesticide Degradation</td>
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<tr>
<td>2C</td>
<td>Second Column - Poor Dual Column Reproducibility</td>
</tr>
<tr>
<td>LR</td>
<td>Concentration Exceeds Linear Range</td>
</tr>
<tr>
<td>MBL, EBL, FBL or TBL</td>
<td>Blank Contamination</td>
</tr>
<tr>
<td>RE</td>
<td>Redundant Result - due to Re-analysis or Re-extraction</td>
</tr>
<tr>
<td>DL</td>
<td>Redundant Result - due to Dilution</td>
</tr>
<tr>
<td>FD</td>
<td>Field Duplicate</td>
</tr>
<tr>
<td>OT</td>
<td>Other - explained in data validation report</td>
</tr>
<tr>
<td>%SOL</td>
<td>High moisture content</td>
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</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Matrix: WATER  
Sample wt/vol: 5.00 (g/mL)  
Level: LOW  
% Moisture: not dec.  
GC Column: DB-624  
Soil Extract Volume:  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: µG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50</td>
<td>U</td>
<td>0.41</td>
<td>0.50</td>
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</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
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<td>J</td>
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<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
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<td>78-87-5</td>
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<td>1.0</td>
<td>U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
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</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrx: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: TRACE/LOW/MED LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (nm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

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<td>J</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
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<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0</td>
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<td>5.0</td>
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Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J2254  
Matrix: WATER  
Sample wt/vol: 5.00 (g/mL)  
Level: LOW  
% Moisture: not dec.  
GC Column: DB-624  
ID: 0.25 (mm)  
Soil Extract Volume: 5.0 (uL)  
Purge Volume: 5.0 (mL)  

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<td>0.41</td>
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<td>5.0</td>
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<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0 μg/L</td>
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<td>0.61</td>
<td>1.0</td>
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Lab Code: MITKEM
Matrix: WATER
Sample wt/vol: 5.00 (g/mL)
Level: LOW
% Moisture: not dec.
GC Column: DB-624
Soil Extract Volume: 5.0 (mL)

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Matrix: WATER
Sample wt/vol: 5.00 (g/mL)
Level: LOW
% Moisture: not dec.
GC Column: DB-624
Soil Extract Volume: 5.0 (mL)
Purge Volume: 5.0 (mL)

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<th>CAS NO.</th>
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<td>71-43-2</td>
<td>Benzene</td>
<td>0.50 U</td>
<td></td>
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</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624  
Soil Extract Volume: 0.25 (mm) (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: µG/L</th>
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Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J2254  
Matrix: (SOIL/SED/WATER)  
WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED)  
LOW  
% Moisture: not dec.  
GC Column: DB-624  
ID: 0.25 (mm)  
Soil Extract Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
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<tr>
<th>CAS NO.</th>
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<td>5.0</td>
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**Lab Name:** MITKEM LABORATORIES  
**Contract:**  
**Lab Code:** MITKEM  
**Case No.:** J2254  
**Mod. Ref No.:** SDG No.: SJ2254  
**Matrix:** (SOIL/SED/WATER) WATER  
**Sample wt/vol:** 5.00 (g/mL) ML  
**Lab Sample ID:** J2254-07A  
**Lab File ID:** V6H7334.D  
**Level:** (TRACE/LOW/MED) LOW  
**% Moisture:** not dec.  
**Date Received:** 11/03/2010  
**Part #:** 0.25 (mm)  
**GC Column:** DB-624  
**Dilution Factor:** 1.0  
**Date Analyzed:** 11/04/2010  
**Soil Extract Volume:** (µL)  
**Soil Aliquot Volume:** (µL)  
**Purge Volume:** 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: µg/L</th>
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</tbody>
</table>
### Lab Name: MITKEM LABORATORIES

### Contract:

### Lab Code: MITKEM

### Case No.: J2254

### Mod. Ref No.:

### SDG No.: SJ2254

### Matrix: (SOIL/SED/WATER) WATER

### Sample wt/vol: 5.00 (g/mL) ML

### Level: (TRACE/LOW/MED) LOW

### % Moisture: not dec.

### GC Column: DB-624 0.25 (mm)

### Soil Extract Volume: 5.0 (mL)

### Soil Aliquot Volume: 0.25 (mL)

### Purge Volume: 5.0 (mL)

### CAS NO. | COMPOUND          | CONCENTRATION: µG/L | Q | DL    | LOD   | LOQ   |
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
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</tbody>
</table>

---

**Note:**

- **GC Column:** DB-624
- **Soil Extract Volume:** 5.0 (mL)
- **Soil Aliquot Volume:** 0.25 (mL)
- **Purge Volume:** 5.0 (mL)
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Matrix: WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM  Case No.: J2254
Matrix: (SOIL/SED/WATER)  WATER
Sample wt/vol:  5.00 (g/mL)  ML
Level: (TRACE/LOW/MED)  LOW
% Moisture: not dec.
GC Column: DB-624  ID: 0.25 (mm)  Dilution Factor: 1.0
Soil Extract Volume:  (uL)  Soil Aliquot Volume:  (uL)
Purge Volume:  5.0  (mL)

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: WATER
Sample wt/vol: 5.00 (g/mL)
Level: LOW
Soil Extract Volume: (uL)
GC Column: DB-624
Soil Aliquot Volume: (uL)
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<th>DL</th>
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<tr>
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<td>1.0 U</td>
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<tr>
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<td>0.61</td>
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**Lab Name:** MITKEM LABORATORIES  
**Lab Code:** MITKEM  
**Case No.:** J2254  
**Matrix:** (SOIL/SED/WATER) WATER  
**Sample wt/vol:** 5.00 (g/mL) ML  
**Level:** (TRACE/LOW/MED) LOW  
**% Moisture:** not dec.  
**GC Column:** DB-624  
**Soil Extract Volume:** (uL)  
**Soil Aliquot Volume:** (uL)  
**Purge Volume:** 5.0 (mL)  

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<td>78-87-5</td>
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Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Contract:  
Case No.: J2254  
Mod. Ref No.:  
SDG No.: SJ2254  
Lab Sample ID: J2254-13A  
Lab File ID: V6H7449.D  
Date Received: 11/05/2010  
Date Analyzed: 11/09/2010  
GC Column: DB-624  
ID: 0.25 (mm)  
Dilution Factor: 1.0  
Soil Extract Volume: (uL)  
Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)  

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254

Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

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Lab Name: MITKEM LABORATORIES

Lab Code: MITKEM

Case No.: J2254

Matrix: (SOIL/SED/WATER) WATER

Sample wt/vol: 5.00 (g/mL) ML

Level: (TRACE/LOW/MED) LOW

% Moisture: not dec.

GC Column: DB-624 ID: 0.25 (mm)

Dilution Factor: 1.0

Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)

Purge Volume: 5.0 (mL)

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Dilution Factor: 1.0
Purge Volume: 5.0 (mL)

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: WATER
Sample wt/vol: 5.00 (g/mL)
Level: LOW
% Moisture: not dec.
GC Column: DB-624
Soil Extract Volume: 5.0 (uL)

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Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Matrix: WATER  
Sample wt/vol: 5.00 (g/mL)  
Level: LOW  
% Moisture: not dec.  
GC Column: DB-624  
Soil Extract Volume: 5.0 (mL)  
Purge Volume: 5.0 (mL)

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<td>U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
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## Lab Name: MITKEM LABORATORIES

### Contract:

### Lab Code: MITKEM

### Case No.: J2254

### Mod. Ref No.: ...

### SDG No.: SJ2254

### Matrix: (SOIL/SED/WATER)

### WATER

### Sample wt/vol: 5.00 (g/mL) ML

### Level: (TRACE/LOW/MED)

### LOW

### % Moisture: not dec.

### GC Column: DB-624

### ID: 0.25 (mm)

### Dilution Factor: 1.0

### Soil Extract Volume: (uL)

### Soil Aliquot Volume: (uL)

### Purge Volume: 5.0 (mL)

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<th>CAS NO.</th>
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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Contract:
Mod. Ref No.:
SDG No.: SJ2254

Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

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Date Received: 11/05/2010
Date Analyzed: 11/09/2010
Lab Sample ID: J2254-19F
Lab File ID: V6H7443.D

% Moisture: 0.00

CAS NO. 18.0..18.1 SW846
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J2254  
Contract:  
Mod. Ref No.:  
SDG No.: SJ2254  
Lab Sample ID: J2254-20A  
Lab File ID: V6H7444.D  
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ID: 0.25 (mm)  
Soil Extract Volume:  
Soil Aliquot Volume:  
Purge Volume: 5.0 (mL)  

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

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Date Received: 11/05/2010
Date Analyzed: 11/09/2010
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL)
Purge Volume: 5.0 (mL)

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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Mod. Ref No.: SDG No.: SJ2254
Matrix: (SOIL/SED/WATER) WATER
Lab Sample ID: J2254-08FMSD
Sample wt/vol: 5.00 (g/mL) ML
Lab File ID: V6H7340.D
Level: (TRACE/LOW/MED) LOW
Date Received: 11/04/2010
% Moisture: not dec.
Date Analyzed: 11/04/2010
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

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Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J2254  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL)  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Soil Extract Volume: (uL)  
Purge Volume: 5.0 (mL)

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<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: µg/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>51</td>
<td></td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>49</td>
<td></td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>52</td>
<td></td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J2254  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Soil Extract Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: µG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
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<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>52</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
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</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>51</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
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<tr>
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<td>1,2-Dichloropropane</td>
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<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Lab Sample ID: J2254-01E
Lab File ID: S3H0517.D

Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Lev el: (LOW/MED) LOW

% Moisture: Decanted: (Y/N)
Concentrated, Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00

GPC Cleanup: (Y/N) N
pH: Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
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<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tr>
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<td>2-Methylnaphthalene</td>
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<td>0.94</td>
<td>1.0</td>
<td>1.0</td>
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<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>1.4</td>
<td>1.3</td>
<td>5.0</td>
<td>5.0</td>
<td></td>
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**Lab Name:** MITKEM LABORATORIES  
**Contract:**  
**Lab Code:** MITKEM  
**Case No.:** J2254  
**Mod. Ref No.:**  
**SDG No.:** SJ2254  
**Matrix:** (SOIL/SED/WATER) WATER  
**Lab Sample ID:** J2254-03E  
**Sample wt/vol:** 1000 (g/mL) ML  
**Lab File ID:** S3H0518.D  
**Level:** (LOW/MED) LOW  
**% Moisture:** Decanted: (Y/N)  
**Concentrated Extract Volume:** 1000 (uL)  
**Injection Volume:** 1.0 (uL)  
**GPC Cleanup:** (Y/N) N  
**pH:**  
**Dilution Factor:** 1.0  

<table>
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<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tbody>
<tr>
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<td>Naphthalene</td>
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<td></td>
<td>0.96</td>
<td>1.0</td>
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<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>20</td>
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<td>0.94</td>
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<td>117-81-7</td>
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<td>5.0</td>
<td>U</td>
<td>1.3</td>
<td>5.0</td>
<td>5.0</td>
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</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N
pH: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tbody>
<tr>
<td>91-20-3</td>
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<td>0.96</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>0</td>
<td>0.94</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>0</td>
<td>1.3</td>
<td>5.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J2254  
Matrix: (SOIL/SED/WATER)  
WATER  
Sample wt/vol: 1000 (g/mL)  
ML  
Level: (LOW/MED)  
LOW  
% Moisture:  
Decanted: (Y/N)  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL)  
GPC Cleanup: (Y/N)  
N  
Date Received: 11/04/2010  
Date Extracted: 11/05/2010  
Date Analyzed: 11/24/2010  
CAS NO. | COMPOUND | CONCENTRATION: UG/L | Q | DL | LOD | LOQ  
--- | --- | --- | --- | --- | --- | ---  
91-20-3 | Naphthalene | 1.0 | U | 0.96 | 1.0 | 1.0  
91-57-6 | 2-Methylnaphthalene | 1.0 | U | 0.94 | 1.0 | 1.0  
117-81-7 | Bis(2-ethylhexyl)phthalate | 5.0 | U | 1.3 | 5.0 | 5.0
<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
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<td>0.96</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>0</td>
<td>0.94</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>0</td>
<td>1.3</td>
<td>5.0</td>
<td>5.0</td>
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<td>CAS NO.</td>
<td>COMPOUND</td>
<td>CONCENTRATION: UG/L</td>
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<td>DL</td>
<td>LOD</td>
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</tr>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.0</td>
<td>U</td>
<td>0.96</td>
<td>1.0</td>
<td>1.0</td>
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<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>U</td>
<td>0.94</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>U</td>
<td>1.3</td>
<td>5.0</td>
<td>5.0</td>
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</tbody>
</table>
Lab Name: MITKEM LABORATORIES  
Lab Code: MITKEM  
Case No.: J2254  
Contract:  
Mod. Ref No.:  
SDG No.: SJ2254  
Lab Sample ID: J2254-15E  
Lab File ID: S3H0525.D  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 1000 (g/mL) ML  
Level: (LOW/MED) LOW  
% Moisture:  
Decanted: (Y/N)  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL)  
GPC Cleanup: (Y/N) N  
GPC Factor: 1.00  
Date Received: 11/05/2010  
Date Extracted: 11/05/2010  
Date Analyzed: 11/24/2010  
CAS NO. | COMPOUND | CONCENTRATION: UG/L | Q | DL | LOD | LOQ  
---|---|---|---|---|---|---  
91-20-3 | Naphthalene | 7.9 | 0.96 | 1.0 | 1.0 |  
91-57-6 | 2-Methylnaphthalene | 7.7 | 0.94 | 1.0 | 1.0 |  
117-81-7 | Bis(2-ethylhexyl)phthalate | 5.0 | 1.3 | 5.0 | 5.0 |
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Case No.: J2254
Contract:
Mod. Ref No.:
SDG No.: SJ2254
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Lab Sample ID: J2254-17E
Lab Code: MITKEM
Case No.: J2254
Mod. Ref No.:
SDG No.: SJ2254
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Lab Sample ID: J2254-17E
Lab File ID: S3H0526.D
Level: (LOW/MED) LOW
Extraction: (Type) SEPF
% Moisture: Decanted: (Y/N) Date Received: 11/05/2010
Concentrated Extract Volume: 1000 (uL) Date Extracted: 11/05/2010
Injection Volume: 1.0 (uL) GPC Factor: 1.00 Date Analyzed: 11/25/2010
GPC Cleanup: (Y/N) N pH: 1.0 Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tbody>
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<tr>
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<td>5.0 U</td>
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<td>1.3</td>
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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM Case No.: J2254
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N pH: Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tbody>
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<td>Naphthalene</td>
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<td>0</td>
<td>0.96</td>
<td>1.0</td>
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<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>1.0</td>
<td>0</td>
<td>0.94</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>5.0</td>
<td>0</td>
<td>1.3</td>
<td>5.0</td>
<td>5.0</td>
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</tbody>
</table>
Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL)
GPC Cleanup: (Y/N) N

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
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<th>Q</th>
<th>DL</th>
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Lab Name: MITKEM LABORATORIES
Lab Code: MITKEM  Case No.: J2254
Matrix: (SOIL/SED/WATER)  WATER
Sample wt/vol:  1000 (g/mL)  ML
Level: (LOW/MED)  LOW
% Moisture:  Decanted: (Y/N)  Date Received:  11/04/2010
Concentrated Extract Volume:  1000 (uL)  Date Extracted:  11/05/2010
Injection Volume:  1.0 (uL)  GPC Factor:  1.00  Date Analyzed:  11/24/2010

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
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<tbody>
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<td>2-Methylnaphthalene</td>
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</table>
**U.S. EPA - CLP**

**INORGANIC ANALYSIS DATA SHEET**

**Lab Name:** Mitkem Laboratories  
**Lab Code:** MITKEM  
**Matrix (soil/water):** WATER  
**Level (low/med):** MED  
**% Solids:** 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
<th>MDL</th>
<th>LOD</th>
<th>PQL</th>
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<tbody>
<tr>
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<td>Iron</td>
<td>100</td>
<td>U</td>
<td>P</td>
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<td>P</td>
<td>10.0</td>
<td>10.0</td>
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</tbody>
</table>

**Comments:**

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**Form I - IN**

**SW846**

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SM_002

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U50

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0328

---
Lab Name: Mitkem Laboratories
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
<th>MDL</th>
<th>LOD</th>
<th>PQL</th>
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<td>P</td>
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<td></td>
<td>31.0</td>
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<td>200</td>
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Comments:

JMC 22-710
**INORGANIC ANALYSIS DATA SHEET**

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
<th>MDL</th>
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<tbody>
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<td>P</td>
<td></td>
<td>31.0</td>
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<td>P</td>
<td></td>
<td>10.0</td>
<td>10.0</td>
<td>50.0</td>
</tr>
</tbody>
</table>

**Comments:**

---

**Lab Name:** Mitkem Laboratories

**Lab Code:** MITKEM

**Matrix (soil/water):** WATER

**Level (low/med):** MED

**Date Received:** 11/03/2010

**Concentration Units:** ug/L or mg/kg dry weight

---

**EPA SAMPLE NO.**

**Contract:** 933562, N62

**Case No.:**

**SAS No.:**

**SDG No.:** SJ2254

**Lab Sample ID:** J2254-03
Lab Name: Mit kem Laboratories
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
<th>MDL</th>
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<tbody>
<tr>
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<td>JOT</td>
<td>10.0</td>
<td>10.0</td>
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</tbody>
</table>

Comments:
I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:

   All samples were prepared within the method-specified holding times.

B. Sample Analysis:

   All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 6010C

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: ICP_W_PR(3005A)

V. INSTRUMENTATION

The following instrumentation was used to perform the analyses:

Instrument Code: OPTIMA2
Instrument Type: ICP
Description: Optima 3100 XL
Manufacturer: Perkin-Elmer
Model: 3100 XL

VI. ANALYSIS

A. Calibration:

   Calibrations met the method/SOP acceptance criteria.

B. Blanks:
All method blanks were within the acceptance criteria.

C. Spikes:

1. Laboratory Control Spikes (LCS/LCSD):
   Percent recoveries and RPD for lab control samples were within the QC limits.

2. Matrix spike (MS):
   No client-requested MS analysis was included in this SDG.

D. Post Digestion/Distillation Spike (PDS):
   No PDS was performed on any sample in this SDG.

E. Duplicate sample:
   No client requested duplicate analysis was included in this SDG.

F. Serial Dilution (SD):
   No SD was performed on any sample in this SDG.

G. Samples:
   No other unusual occurrences were noted during sample analysis.
   No sample in this SDG required reanalysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Mitkem, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: [Signature]

Date: 12/3/10
**CHAIN OF CUSTODY RECORD**

<table>
<thead>
<tr>
<th>Lab Id:</th>
<th>Sample Id:</th>
<th>Date:</th>
<th>Time:</th>
<th>Type</th>
<th>Matrix</th>
<th># of Amber Glass</th>
<th># of Plastic</th>
<th>8260 Vocs</th>
<th>8260N Vocs</th>
<th>9836 Vocs</th>
<th>9836H Vocs</th>
<th>HOLD</th>
<th>Notes</th>
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<tbody>
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<td>GW</td>
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</tr>
<tr>
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<td>09:20</td>
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<td>2</td>
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</tr>
<tr>
<td>05</td>
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<td>11/2/2010</td>
<td>11:25</td>
<td>GW</td>
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</table>

**Notes:**
- Hold Vocs w/ HCl present
- 1 my Ascorbic Acid Used
- Hold Vocs w/ HCl present
- 1 my Ascorbic Acid Used
- All 8260 Vocs are for List I Vocs

**Condition upon receipt:** Ambient 10°C
## CHAIN OF CUSTODY RECORD

**Special Handling:**
- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rushes.
- Samples disposed of after 30 days unless otherwise instructed.

**Report To:** CH2M HILL  
**Invoice To:** CH2M HILL

**Project No.:** 392485, FL FK

**Site Name:** J01-I  
**Location:** Vice  
**State:** PA

**Project Mgr.:** Steven Bench  
**P.O. No.:**  
**RQN.:**

**Sampler(s):** Kenji Butler / Chris Reed

---

**List preservative code below:**

<table>
<thead>
<tr>
<th>Container</th>
<th>Analyses</th>
</tr>
</thead>
<tbody>
<tr>
<td>G</td>
<td>Grab</td>
</tr>
<tr>
<td>C</td>
<td>Composite</td>
</tr>
</tbody>
</table>

**QA/QC Reporting Level:**

- Level I
- Level II
- Level III
- Level IV
- Other

**State specific reporting standards:**

- DW = Drinking Water
- GW = Groundwater
- WW = Wastewater
- O = Oil
- SW = Surface Water
- SO = Soil
- SL = Sludge
- A = Air

**Lab Id:** | **Sample Id:** | **Date:** | **Time:** | **Matrix** | **# of VOA Vials** | **# of Amber Glass** | **# of Plastic** | **State:** |
<table>
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<tr>
<td>01</td>
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<td>11/2/2010</td>
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<td>2</td>
<td>2/1/1</td>
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<tr>
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<td>2</td>
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<tr>
<td>05</td>
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<td>11:25</td>
<td>G</td>
<td>6</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

**Notes:**

- 0.45 micron Filtration on Poly
- 0.45 micron Filtration on Poly

---

**Relinquished by:**

- EDI Format

- E-mail to

- EDD Format

- Condition upon receipt: Iced: Ambient: C

---

**Received by: 11/2/2010 12:30**

---

**Date:** 11/3/2010 08:00
### CHAIN OF CUSTODY RECORD

**Special Handling:**
- TAT - Indicate Date Needed: __ ___ __
- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rushes.
- Samples disposed of after 30 days unless otherwise instructed.

**MITKEM LABORATORIES**

**CHAIN OF CUSTODY RECORD**

**Report To:** CH2M HILL

**Invoice To:** CH2M HILL

**Project No.:** 392485.FT.PK

**Site Name:** AOX-I

**Location:** Vieques

**State:** PR

**Project Mgr.:** Stephen Brown

**P.O. No.:** RQN:

**Sampler(s):** Kenji Butler / Chris Reed

---

<table>
<thead>
<tr>
<th>G</th>
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</tr>
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</table>

**List preservative code below:**

- DW = Drinking Water
- GW = Groundwater
- WW = Wastewater

**Contents:**

- 

**QA/QC Reporting Level:**
- Level I
- Level II
- Level III
- Level IV
- Other

**State specific reporting standards:**

- Hold for Analysis
- 2mg AAS used
- 2mg AAS used
- 2mg AAS used
- 2mg AAS used

**Condition upon receipt:**
- Salted: Ambient
- 3°C

---

**Relinquished by:** [Signature]

**Received by:** [Signature]

**Date:** 11/30

**Time:** 08:57

175 Metro Center Boulevard • Warwick, RI 02886-1755 • 401-732-3400 • Fax 401-732-3499 • www.mitkem.com
Special Handling:
- TAT- Indicate Date Needed: ____ __
  - MITKEM LABORATORIES CHAIN OF CUSTODY RECORD
  - All TATs subject to laboratory approval.
  - Min. 24-hour notification needed for rushes.
  - Samples disposed of after 30 days unless otherwise instructed.

Project No.: 392485, FT, EK
Site Name: AOC-1
Location: Vieques State: PR
Sampler(s): Kenji Butler / Chris Reed

<table>
<thead>
<tr>
<th>Sample Id</th>
<th>Date</th>
<th>Time</th>
<th>Type</th>
<th>Matrix</th>
<th>No. of VOA Vials</th>
<th>No. of Amber Glass</th>
<th>No. of Clear Glass</th>
<th>No. of Plastic</th>
<th>List of SVOC</th>
<th>Analyses</th>
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</thead>
<tbody>
<tr>
<td>VWAF-MW02-110</td>
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<td>DW</td>
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<td>0925</td>
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<td>DW</td>
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<td>0925</td>
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<tr>
<td>VWAF-MW02-K5</td>
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<td>0925</td>
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<td>1110</td>
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</table>

QA/QC Reporting Level:
- Level I
- Level II
- Level III
- Level IV
- Other

State specific reporting standards:

Condition upon receipt:
- temper: 4°C
- lead: 0%

Relinquished by: [Signature]
Received by: [Signature]
Date: 11/4/10
Time: 08:57

E-mail to: ____________________________
EDD Format: ____________________________
## CHAIN OF CUSTODY RECORD

**Report To:** CH2M Hill  
**Invoice To:** CH2M Hill  
**Project No.:** 392485, FL.FK  
**Site Name:** Aol-I  
**Location:** Vieques  
**State:** PR

### Special Handling:
- **TAT** - Indicate Date Needed: ___
- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rushes.
- Samples disposed of after 30 days unless otherwise instructed.

### Project Mgr.: Stephen Brand

### List preservative code below:

|  |  |  |  
|---|---|---|---|
| 1 | Na$_2$S$_2$O$_3$ | 2 | HCl | 3 | H$_2$SO$_4$ | 4 | HNO$_3$ | 5 | NaOH | 6 | Ascorbic Acid | 7 | CH$_3$OH | 8 | NaHSO$_4$ | 9 | H$_3$PO$_4$ | 10 | Unpreserved | 11 |  

### G=Grab C=Composite

### Analyses:

<table>
<thead>
<tr>
<th>Lab Id</th>
<th>Sample Id</th>
<th>Date</th>
<th>Time</th>
<th>Type</th>
<th>Matrix</th>
<th>No. of VOA Vials</th>
<th>No. of Amber Glass</th>
<th>No. of Clear Glass</th>
<th>No. of Plastic</th>
<th>List I Vol (AA)</th>
<th>List II Vol (AA)</th>
<th>List III Vol (AA)</th>
<th>List IV Vol (AA)</th>
<th>TOC</th>
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<td>17</td>
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### Notes:
- **QA/QC Reporting Level**
  - Level I
  - Level II
  - Level III
  - Level IV
  - Other
- **State specific reporting standards:**
  - Other ______

### Relinquished by:  
**Received by:**  
**Date:** 12/30  
**Time:** 08:05

---

**Condition upon receipt:** [ ] Feed  [ ] Ambient  [ ] $^\circ$C  

---

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Special Handling:
- TAT- Indicate Date Needed: ___ _
- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rushes.
- Samples disposed of after 30 days unless otherwise instructed.

Project No.: 392485, FT, FK
Site Name: AOC-I
Location: Vieques State: PR
Sampler(s): Kenji Butler / Chris Reed

1=Na2S2O3  2=HCl  3=H2SO4  4=HNO3  5=NaOH  6=Ascorbic Acid  7=CH3OH
8=NaHSO4  9=  10=  

DW=Drinking Water GW=Groundwater WW=Wastewater
O=Oil SW=Surface Water SO=Soil SL=Sludge A=Air
X1= X2= X3=  

G=Grab C=Composite

<table>
<thead>
<tr>
<th>Lab Id</th>
<th>Sample Id</th>
<th>Date</th>
<th>Time:</th>
<th>Type</th>
<th>Matrix</th>
<th># of VOA Vials</th>
<th># of Amber Glass</th>
<th># of Clear Glass</th>
<th># of Plastic</th>
</tr>
</thead>
<tbody>
<tr>
<td>13</td>
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<td>VvAI-NW07P-1110</td>
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</table>

State specific reporting standards:
- 0.45 micron filter under Fell.
- 0.15 micron filter under Fell.

175 Metro Center Boulevard • Warwick, RI 02886-1755 • 401-732-3400 • Fax 401-732-3499 • www.mitkem.com
Analysis Groups v. Analytical Methods for Navy CLEAN 1000-CTO-0083 Vieques AOC I First Post-Injection Event

For this sample collection effort, the field team will mark the chain-of-custody for each sample to be analyzed for one or more of the following analysis groups: List I VOC (HCl), List I VOC (unpres), List I VOC (AA), List I SVOC, FMETAL, and/or WCHEM. These analysis groups correspond to the following analytical methods:

- **List I VOC (HCl):** VOCs via SW-846 8260C (TCL from Worksheet 15-6 List I). LOQ = 5ug/L for all compounds. Preserved with HCl (holding time = 14 days). Note that this will likely be marked "hold for analysis" and we do not intend to analyze these samples at this time.

- **List I VOC (unpres):** VOCs via SW-846 8260C (TCL from Worksheet 15-6 List I). LOQ = 5ug/L for all compounds. Unpreserved (holding time = 7 days).

- **List I VOC (AA):** VOCs via SW-846 8260C (TCL from Worksheet 15-6 List I). LOQ = 5ug/L for all compounds. Preserved with 4:1 molar (AA: persulfate) ascorbic acid (holding time = 7 days).

- **List I SVOC:** SVOCs via SW-846 8270D (TCL from Worksheet 15-7 List I). LOQ = 1ug/L for Naphthalene and 2-Methylnaphthalene and LOQ = 5ug/L for bis(2-ethylhexyl)phthalate.

- **FMETAL:** Field-Filtered Iron and Manganese via SW-846 6010B.

- **WCHEM:** Sulfate and Nitrate via EPA 300.0

Total Organic Carbon (TOC) via SM5310B Quad

Note that the acronym "H" refers to "hold for analysis" and that we do not intend to analyze these samples at this time.

Please ensure that this memo is appended to each chain-of-custody record.
Edward Lawler [Mitkem]

From: Michael.Zamboni@CH2M.com
Sent: Friday, November 05, 2010 2:48 PM
To: Edward Lawler [Mitkem]
Cc: Michael.Zamboni@CH2M.com; Victoria.Brynildsen@CH2M.com; Stephen.Brand@CH2M.com; Brett.Doerr@CH2M.com
Subject: RE: Vieques, final COC and Login and...

...and please pull those samples off of hold! We think the HCl v. AA v. unpres comparison will be useful.

<table>
<thead>
<tr>
<th>COC Sample ID</th>
<th>Preservation</th>
<th>Lab Samp ID</th>
<th>Action</th>
<th>Rename (Client Sample ID) to:</th>
<th>New ID?</th>
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</thead>
<tbody>
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<td>J2254-15A</td>
<td>Analyze</td>
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<td>✓</td>
</tr>
</tbody>
</table>

Thanks for your help, Ed. Please let me know if that table isn’t legible for you and I’ll resend it in Excel. Have a great weekend!

Thanks,
Mike Z.

From: Edward Lawler [Mitkem] [mailto:elawler@mitkem.com]
Sent: Friday, November 05, 2010 1:50 PM
To: Zamboni, Michael/WDC; Brynildsen, Victoria/VBO
Subject: Vieques, final COC and Login and...

Hi Mike, Vickie——

Attached are the final COCs and Logins for all the samples received from the Vieques project.

The other file is what appears to be someone’s To-Do list, which was written on the back side of one of the Technical Memorandum pages. I assume all these items have been accomplished, but just in case...... here it is again.

Have a great weekend!

--Ed

Edward A. Lawler
Deputy Director for Quality Services, Mitkem Laboratories
a Division of Spectrum Analytical inc. featuring Hanibal Technology
401-732-3400 x315 401-732-3499 (fax)

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# MITKEM LABORATORIES
## Sample Condition Form

<table>
<thead>
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<th>Received By:</th>
<th>Reviewed By: SK</th>
<th>Date: 11/3</th>
<th>Mitkem Work Order #: 1223</th>
<th>Client: CH2M</th>
<th>VOA Matrix Key:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Client Project: UPG03</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1) Cooler Sealed</td>
<td>Yes</td>
<td>J2284 01</td>
<td>L2</td>
<td>01</td>
<td>H2U</td>
</tr>
<tr>
<td>2) Custody Seal(s)</td>
<td>Present</td>
<td>01</td>
<td>02</td>
<td>AA</td>
<td></td>
</tr>
<tr>
<td>Coolers / Bottles</td>
<td>Intact</td>
<td>01</td>
<td>02</td>
<td>AA</td>
<td></td>
</tr>
<tr>
<td>Intact / Broken</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3) Custody Seal Number(s)</td>
<td>tape</td>
<td>J2284</td>
<td>07</td>
<td>Y</td>
<td></td>
</tr>
<tr>
<td>4) Chain-of-Custody</td>
<td>Present</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5) Cooler Temperature</td>
<td></td>
<td></td>
<td>3°</td>
<td>2°</td>
<td></td>
</tr>
<tr>
<td>IR Temp Gun ID</td>
<td></td>
<td></td>
<td>M</td>
<td>M</td>
<td></td>
</tr>
<tr>
<td>Coolant Condition</td>
<td></td>
<td></td>
<td>Ice</td>
<td>Ice</td>
<td></td>
</tr>
<tr>
<td>6) Airbill(s)</td>
<td>Present</td>
<td>8627-2205-3289</td>
<td>8627-2205-3302</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Airbill Number(s)</td>
<td></td>
<td></td>
<td>8627-2205-3289</td>
<td>8627-2205-3302</td>
<td></td>
</tr>
<tr>
<td>7) Samples Bottles</td>
<td>Intact</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Broken / Leaking</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8) Date Received</td>
<td>11/3</td>
<td>01</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9) Time Received</td>
<td>09:00</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Preservative Name/Lot No.:</td>
<td>AA = Acidic</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Preservation (pH)</th>
<th>VOA Matrix</th>
<th>Soil Headspace or Air Bubble ≥ 1/4&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>HNO3</td>
<td>H2SO4</td>
<td>HCl</td>
</tr>
</tbody>
</table>

*See Sample Condition Notification/Corrective Action Form yes / no*

*Form ID: QAF.0006*

*Y:\Controlled Forms\QAF.0006 sample condition form*
<table>
<thead>
<tr>
<th>Client Project:</th>
<th>Client: CH2M Hill</th>
<th>Soil Headspace or Air Bubble ≥ 1/4&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Cooler Sealed</td>
<td>Present: Y / No: N</td>
<td>Preservation (pH)</td>
</tr>
<tr>
<td>2) Custody Seal(s)</td>
<td>Present: Y / Absent: N</td>
<td>HNO₃</td>
</tr>
<tr>
<td>3) Custody Seal Number(s)</td>
<td>Intact: Y / Broken: N</td>
<td>AA</td>
</tr>
<tr>
<td>4) Chain-of-Custody</td>
<td>Present: Y / Absent: N</td>
<td>AA</td>
</tr>
<tr>
<td>5) Cooler Temperature</td>
<td>3.0°C / 4.0°C</td>
<td>AA</td>
</tr>
<tr>
<td>IR Temp Gun ID</td>
<td>Ice / Ice = OK</td>
<td>AA</td>
</tr>
<tr>
<td>6) Airbill(s)</td>
<td>Present: Y / Absent: N</td>
<td>AA</td>
</tr>
<tr>
<td>7) Samples Bottles</td>
<td>Intact: Y / Broken / Leaking: N</td>
<td>AA</td>
</tr>
<tr>
<td>8) Date Received</td>
<td>11/4/12</td>
<td>AA</td>
</tr>
<tr>
<td>9) Time Received</td>
<td>08:57</td>
<td>AA</td>
</tr>
</tbody>
</table>

Preservative Name/Lot No.:  

VOA Matrix Key:  

US = Unpreserved Soil  
UA = Unpreserved Aqueous  
A = Air  
H = HCl  
M = MeOH  
E = Encore  
N = NaHSO₄  
F = Freeze  

See Sample Condition Notification/Corrective Action Form yes / no  
Form ID: QAF.0006  
Rad OK yes / no  

Y:\Controlled Forms\QAF.0006 sample condition form
I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: J2254  LAB: Mittem

SITE NAME: Vieques C70-83

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent? 

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, and/or cover letter signed release present? 

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? 

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, and/or Chain of Custodies from the field samplers present for all samples sign release present? 

ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)? 

1.3 Sample Conditions/Problems
1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

**ACTION:** If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

**ACTION:** If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

**ACTION:** If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects non "UJ".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

The maximum holding time for aqueous samples is 14 days.

The maximum holding time for soils non aqueous samples is 14 days.

**NOTE:** If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with HCL acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. For non-aqueous samples for volatile components that are frozen (less than 7°C) or are properly cooled (4°C ± 2°C) and perserved with NaHSO₄, the maximum holding time is 14 days from sample collection. If
uncertain about preservation, contact the laboratory /sampling team to determine whether or not samples were preserved.

ACTION: Qualify sample results according to Table 1:

Table 1. Holding Time Actions for Trace Volatile Analysis

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Preserved</th>
<th>Criteria</th>
<th>Detected Associated Compounds</th>
<th>Non-Detected Associated Compounds</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aqueous</td>
<td>No</td>
<td>≤ 7 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>No</td>
<td>&gt; 7 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤ 14 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>&gt; 14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td>Non Aqueous</td>
<td>No</td>
<td>≤ 14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤ 14 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes/No</td>
<td>&gt; 14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
</tbody>
</table>

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Water

b. Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:

a. Water

b. Soil

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data
Assessments and contact the laboratory/project officer/appropriate official for an explanation/resubmittal, make any necessary corrections and document effect in the Data Assessment.

3.3 Were the surrogate recovery limits followed per Table 2. If Table 2 criteria were not followed, the laboratory may use in-house performance criteria (per SW-846, Method 8000C, section 9.7). Other compounds may be used as surrogates, depending upon the analysis requirements.

<table>
<thead>
<tr>
<th>DMC</th>
<th>Recovery Limits (%) Water</th>
<th>Recovery Limits Soil/Sediment</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Bromofluorobenzene</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dibromofluoromethane</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Toluene-d₈</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dichloroethane-d₄</td>
<td>80-120</td>
<td>70-130</td>
</tr>
</tbody>
</table>

Note: Use above table if laboratory did not provide in house recovery criteria.

Note: Other compounds may be used as surrogates depending upon the analysis requirements.

3.4 Were outliers marked correctly with an asterisk?

ACTION: Circle all outliers with a red pencil.

3.5 Were one or more volatile surrogate recoveries out of specification for any sample or method blank. Table 2.

If yes, were samples reanalyzed?

Were method blanks reanalyzed?
USEPA Region II
SW846 Method 8260B VOA

Date: August 2008
SOP: HW-24, Rev. 2
YES NO N/A

ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects, but qualify positive results as estimated "J".

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. If one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose the blank problem to be an isolated occurrence.

3.6 Are there any transcription/calculation errors between raw data and reported data?

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Sample (Form III/Equivalent)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG.
Note: LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation/resubmittals. Make note in the data assessment.

4.2 Were the Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

A. Water

B. Soil

C. Med Soil

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

4.3 Have in house LCS recovery limits been developed (Method 8000C, Sect 9.7).

4.4 If in house limits are not developed, are LCS acceptance recovery limits between 70 - 130% (Method 8000C Sect 9.5)?

4.5 Were one or more of the volatile LCS recoveries outside the in house laboratory recovery criteria for spiked analytes? If in house limits are not present use 70 - 130% recovery limits.
Table 3. LCS Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Detected Spiked Compounds</th>
<th>Action</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td></td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td></td>
<td>UJ</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td>No Qualifications</td>
<td></td>
</tr>
</tbody>
</table>

5.0 Matrix Spikes (Form III or equivalent)

5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix? Yes

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, Method 8260B, Sect 8.4.2).

5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III? Yes

ACTION: If any data are missing take action as specified in section 3.2 above.

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples)
of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month [page 8000C, section 9.5.]

a. Water
b. Waste
c. Soil/Solid

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The concentration of the LCS should be determined as described SW-Method 8000C Section 9.5.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

5.4 Have in house MS recovery limits been developed (Method 8000C, Sect 9.7) for each matrix.

5.5 Were one or more of the volatile MS/MSD recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000C, Sect. 9.5.4.

ACTION: Circle all outliers with a red pencil.

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.
NOTE: No qualification of data is necessary on MS and MSD data alone. However, using informed professional judgement, the data reviewer may use MS and MSD results in conjunction with other QC criteria to determine the need for some qualification.

Note: The data reviewer should first try to determine to what extent the results of the MS and MSD affect the associated data. This determination should be made with regard to the MS and MSD sample itself, as well as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determine that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affect all associated samples, and the reviewer must use professional judgement to qualify the data from all associated samples.

Note: The reviewer must use professional judgement to determine the need for qualification of non-spiked compounds.

ACTION: Follow criteria in Table 4 when professional judgement deems qualification of sample.

Table 4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Detected Spiked Compounds</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td>UJ</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td>No Qualifications</td>
</tr>
</tbody>
</table>
6.0 Blank (CLP Form IV Equivalent)

6.1 Is the Method Blank Summary form present?  

6.2 Frequency of Analysis: Has a method blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch?  

6.3 Has a method blank been analyzed for each GC/MS system used?  

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable for volatile organic compounds?

7.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.
7.2 Do any field/rinse blanks have positive volatile organic compound results? 

**YES NO N/A**

**ACTION:** Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

**NOTE:** All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

**ACTION:** Follow the directions in Table 5 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

```
VWA1 - TB01-110410 MoG
EB01 - 1110A MoG
EB01 - 110410 MoG
TB01 - 110310 MoG
EB01 - 110310C MoG
EB01 - 110316 MoG
TB01 - 110216 MoG
EB01 - 110210A MoG
EB01 - 110210 MoG
```
## Table 5. Volatile Organic Analysis Blank Contamination Criteria

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td>No qualification</td>
<td></td>
</tr>
<tr>
<td>&lt; CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>&gt; CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report the concentration for the sample with a U, or qualify the data as unusable R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and ≥ blank contamination</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>= CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>Gross contamination</td>
<td>Detects</td>
<td>Qualify results as unusable R</td>
<td></td>
</tr>
</tbody>
</table>

* 2x the CRQL for methylene chloride, 2-butanone, and acetone

** Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 µg/L.

NOTE: If gross blank contamination exists (e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected volatile organic target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
7.3 Are there field/rinse/equipment blanks associated with every sample?

YES NO N/A

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.

YES NO N/A

NOTE: For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?

YES NO N/A

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?

YES NO N/A

9.3 Has an instrument performance check solution (BFB)-18VOA-
been analyzed for every twelve hours of sample analysis per instrument? (see Table 4, SW-846, page 82608-36)

YES NO N/A

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS GC/MS tuning data are available.

ACTION: If the laboratory/project officer cannot provide missing data, reject (“R”) all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable, “R”.

9.4 Have the ion abundances been normalized to m/z 95? 

9.5 Have the ion abundance criteria been met for each instrument used?

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.)

9.7 Have the appropriate number of significant figures (two) been reported?

ACTION: If large errors exist, take action as specified in section 3.2.

9.8 Are the spectra of the mass calibration compounds acceptable?

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.
10.0 Target Analytes (CLP Form I Equivalent)

10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

   a. Samples and/or fractions as appropriate
   b. Matrix spikes and matrix spike duplicates
   c. Blanks
   d. Laboratory Control Samples

10.2 Are the reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

   a. Samples and/or fractions as appropriate
   b. Matrix spikes and matrix spike duplicates (Mass spectra not required)
   c. Blanks
   d. Laboratory Control Samples

ACTION: If any data are missing, take action specified in 3.2 above.

10.3 Is chromatographic performance acceptable with respect to:

   Baseline stability?
Resolution?  
Peak shape?  
Full-scale graph (attenuation)?  
Other: __________________

**ACTION:** Use professional judgement to determine the acceptability of the data.

**10.4** Are the lab-generated standard mass spectra of identified volatile compounds present for each sample?  

**ACTION:** If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, contact the lab for missing spectra.

**10.5** Is the RRT of each reportee compound within 0.06 RRT units of the standard RRT in the continuing calibration?  

**10.6** Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?  

**10.7** Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?  

**ACTION:** Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be
positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

11.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

11.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier?

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

11.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate

b. Blanks

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "IN" qualifier only to analytes identified by a CAS#.

NOTE: If TICs are present in the associated blanks take action as specified in section 3.2 above.
11.3 Are any priority pollutants listed as TIC compounds (i.e., an EBN compound listed as a VOA TIC)?

**ACTION:**
1. Flag with "R" any target compound listed as a TIC.
2. Make sure all rejected compounds are properly reported if they are target compounds.

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

11.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

**ACTION:** Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO₂(M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found?

**NOTE:** Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be
reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL's adjusted to reflect sample dilutions and, for soils, sample moisture?

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC accedence dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration?

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above.
14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction?  

**YES**  **NO**  **N/A**

**ACTION:** If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

**ACTION:** If the percent relative standard deviation (% RSD) is > 20%, qualify positive results for that analyte "J". When % RSD > 90%, qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.2 Are all average RRFs > 0.050?

**YES**  **NO**  **N/A**

**NOTE:** (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

<table>
<thead>
<tr>
<th>Compound</th>
<th>RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloromethane</td>
<td>0.10</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>0.10</td>
</tr>
<tr>
<td>Bromoform</td>
<td>0.10</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>0.30</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>0.30</td>
</tr>
</tbody>
</table>

**ACTION:** Circle all outliers with red pencil.

**ACTION:** For any target analyte with average RRF < 0.05, or for the requirements for the 5 compounds in 14.2 above, qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration?

**YES**  **NO**  **N/A**

**NOTE:** (Method Requirement) For the following CCC compounds, the %RSD values must be ≤ 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment.
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 20.0%, or > 30% for the 6 compounds in 14.3 above, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF? [X] __ __

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.) [X] __ __

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

15.0 GC/MS Calibration Verification (CLP Form VII Equivalent)
15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest?  

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

15.3 Was the % D determined from the calibration verification determined using RRF or CF?

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.

15.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2).

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be ≤ 20.0%. If %D values reported are > 20.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride
ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

15.5 Do any volatile compounds have a RRF < 0.05? [ ] [ ] [ ]

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

<table>
<thead>
<tr>
<th>Compound</th>
<th>RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloromethane</td>
<td>0.10</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>0.10</td>
</tr>
<tr>
<td>Bromoform</td>
<td>0.10</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>0.30</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>0.30</td>
</tr>
</tbody>
</table>

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, or < the requirements for the 5 compounds is section 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard (IS) areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to +100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)? [ ] [ ]
ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area Lower Limit</th>
<th>Area Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when the associated IS are counts area > + 100%.

3. If the IS area is below the lower limit (< - 50%), qualify all associated non-detects (U-values) "J".

4. If extremely low area counts are reported (< - 25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.
17.0 Field Duplicates

17.1 Were any field duplicates submitted for volatile analysis?

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.
#DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! #DIV/0! 

**Sample ID:** VWAI-MW07-1110  
**Duplicate Sample ID:** VWAI-MW07-1110

Water: RPD>75%  
Soil: RPD>100%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>benzene</td>
<td>9.5</td>
<td>10</td>
<td>5</td>
</tr>
</tbody>
</table>

**COMMENTS:**  
No qualifications

* one of the results below the CRQL
Sample ID: VWAI-MW07-1110A
Duplicate Sample ID: VWAI-MW07P-1110A

Water: RPD > 75%
Soil: RPD > 100%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>benzene</td>
<td>9.5</td>
<td>9.5</td>
<td>#DIV/O!</td>
</tr>
</tbody>
</table>

COMMENTS: No qualifications

* one of the results below the CRQL
REPORT NARRATIVE

Mittem Laboratories, a Division of Spectrum Analytical, Inc.
Client: CH2M-Hill, Inc.
Project: CTO-0083 Vieques AOC E and I
Laboratory Workorder / SDG #: J2254
SW846 8260C

I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

Samples for volatile organics analyses were received with multiple preservations, including ice only, ice + ascorbic acid, and ice + hydrochloric acid.

Vials containing hydrochloric acid preservative were originally identified as "HOLD", but subsequently requested for analysis. Identifications for these samples had the letter "H" appended. Please note that the instructions to analyze these sample aliquots and append the letter "H" were not listed on the original chain of custody forms.

II. HOLDING TIMES

All samples were analyzed within the holding times specified in the method, shortened by the analytical specification and instructions for this program.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 8260C. A select list of volatile compounds were analyzed-for and reported.

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW5030B_PR(METHOD).

V. INSTRUMENTATION

The following instrumentation was used
- Instrument Code: V6
- Instrument Type: GCMS-VOA
- Description: HP6890 / HP5973
- Manufacturer: Hewlett-Packard
- Model: 6890 / 5973
- GC Column used: 30 m X 0.25 mm ID [1.40 um thickness] DB-624 capillary column.

VI. ANALYSIS
A. Calibration:
Calibrations met the method/SOP acceptance criteria.

B. Blanks:
All method blanks were within the acceptance criteria.

C. Surrogates:
Surrogate standard percent recoveries were within the QC limits.

D. Spikes:
1. Laboratory Control Spikes (LCS):
   Percent recoveries for lab control samples were within the QC limits.
2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):
   Duplicate matrix spikes were performed on samples: VWAI-MW02-1110 (J2254-08FMS/MSD) VWAI-MW02-1110A (J2254-09AMS/MSD).
   Percent recoveries were within the QC limits.

E. Internal Standards:
Internal standard peak areas were within the QC limits.

F. Dilutions:
No sample in this SDG required analysis at dilution.

G. Samples:
No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Mitkem, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: [Signature]
Date: 12/3/03
The concentration of this analyte exceeds the calibration range of the instrument.

Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: J2254  LAB: MitKem
SITE NAME: Vieques CT0-83

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format?

ACTION: If not, note the effect on review of the data in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present?

2.2 Are case number and SDG number(s) contained in the narrative or cover letter?
II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples?

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all non-detects data are qualified as unusable (R), and detects are flagged "J".

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any semivolatile technical holding times, determined from date of collection to date of extraction, been exceeded?

Continuous extraction of water samples for semivolatile analysis must be started within 7 days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within...
40 days of the date of extraction.

Table of Holding Time Violations

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Matrix</th>
<th>Date Sampled</th>
<th>Date Received</th>
<th>Date Extracted</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

**ACTION:** If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("U") and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).
3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water [ ] [ ] [ ]

b. Low/Med Soil [ ] [ ] [ ]

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water [ ] [ ] [ ]

b. Low/Med Soil [ ] [ ] [ ]

ACTION: If CLP deliverables are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk? [ ] [ ] [ ]

ACTION: Circle all outliers in red.

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank (Reviewer should use lab in house recovery limits. Use surrogate recovery limits from USEPA National Functional Guidelines January 2005 page 130, if in house limits are not available. See Method 80008-43 or 80000C-24).

[ ] [ ]

Note: Examine lab in house limits for reasonableness.

If yes, were samples re-analyzed? [ ] [ ] [ ]
Were method blanks re-analyzed?

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".
2. Non-detects for that fraction should be qualified as unusable (R).

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II?

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document
4.0 Matrix Spikes (Form III/Equivalent)

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate/or duplicate unspiked Sample recoveries been listed on the Recovery Form (Form III)?

NOTE: Method 3500B/page 4 states the spiking compounds:

**Base/neutrals**
- 1,2,4-Trichlorobenzene
- Acenaphthene
- 2,4-Dinitrotoluene
- Pyrene
- N-Nitroso-di-n-propylamine
- 1,4-Dichlorobenzene

**Acids**
- Pentachlorophenol
- Phenol
- 2-Chlorophenol
- 4-Chloro-3-methylphenol
- 4-Nitrophenol

**Note:** Some projects may require the spiking of specific compounds of interest.

Note: See Method 8270D-sec 8.4.2 for deciding on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate. If samples are expected to contain target analytes, then laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratory should use a matrix spike and matrix spike duplicate pair.

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water

b. Low Solid

c. Med Solid
ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. It may be necessary to contact the lab to obtain the required data.

NOTE: If the data has not been reported on CLP equivalent form, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

4.3 Were matrix spikes performed at concentration equal to 100μg/L for acid compounds, and 200μg/l for base compounds (Method 3500B-4), or specified in project plan.

4.4 How many semivolatile spike recoveries are outside laboratory in house MS/MSD recovery limits (use recovery limits values in Method 8270D-43&44 Table 6 if in house values not available).

Water

0 out of 50

Solids

___ out of ___
4.5 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water
\[ \text{0 out of 2} \]

Solids
\[
\text{___ out of ___}
\]

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a Laboratory Control Sample (LCS) analyzed with each analytical batch?

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 Blanks (Form IV/Equivalent)

5.1 Is the Method Blank Summary (Form IV) present? \[ \checkmark \]

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch? \[ \checkmark \]

5.3 Has a method blank been analyzed either after
the calibration standard or at any other time during the analytical shift for each GC/MS system used?

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for the semivolatiles?

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

6.2 Do any field/rinse/ blanks have positive results for target analytes and/or TICs (if required, see section 10 below)?
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

VWAI - EB01 - 110210 moQ
EB01 - 110310 moQ
EB01 - 110410 moQ
## Blank Action for Semivolatile Analyses

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td>No qualification required</td>
<td></td>
</tr>
<tr>
<td>&lt; CRQL *</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
<td></td>
</tr>
<tr>
<td>= CRQL *</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
<td></td>
</tr>
<tr>
<td>&gt; CRQL *</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report concentration of sample with a U</td>
<td></td>
<td></td>
</tr>
<tr>
<td>≥ CRQL and ≥ blank contamination</td>
<td>No qualification required</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**NOTE:** Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

**NOTE:** If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required.

### 6.3 Are there field/rinse/equipment blanks associated with every sample?

**ACTION:** For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

### 6.4 Was a instrument blank analyzed after each sample/dilution which contained a target compound
that exceeded the initial calibration range.

6.5 Does the instrument blank have positive results for target analytes and/or TICs?

Note: Use professional judgement to determine if carryover occurred and qualify analytes accordingly.

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270D? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column.

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (Form V/Equivalent)

8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)?

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5
8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? Yes __ No __ N/A __

8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument? Yes __ No __ N/A __

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

<table>
<thead>
<tr>
<th>DATE</th>
<th>TIME</th>
<th>INSTRUMENT</th>
<th>SAMPLE NUMBERS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tr>
</tbody>
</table>

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

8.4 Have the ion abundances been normalized to m/z 198? Yes __ No __ N/A __

8.5 Have the ion abundance criteria been met for each instrument used? Yes __ No __ N/A __

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).
ACTION: If ion abundance criteria are not met, take action specified in section 3.2

8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) YES NO N/A

8.7 Have the appropriate number of significant figures (two) been reported? YES NO N/A

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

8.8 Are the spectra of the mass calibration compound acceptable? YES NO N/A

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

9.0 Target Analytes

9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate YES NO N/A

b. Matrix spikes and matrix spike duplicates YES NO N/A

c. Blanks YES NO N/A

9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270D-14)? YES NO N/A
ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

a. Samples and/or fractions as appropriate

b. Matrix spikes and matrix spike duplicates (Mass spectra not required)

c. Blanks

ACTION: If any data are missing, take action specified in 3.2 above.

9.4 Are the response factors shown in the Quant Report?

9.5 Is chromatographic performance acceptable with respect to:

Baseline stability?

Resolution?

Peak shape?

Full-scale graph (attenuation)?

Other: ____________________________

ACTION: Use professional judgement to determine the acceptability of the data.

9.6 Are the lab-generated standard mass spectra of identified semivolatile compounds present for
each sample?

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

9.8 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

9.9 Do the relative intensities of the characteristic ions in the sample agree within ±30% of the corresponding relative intensities in the reference spectrum?

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.7, 9.8, and 9.9.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.
10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form Is, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?

NOTE: Review sampling reports to determine if the lab was required to identify non target analytes (refer to section 7.6.2, page 8270D-21).

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate

b. Blanks

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)?

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the
sample mass spectrum?

10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R."

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and, for soils, sample moisture?
ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration?

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI/Equivalent)

13.1 Is the Initial Calibration Form (Form VI/Equivalent) present and complete for the semivolatile fraction?

ACTION: If any calibration forms or standard row data are missing, take action specified in 3.2 above.

13.2 Are all base neutral or acid RRFs > 0.050?
Check the **average RRFs** of the four System Performance Check Compounds (SPCCs):
N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. These compounds must have **average RRFs** greater than or equal to 0.05 before running samples and should not show any peak tailing.

**ACTION:** Circle all outliers in red.

**ACTION:** For any target analyte with **average RRF <0.05**

1. "R" all non-detects;
2. "J" all positive results.

13.3 Are response factors for base neutral or acid target analytes stable over the concentration range of the calibration (% Relative standard deviation [%RSD] < 20.0%)?

**NOTE:** The % RSD for each individual Calibration Check Compound (CCC, Method 8270D-40 see Table 4) must be less than 30% before analysis can begin. If greater 30%, the lab must clean and recalibrate the instrument.

**CALIBRATION CHECK COMPOUNDS**

<table>
<thead>
<tr>
<th>Base/Neutral Fraction</th>
<th>Acid Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acenaphthene</td>
<td>4-Chloro-3-methylphenol</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>2,4-Dichlorophenol</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>2-Nitrophenol</td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>Phenol</td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>2,4,6-Trichlorophenol</td>
</tr>
</tbody>
</table>

- 25 - 116
Benzo(a)pyrene

**ACTION:** If the %RSD for any CCC >30% and no corrective action taken, then "J" qualify all positive hits and "UJ" qualify all non-detects.

**ACTION:** Circle all outliers in red.

**ACTION:** If the % RSD is ≥ 20.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detect results for that analyte "R," unusable. Alternatively, the lab should calculate first or second order regression fit of the calibration curve and select the fit which introduces the least amount of error.

**NOTE:** Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

13.4 Did the laboratory calculate the calibration curve by the least squares regression fit?  

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.)

**ACTION:** Circle Errors in red.

**ACTION:** If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.5 Do the target compounds for this SDG include Pesticides?
13.6 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%?  

**YES** [ ]  **NO** [ ]  **N/A** [ ]

**ACTION:**  If DDT percent breakdown exceeds 20%:

i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

14.0 GC/MS Calibration Verification (Form VII/Equivalent)

14.1 Are the Calibration Verification Forms (Form VII) present and complete for all compounds of interest?  

[ ]  [ ]  [ ]

14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?  

[ ]  [ ]

**ACTION:**  List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

__________________________  
__________________________  
__________________________

**ACTION:**  If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis,
call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

14.3 Do any of the SPCCs have an RRF < 0.05? __ NO __ N/A

If YES, make a note in data assessment if the lab did not take corrective action specified in section 7.4.4, page 8270D-18.

14.4 Do any of the CCCs have a %D between the initial and continuing RRF which exceeds 20.0%?

ACTION: If yes, make a note in data assessment.

14.5 Do any semivolatile compounds have a % Difference (% D) between the initial and continuing RRF which exceeds 20.0%? __ NO __ N/A

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is above 90%, qualify all non-detects for that analyte as "R", unusable.

14.6 Do any semivolatile compounds have a RRF < 0.05? __ NO __ N/A

ACTION: Circle all outliers in red.

ACTION: If RRF < 0.05, qualify as unusable ("R") associated non-detects and "J" associated positive values.

14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more). __ NO __ N/A
ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.
ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area</th>
<th>LowerLimit</th>
<th>Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
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<td></td>
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</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

Note: Check Table 5, 8270D-41 for associated analytes.

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.
iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Laboratory Control Samples (LCS)

16.1 Were any LCS samples run in order to verify analytes which failed criteria for spike recovery?

16.2 Did the lab spike LCS sample spiked with the same analytes and the same concentrations as the matrix spike?

16.3 Were the mean and standard deviation of all analytes within the QC acceptance ranges as shown in Table 6, 8270D-43?

ACTION: If the recovery of any analyte falls out of the designated range, the analytical results for that compound is suspect and should be qualified "J" in the unspiked samples.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for semivolatile analysis?
**FIELD DUPLICATE SAMPLE SUMMARY**

**Sample ID:** VWAI-MW07-1110  
**Duplicate Sample ID:** VWAI-MW07-1110D  

Water: RPD > 50%  
Soil: RPD > 75%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>napthalene</td>
<td>7.9</td>
<td>10</td>
<td>23</td>
</tr>
<tr>
<td>2-methylnapthalene</td>
<td>7.7</td>
<td>9.9</td>
<td>25</td>
</tr>
</tbody>
</table>

* one or both values below CRQL

**COMMENTS:** No qualifications required.
REPORT NARRATIVE
Mitkem Laboratories, a Division of Spectrum Analytical, Inc.

Client: CH2M-Hill, Inc.
Project: CTO-0083 Vieques AOC E and I
Laboratory Workorder / SDG #: J2254
SW846 8270D

I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:
All samples were prepared within the method-specified holding times.

B. Sample Analysis:
All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 8270D. A select list of semivolatile compounds were analyzed-for and reported.

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: BNA_W_PR(SEPF)

V. INSTRUMENTATION

The following instrumentation was used
Instrument Code: S3
Instrument Type: GCMS-SEMI
Description: HP6890 / HP5973
Manufacturer: Hewlett-Packard
Model: 6890 / 5973
GC Column used: 30 m X 0.25 mm ID [0.25 um thickness] Rxi-5sil MS capillary column.

VI. ANALYSIS

A. Calibration:
Calibrations met the method/SOP acceptance criteria.

B. Blanks:
All method blanks were within the acceptance criteria.

C. Surrogates:

Surrogate standard percent recoveries were within the QC limits.

D. Spikes:

1. Laboratory Control Spikes (LCS):

   Percent recoveries for lab control samples were within the QC limits.

2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):

   Duplicate matrix spikes were performed on sample: VWAI-MW02-1110 (J2254-08EMS/MSD)

   Percent recoveries were within the QC limits.

G. Internal Standards:

Internal standard peak areas were within the QC limits.

H. Dilutions:

No sample in this SDG required analysis at dilution.

H. Samples:

No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Mitkem, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: [Signature]

Date: 12/3/18
Site: Viquez AOC E & I
Case #:
SDG #: j2354
Samples: Soil

Note - Most CIP forms were not used (receipt forms & some documentation forms). Report forms were CIP format.
### SOP: HW-2 Revision 13 Appendix A.1 Sept. 2006

<table>
<thead>
<tr>
<th>A.1.1 Contract Compliance Screening Report</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present?</td>
<td></td>
<td></td>
<td>✔</td>
</tr>
<tr>
<td><strong>ACTION:</strong> If no, contact RSCC/PO.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.2 Record of Communication (from RSCC)</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>ACTION:</strong> If no, request from the RSCC.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.3 Sampling Trip Report</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present and complete?</td>
<td></td>
<td></td>
<td>✔</td>
</tr>
<tr>
<td><strong>ACTION:</strong> If no, contact RSCC/PO.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.4 Chain of Custody/Sample Traffic Report</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present?</td>
<td>✔</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Legible?</td>
<td>✔</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Signature of sample custodian present?</td>
<td>✔</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>ACTION:</strong> If no, contact RSCC/WAM/PO.</td>
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<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.5 Cover Page</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present?</td>
<td></td>
<td></td>
<td>✔</td>
</tr>
<tr>
<td>Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?</td>
<td>✔</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Do the sample identification numbers on the Cover Page agree with sample Identification numbers on:</td>
<td></td>
<td></td>
<td>✔</td>
</tr>
<tr>
<td>(a) Traffic Report Sheet?</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
(b) Form I's?

Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?

ACTION:
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 SDG Narrative, DC-1 & DC-2 Form

Is the SDG Narrative present?

Is Sample Log-In Sheet (Form DC-1) present and complete?

Is Complete SDG Inventory Sheet (Form DC-2) present and complete?

ACTION:
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

A.1.7 Form I to XV

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name?

Laboratory Code?

RAS/Non-RAS Case No.?

SDG No.?
Standard Operating Procedure
USEPA Region 2
Evaluation of Metals Data for the Contract Laboratory Program
Data Assessment and Contract Compliance Review

SOP: HW-2  Revision 13  Appendix A.1  Sept. 2006

Contract No.?

ACTION:
If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

A.1.7.2 After comparing values on Form:s I-IX against the raw data, do any computation/transcription errors exceed 10% of the reported values on the Forms for:

(a) all analytes analyzed by ICP-AES? ___ [ ] __
(b) all analytes analyzed by ICP-MS? ___ [ ] __
(c) Mercury? ___ [ ] __
(d) Cyanide? ___ [ ] __

ACTION:
If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 Raw Data
Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 Digestion/Distillation Log

Digestion Log for ICP-AES (Form XII) present? [ ] __ __

Digestion Log for ICP-MS (Form XII) present? ___ [ ] __

Digestion Log for mercury (Form XII) present? ___ [ ] __

Distillation Log for cyanide (Form XII) present? ___ [ ] __
Procedure 1.8.2

Is the analytical instrument real-time printouts present for:

- ICP-AES? [✓] 
- ICP-MS? [✓] 
- Mercury? [✓] 
- Cyanide? [✓]

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:

- Legible? [✓] 
- Properly labeled? [✓] 
- Are all field samples, QC samples and field QC samples present on:
  - Digestion/Distillation log? [✓]
  - Instrument Printouts? [✓]

**ACTION:**

If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.
A.1.9  **Technical Holding Times:** (Aqueous and soil samples)

(Examine sample traffic reports and digestion/distillation logs to
determine the holding time from the sample collection date to the sample
preparation date.)

A.1.9.1 Cyanide distillation (14 days) exceeded?  
Mercury analysis (28 days) exceeded? 
Other Metals analysis (180 days) exceeded?

**ACTION:**
If yes, reject (R) and red-line non-detects
and flag as estimated (J) results ≥ MDL even
if sample(s) was preserved properly.

**NOTE:**
In addition to qualifying the data,
a list of all samples and analytes
which exceeded the holding times must
be prepared. Report for each sample
the number of days that were exceeded.
(Subtract the sample collection date
from the sample preparation date).
Attach this list to the data review
narrative.

A.1.9.2 Is pH of aqueous samples for:

<table>
<thead>
<tr>
<th>Metals Analysis</th>
<th>≤ 2?</th>
<th>[✓]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyanide Analysis</td>
<td>≥ 12?</td>
<td>[✓]</td>
</tr>
</tbody>
</table>

**ACTION:**
If no for any of the above, flag
non-detects as “R” and detects as “J”.

A.1.9.3 Is the cooler temperature ≤ 10°C?

[✓]

**ACTION:**
If cooler temperature is >10°C, flag
non-detects as “UJ” and detects as
“J”.

A.1.10  **Final Data Correctness - Form I**

A.1.10.1 Are Form I’s for all samples
A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

- Is the calculation error less than 10% of the correct result? [✓] 
- Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)? [✓] 
- Are results on Form I's reported by correct significant figures? [✓] 
- Are soil sample results on Form I's corrected for percent solids? [✓] 
- Are all "less than MDL" values reported by the CRLs and coded with "U"? [✓] 
- Are values less than the CRLs but greater than or equal to the MDLs flagged with "J"? [✓] 
- Are appropriate contractual quality control and Method qualifiers used? [✓]

**ACTION:**
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.
of the samples before and after
digestion given on the Form I's?

Was any sample result outside the
mercury/cyanide calibration range
or the ICP-AES/ICP-MS linear range
diluted and noted on the Form I?

**ACTION:**
If no for any of the above, note under
the Contract-Problem/Non-Compliance
Section of the Data Review Narrative.

**A.1.11 Initial Calibration**

**A.1.11.1** Is a record of at least 2 point
(A blank and a standard)calibration
present for ICP-AES analysis?

**ACTION:**
If incomplete or no initial calibration
was performed, reject (R) and red-line
the associated data (detects & non-detects).

Is one initial calibration standard
at the CRQL level for cyanide and
mercury?

**ACTION:**
If no, write in the Contract Problem/
Non-Compliance Section of the Data
Review Narrative.

**A.1.11.2** Is the curve correlation
coefficient ≥ 0.995 for:
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<table>
<thead>
<tr>
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</thead>
<tbody>
<tr>
<td><strong>Mercury Analysis?</strong></td>
<td><img src="%E2%9C%93" alt="Yes" /></td>
<td><img src="%E2%98%90" alt="No" /></td>
</tr>
<tr>
<td><strong>Cyanide Analysis?</strong></td>
<td><img src="%E2%9C%93" alt="Yes" /></td>
<td><img src="%E2%98%90" alt="No" /></td>
</tr>
<tr>
<td>ICP-AES (more than 2 point Calib.)?</td>
<td><img src="%E2%9C%93" alt="Yes" /></td>
<td><img src="%E2%98%90" alt="No" /></td>
</tr>
<tr>
<td>ICP-MS (more than 2 point calib.)?</td>
<td><img src="%E2%9C%93" alt="Yes" /></td>
<td><img src="%E2%98%90" alt="No" /></td>
</tr>
</tbody>
</table>

**ACTION:**

If no, qualify the associated sample results ≥ MDL as estimated "J" and non-detects as "UJ".

**NOTE:**

The correlation coefficient shall be calculated by the data validator using standard concentrations and the corresponding instrument response (e.g. absorbance, peak area, peak height, etc.).

### A.1.12

**Initial and Continuing Calibration Verification - Form IIIA**

- **A.1.12.1** Present and complete for every metal and cyanide?  
  
  ![Yes](✓) | ![No](☐) | ![N/A](☐)

- Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte?  
  
  ![Yes](✓) | ![No](☐) | ![N/A](☐)

**ACTION:**

If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.

- **A.1.12.2** Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent?  
  
  ![Yes](✓) | ![No](☐) | ![N/A](☐)

**ACTION:**

If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

- **A.1.12.3** Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples?  
  
  ![Yes](✓) | ![No](☐) | ![N/A](☐)
A.1.12.2 Circle on each Form IIA all percent recoveries that are outside the contract windows.

Are ICV/CCVs within control limits for:

- Metals - 90-110%R?
  - [ ]
  - [ ]
  - [ ]

- Hg - 80-120%R?
  - [ ]
  - [ ]
  - [ 

- Cyanide - 85-115%R?
  - [ ]
  - [ ]
  - [ ]

**ACTION:**
If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89%(65-79% for Hg; 70-84% for CN). Qualify only positive results (> MDL) as "J" if the ICV/CCV %R is between 111-125%(121-135% for Hg; 116-130% for CN). Reject (R) and red-line only detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detests) if the recovery is less than 75%(65% for Hg; 70% for CN).

**NOTE:**
For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

A.1.12.3 Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)?

- [ ]

**ACTION:**
If no, Qualify all cyanide results ≥ MDL as "J".

A.1.13 CRQL Standard Analysis - Form IIB

A.1.13.1 For each ICP-AES run, was a CRI
(CRQL or MDL when MDL > CRQL) standard analyzed?
(Note: CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)

For each ICP-MS run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis?

For each mercury run, was a CRQL standard analyzed?

For each cyanide run, was a CRQL standard analyzed?

**ACTION:**
If no for any of the above, write this deficiency in the Contract Problems/Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL) as J and non-detects UJ.

The affected ranges are:
ICP-AES Analysis - *True Value + CRQL
ICP-MS Analysis - *True Value + CRQL
Mercury Analysis - *True Value + CRQL
Cyanide Analysis - *True Value + CRQL

* True value of the CRQL Standard

**A.1.13.2** Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis?

**ACTION:**
If no, write in the Contract Problem/Non-Compliance Section of the "Data Review Narrative".

**A.1.13.3** Circle on each Form IIB all percent recoveries that are outside the acceptance windows.
Is the CRQL standard within control limits for:

Metals (ICP-AES/ICP-MS) - 70 - 130%?
[ ] [ ] [ ]

Mercury - 70 - 130%?
[ ] [ ] [ ]

Cyanide - 70 - 130%?
[ ] [ ] [ ]

**ACTION:**
If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag (J) only detects ≤2xCRQL if the recovery is between 131% and ≤180%. If the recovery is less than 950%, reject (R) and red-line non-detects and detects <2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detect ≥2xCRQL and flag (J) detects >2xCRQL but <ICV/CCV if the recovery is >180%.

**NOTE:**
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard.
2. Flag (J) or reject (R) only the final sample results on Form I's when Sample raw data are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

**A.1.14 Initial and Continuing Calibration Blanks - Form III**

**A.1.14.1** Present and complete for all the instruments used for the metals and cyanide analyses?
[ ] [ ] [ ]

Was an initial Calibration Blank analyzed after ICV?
[ ] [ ] [ ]

Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?
[ ] [ ] [ ]

Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by (J)?
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using MDLs from direct analysis (Preparation Method "NP1")?
(Check Form III against the raw data)

ACTION:
If no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".

A.1.14.2 Circle with red pencil on each Form III all Calib. Blank values that are:

> MDL but < CRQL
> CRQL

A.1.14.2.1 When MDL < CRQL, is any Calib. Blank value > MDL but < CRQL?

ACTION:
If yes, change sample results > MDL but < CRQL to the CRQL with a "U".
Do not qualify non-detects.

A.1.14.2.2 When MDL < CRQL, is any Calib. Blank value > CRQL?

ACTION:
If yes, reject (R) and red line the associated sample results > CRQL but < ICB/CCB Blank Result. Flag as "J" detects > ICB/CCB blank value but < 10xICB/CCB value. Change the sample results > MDL but < the CRQL to CRQL with a "U".

A.1.14.2.3 Is any Calibration Blank value below the negative CRQL?

ACTION:
If yes, flag (J) as estimated all associated sample results > CRQL but < 10xCRQL.

NOTE:
1. For ICB that does not meet the technical QC Criteria, apply the action to all samples.
<table>
<thead>
<tr>
<th>Analyte</th>
<th>Initial Calibration Blank (ug/L)</th>
<th>Continuing Calibration Blank (ug/L)</th>
<th>Preparation Blank</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C 1 C 2 C 3</td>
<td>C 1 C 2 C 3</td>
<td></td>
</tr>
<tr>
<td>Iron</td>
<td>31.0 U 31.0 U 44.3 B</td>
<td>31.0 U 44.3 B 31.000 U P</td>
<td></td>
</tr>
<tr>
<td>Manganese</td>
<td>10.0 U 10.0 U 10.0 U</td>
<td>10.0 U 10.000 U P</td>
<td></td>
</tr>
</tbody>
</table>

Flagged @ LOD 03V 02V
reported from the analytical run.
2. For CCBs that do not meet the technical QC criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

A.1.15 Preparation Blank - FORM III
NOTE: The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1 Was one Preparation Blank prepared with and analyzed for:

- Each Sample Delivery Group (SDG)? 
- Each batch of the SDG samples digested/distilled?
- Each matrix type?
- All instruments used for metals and cyanide analyses?

ACTION:
If no for any of the above, flag as estimated (J) all the associated positive data <10xMDL for which the Preparation Blank was not analyzed.

NOTE:
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated (J), but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

≥ MDL but ≤ CRQL, and
> CRQL

A.1.15.2.1 When MDL < CRQL, is any preparation blank value ≥ MDL but ≤ CRQL?

ACTION:
If yes, change sample result ≥ MDL
If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.

A.1.16 ICP-AES/ICP-MS Interference Check Sample (ICS) - Form IV

NOTE: Not required for CN, Hg, Al, Ca, Fe and Mg.

A.1.16.1 Present and complete? [✓] __ __

Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples? [✓] __ __

Was ICS analyzed at the beginning of the ICP-MS analytical run? [ ] __ ✓

ACTION:
If no, flag as estimated (J) all sample results.

A.1.16.2 ICP-AES Method

A.1.16.2.1 ICSA Solution:
For ICP-AES, are the ICSA “Found” analyte values within the control limits ± of CRQL of the true/established mean value? [✓] __ __

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (µg/L or µG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV? [ ] __ ✓

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results ≥ MDL.
for which the ICSA “Found” value is greater than (True value+CRQL). Do not qualify non-detects.
If the ICSA “Found” value is less than (True value-CRQL), flag non-detects as “UJ” and detects as “J”.

A.1.16.2.3 ICSAB Solution

For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value?

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSAB Solution on Form IV?

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79%, qualify sample results ≥ MDL as “J” and non-detects as “UJ”. Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.

A.1.16.3 ICP-MS Method

A.1.16.3.1 ICSA Solution:

For ICP-MS, are the ICSA “Found” analyte values within the control limits of ± CRQL of the true/established mean value?

ACTION:
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated only sample results ≥ MDL if the ICSA “Found” value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA “Found” value is less than (True value-CRQL), flag the associated sample detects as “J” and non-detects as “UJ”.

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A.1.16.3.3  ICSAB Solution

For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater?  

[ ]  [ ]   [V]

**ACTION:**

If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only detects (≥ MDL).

A.1.17  Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A

**Note:** Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only)

A.1.17.1  Was Matrix Spike analysis performed:

For each matrix type?  
[ ]  [ ]   [V]

For each SOG?  
[ ]  [ ]   [V]

On one of the SDG samples?  
[ ]  [ ]   [V]

For each concentration range (i.e., low, med., high)?  
[ ]  [ ]   [V]

For each analytical Method (ICP-AES, ICP-MS, Hg, CN) used?  
[ ]  [ ]   [V]

Was a spiked sample prepared and analyzed with the SDG samples?  
[ ]  [ ]   [V]

**ACTION:**

If no for any of the above, flag as estimated (J) all the positive data for which a spiked sample was not analyzed.

**NOTE:**

If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.
A.1.17.2 Was a field blank or PE sample used for the spiked sample analysis?

[ ] [ ] [X]

**ACTION:**
If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.

A.1.17.3 Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.

[ ] [ ] [X]

**NOTE:**
Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.

Are results outside the control limits (75-125%) flagged with Lab Qualifier "N" on Form I's and Form VA?

[ ] [ ] [X]

**ACTION:**
If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.

A.1.17.4 Aqueous

Are any spike recoveries:

(a) less than 30%?

[ ] [ ] [X]

(b) between 30-74%?

[ ] [ ] [ ]

(c) between 126-150%?

[ ] [ ] [ ]

(d) greater than 150%?

[ ] [ ] [ ]

**ACTION:**
If the matrix spike recovery is less than 30%, reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as “J” and non-detects,
A.1.17.5 Soil/Sediment

Are any spike recoveries:

(a) less than 10%? 
(b) between 10-74%? 
(c) between 126-200%? 
(d) greater than 200%?

ACTION:
If yes for any of the above, proceed as follows:

If the matrix spike recovery is less than 10%, reject (R) and red-line all associated data (detects & non-detects);
if between 10-74%, qualify all associated data ≥ MDL as “J” and non-detects as “UJ”;
if between 126-200%, flag (J) all associated data ≥ MDL as “J” if greater than 200%, reject (R) and red-line all associated data ≥ MDL.
(NOTE: Replace “N” with “J”, “R” as appropriate.)

A.1.18 Lab Duplicates - Form VI

A.1.18.1 Was the lab duplicate analysis performed:

For each SDG? 
For one of the SDG samples? 
For each matrix type? 
For each concentration range (low or med.)? 
For each analytical Method (ICP-AES/ICP-MS, Hg, CN) Used? 
Was a lab duplicate prepared and analyzed with the SDG samples?
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**ACTION:**
If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.

**NOTE:**
If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

A.1.18.2 Was a Field Blank or PE sample used for the Lab Duplicate analysis?

- [ ]

**ACTION:**
If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.

A.1.18.3 Circle on each Form VI all values that are:

- RPD > 20%, or
- Absolute Difference > CRQL

Are all values within control limits (RPD ≤ 20% or absolute difference ≤ ±CRQL)?

- [ ]

If no, are all results outside the control limits flagged with an "**" (Lab Qualifier) on Form VI and on all Form I's?

- [ ]

**ACTION:**
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

**NOTE:**
The laboratory is not required to report on Form VI the RPD when both values are non-detects.

A.1.18.4 Aqueous

A.1.18.4.1 When sample and duplicate values are both > 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

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**ACTION:**

If the RPD is > 20% but < 100%,

flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

*(NOTE: Replace "**" with "J" or "R" as appropriate.)*

---

A.1.18.4.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate values:

> ± CRQL?

> ± 2xCRQL?

---

**ACTION:**

If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 2xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but < 5xCRQL.

*(NOTE:)*

1. Replace "**" with "J", "UJ" or "R" as appropriate.
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

---

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?

is any RPD ≥ 120%?

---

**ACTION:**

If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample
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A.1.18.5.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

> ± 2 x CRQL?

> ± 4 x CRQL

ACTION:
If the absolute difference is > 2 x CRQL, flag all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 4xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but <5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.19

Field Duplicates

Aqueous Field Duplicates

A.1.19.1 Was an aqueous Field Duplicate pair collected and analyzed?

(Check Sampling Trip Report)

ACTION:
If yes, prepare a Form (Appendix A.4) for each aqueous Field Duplicate pair. Report the sample and Field Duplicate results on Appendix A.4 from their respective Form I's. Calculate and report RPD on Appendix A.4 when sample and its Field Duplicate values are both > 5xCRQL. Calculate and report the absolute difference on Appendix A.4 when at least one value (sample or duplicate) is <5xCRQL. Evaluate the aqueous Field Duplicate analysis in accordance with the
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</thead>
</table>

QC criteria stated in Sections A.1.19.2 and A.1.19.3.

**NOTE:**
1. Do not transfer "**" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

### A.1.19.2

Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

- **RPD ≥ 20%** or
- **Difference ≥ 1 CRQL**

When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL), is any RPD ≥ 20%?  

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**ACTION:**
If the RPD is > 20% but < 100%, flag (J) only the associated sample and its Field Duplicate results ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line only the associated sample and its Field Duplicate result ≥ CRQL.

### A.1.19.3

When the sample and/or duplicate value(s) < 5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

- **> 1 CRQL**  
- **> 2 x CRQL**

<p>| | | |</p>
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**ACTION:**
If the absolute difference is > CRQL, flag detects ≥ MDL but < 5xCRQL as "J" and non-detects as "XJ". If the difference is > 2xCRQL, reject (R) and red-line non-detects.
and results ≥ MDL but <5xCRQL of the sample and its Field Duplicate.

**Soil/Sediment Field Duplicates**

A.1.19.4 Was a soil field duplicate pair collected and analyzed? (Check Sampling Trip Report)

**ACTION:**
If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I’s. Calculate and report RPD when sample and its duplicate values are both greater than 5xCRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is <5xCRQL. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

**NOTE:**
1. Do not transfer "**" from Form I’s to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and apply the criteria to qualify the results.

A.1.19.5 Circle on each Appendix A.4 all values that have:

RPD ≥ 35%, or Difference > ± 2xCRQL
When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%? [___] __

is any RPD ≥ 120%? [___] __

**ACTION:**
If the RPD is ≥ 35% but < 120%,
flag only the associated sample
and its Field Duplicate results
≥ CRQL as "J". If the RPD is ≥ 120%,
reject (R) and red-line only the sample
and its Field Duplicate results ≥ CRQL.

A.1.19.6 When the sample and/or duplicate value(s)
<5xCRQL (substitute MDL for CRQL when MDL > CRQL),
is the absolute difference between sample
and Field Duplicate:

> ± 2 x CRQL?

> ± 4 x CRQL?

ACTION:
If the absolute difference is > 2xCRQL, flag
Sample and its Field Duplicate results ≥ MDL
but <5xCRQL as "J" and non-detects as "UJ".
If the difference is >4xCRQL, reject (R) and
red-line non-detects and detects ≥ MDL but
<5xCRQL of the sample and its Field Duplicate.

A.1.20 Laboratory Control Sample (LCS) - Form VII

A.1.20.1 Was one LCS prepared and analyzed for:

Each SDG? [✓] ___ ___

Each matrix type? [✓] ___ ___

Each batch samples digested/distilled? [✓] ___ ___
For each Method(ICP-AES, ICP-MS, Hg, CN)
used? ___ ___ ___

Was an LCS prepared and analyzed with
the samples? [✓] ___ ___

ACTION:
If no for any of the above, prepare
Telephone Record Log and contact
CLP FO or TOPO for submittal of the
LCS results. Flag (J) as estimated all
the data for which an LCS was not
analyzed.

NOTE:
If only one LCS was analyzed for...
A.1.20.2 **Aqueous LCS**

Circle on each Form VII the LCS percent recoveries outside control limits 80-120%.

**NOTE:**
1. Use digested ICV as LCS for aqueous mercury
2. Use distilled ICV as LCS for aqueous cyanide

Is any LCS recovery:
- Less than 50%? 
- Between 50% and 79%? 
- Between 121% and 150%? 
- Greater than 150%?

**ACTION:**
If the LCS recovery is less than 50%, reject (R) and red-line all associated sample data (detects & non-detects); for a recovery between 50-79%, flag detects as "J" all non-detects as "UJ". if the LCS recovery is between 121-150%, flag only detects as "J". if the recovery is greater than 150%, reject (R) and red-line all detects.

A.1.20.3 **Solid LCS**

If an analyte's MDL is equal to or greater than the true value of LCS, disregard the "Action" below for that analyte even though the LCS is out of control limits.

Is the LCS "Found" value greater than the Upper Control Limit reported on Form VII?

**ACTION:**
If yes, flag (J) all the associated detects ≥ MDL as estimated (J).

Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII?

ACTION:
If yes, flag detects as "J" and non-detects as "UJ".

A.1.21 ICP-AES/ICP-MS Serial Dilution - Form VIII
NOTE: Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 x MDL.

A.1.21.1 Was a Serial Dilution analysis performed:
For each SDG? [___] ✓ ___
On one of the SDG samples? [___] ✓ ___
For each matrix type? [___] ✓ ___
For each concentration range (low or med.)? [___] [___] ✓ ___

ACTION:
If no for any of the above, flag as estimated (J) detects ≥ MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.

A.1.21.2 Was a Field Blank or FE sample used for the Serial Dilution Analysis?

ACTION:
If yes, flag as estimated (J) detects ≥ MDL of all the SDG samples

A.1.21.3 Circle on Form VIII the Percent Differences (±10%) between sample results and its dilution results that are outside the control limits ± 10%
when initial concentrations ≥ 50 x MDLs.

Are results outside the control limits flagged with an "E" (Lab Qualifier) on Form VIII and all Form I's? [___] __ YES

**ACTION:**
If no, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.21.4 Are any %D values:

> 10%? [___] __ YES

≥ 100%? [___] __ YES

**ACTION:**
If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose raw data ≥ MDL; if the %D is ≥ 100%, reject (R) and red-line all associated samples with raw data ≥ MDL.

(NOTE: Replace “E” with “J” or “R” as appropriate.)

**Total/Dissolved or Inorganic/Total Analytes**

A.1.22 Were any analyses performed for dissolved as well as total analytes on the same sample(s)? [___] __ YES

 Were any analyses performed for inorganic as well as total analytes on the same sample(s)? [___] __ YES

**ACTION:**
If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic) and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:

1. The dissolved (or inorganic) concentration is greater than total concentration, and
2. greater than or equal to 5xMDL.

A.1.22.2 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%? [___] __ YES
A.1.22.3  Is any dissolved (or inorganic) concentration greater than its total concentration by more than 50%?

**ACTION:**
If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23  **Field Blank - Form I**

**NOTE:** Designate "Field Blank" as such on Form I

A.1.23.1  Was a Field/Rinsate Bank collected and analyzed with the SDG samples?

If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL (or 2xMDL when MDL>CRQL)?

If yes, circle the Field Blank value on Form I that is greater than the CRQL, (or 2 x MDL when MDL > CRQL).

Is any Field Blank value greater than CRQL also greater than the Preparation Blank value?

If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria?

**ACTION:**
If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results) greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result > MDL but ≤ CRQL, replace it with CRQL-U.

If the Field Blank value is less than the
Prep. Blank value, do not qualify the sample results due to the Field Blank criteria.

**NOTE:**
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

### A.1.24 Verification of Instrumental Parameters - Form IX, XA, XB, XI

#### A.1.24.1 Is verification report present for:

- Method Detection Limits (Form IX - Annually)?
- ICP-AES Interelement Correction Factors (Form XA & XB - Quarterly)?
- ICP-AES & ICP-MS Linear Ranges (Form XI - Quarterly)?

**ACTION:**
If no, contact CLP PO/TOPO for submittal from the laboratory.

### A.1.24.2 Method Detection Limits - Form IX

#### A.1.24.2.1 Are MDLs present on Form IX for:

- All the analytes?
- All the instruments used?
- Digested and undigested samples and Calib. Blanks?
- ICP-AES and ICP-MS when both instruments are used for the same analyte?

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than $\%_{CRQL}$.
A.1.24.2.2 Is MDL greater than the CRQL for any analyte?

If yes, is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?

ACTION:
If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.

A.1.24.3 Linear Ranges - Form XI

A.1.24.3.1 Was any sample result higher than the high linear range for ICP-ABS or ICP-MS?

Was any sample result higher than the highest calibration standard for mercury or cyanide?

If yes for any of the above, was the sample diluted to obtain the result reported on Form I?

ACTION:
If no, flag (J) as estimated the affected detects (> MDL) reported on Form I.

A.1.25 ICP-MS Tune Analysis - Form XIV

A.1.25.1 Was the ICP-MS instrument tuned prior to calibration?

ACTION:
If no, reject (R) and red-line all sample data for which tuning was not performed.

A.1.25.2 Was the tuning solution analyzed or scanned at least five times consecutively?

Were all the required isotopes spanning the analytical range present in the tuning solution?

Was the mass resolution within
Standard Operating Procedure
USEPA Region 2
Evaluation of Metals Data for the Contract Laboratory Program
Data Assessment and Contract Compliance Review

OP: HW-2 Revision 13 Appendix A.1 Sept. 2006

YES NO N/A

0.1 amu for each isotope in the tuning solution? [✓]    [ ]    [ ]

Was %RSD less than 5% for each isotope of each analyte in the tuning solution? [✓]    [ ]    [ ]

ACTION:
If no for any of the above, qualify all results ≥ MDL associated with that Tune as estimated “UJ”, and all non-detects associated with that Tune as “UJ”.

A.1.26 ICP-MS Internal Standards - Form XV

A.1.26.1 Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)? [✓]    [ ]    [ ]

Were all the target analyte masses bracketed by the masses of the five internal standards? [✓]    [ ]    [ ]

ACTION:
If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.

A.1.26.2 Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank? [✓]    [ ]    [ ]

If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed? [✓]    [ ]    [ ]

Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)? [✓]    [ ]    [ ]

ACTION:
If no for any of the above, flag detects as “UJ” and non-detects “UJ” of all the analytes with atomic masses between the atomic mass of the internal standard lighter -45-
than the affected internal standard, and the
atomic mass of the internal standard heavier
than the affected internal standard.

A.1.27 **Percent Solids of Sediments**

A.1.27.1 Are percent solids in sediment(s):

< 50%? [___] [___] ✓

**ACTION:**
If yes, qualify as estimated (J) all detects and
non-detects of a sample that has percent solids
less than 50% (i.e., moisture content greater than 50%).

**NOTE:**
Flag(J) only the sample results
that were not previously flagged
due to other QC criteria.

---

**Inorganic Data Review Narrative**

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<thead>
<tr>
<th>Case#</th>
<th>Site:</th>
<th>Matrix: Soil</th>
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<tbody>
<tr>
<td>SDG#</td>
<td>Lab:</td>
<td>Water</td>
</tr>
<tr>
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</tr>
</tbody>
</table>

**Sampling Team:** [_______]  **Reviewer:** [_______]  **Other:** [_______]

**A.2.1 Data Validation Flags:**
The following flags may have been applied in red by the data validator and must
be considered by the data user.

**J** - This flag indicates the result qualified as estimated

**R and Red-Line** - A red-line drawn through a sample result indicates unusable value.
The red-lined data are known to contain significant errors based on
documented information and must not be used by the data user.

**U** - This data validation qualifier is applied to sample results
≥ MDL when associated blank is contaminated

**Fully Usable Data** - The results that do not carry "J" or "red-line" are fully usable.

---

**2.2 Laboratory Qualifiers:**
The CLP laboratory applies a contractual qualifier on all
SAMPLE CALCULATION

EPA SAMPLE ID: VWAI-MW02-1110
COMPOUND: Manganese
CONCENTRATION: 70.7 ug/L
%Solids - NA

Raw Data result: 0.0707 mg/L

0.0707 mg/L (1000ug/1mg) = 70.7 ug/L

FIELD DUPLICATE SAMPLE SUMMARY

Note: All reported results are noted in the table below because the client requested that the MDL be used as reporting limit instead of the RL for this project. RPDs or absolute differences were calculated based on Region II guidelines: if results are >5X RL RPD is calculated, if results are <5X RL the absolute difference is calculated. Flags are applied to field duplicate pair only as follows: For RPD values - RPD ≥ 35% but <120% results are J, RPD >120%, results are R. For absolute difference values - >+/- 2X RL results are J, >+/- 4X RL results are R.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Duplicate Sample ID</th>
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<td>Analyte</td>
<td>Sample Conc.</td>
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</table>

Comments: No qualifications required.
January 16, 2012
SDG# SK2359, Spectrum Analytical, Inc.
Vieques Island, Puerto Rico - CTO-083, AOC-I

Dear Ms. Ott,

The following Data Validation report is provided as requested for the parameters noted in the table below for SDG # SK2359. The data validation was performed in accordance with the SW-846 methods utilized by the laboratory, the Region II Standard Operating Procedures for the Validation of Organic Data Acquired Using SW-846 Methods (8260B-Rev 2, August 2008- SOP #HW-24, 8270D-Rev 4, August 2008-SOP #HW-22), and professional judgment. Region II has not developed a validation checklist SOP for the inorganic method in this SDG (SW-846 methods 6010C) or the organic methods used to assess the fuels (SW-846 8015G for gasoline and 8015D_TPH for diesel range organics). The Region II Standard Operating Procedure for the Evaluation of Metals Data for the CLP was used as applicable for the metals data. For the other fraction alternative worksheets were provided. Region II flagging conventions were used. All areas of concern are discussed in the body of the report and a summary of data qualifications is provided.

<table>
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<th>Sample ID</th>
<th>Lab ID</th>
<th>Matrix</th>
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<th>SVOA</th>
<th>GRO</th>
<th>DRO</th>
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</table>

The following quality control samples were provided with this SDG: samples VWAI-TB01-110811, VWAI-TB01-110911 and VWAI-TB01-111011-trip blanks; samples VWAI-EB01-110811, VWAI-EB01-110911 and VWAI-EB01-111011-equipment.
blanks; and sample VWAI-MW05BP-1111-field duplicate of sample VWAI-MW05B-1111; sample VWAI-MW05P-1111-field duplicate of sample VWAI-MW05-1111.

The samples were evaluated based on the following criteria:

- Data Completeness
- Sample Condition
- Technical Holding Times
- GC/MS Tuning
- GC Performance
- ICP MS Tuning
- Initial/Continuing Calibrations
- ICSA/ICSAB Standards
- RL Standards
- Blanks
- Internal Standards
- Surrogate Recoveries
- Laboratory Control Samples
- Matrix Spike Recoveries
- Matrix Duplicate RPDs
- Serial Dilutions
- Field Duplicates
- Identification/Quantitation
- Reporting Limits
- Tentatively Identified Compounds

* - indicates that qualifications were not required based on this criteria

**Overall Evaluation of Data/Potential Usability Issues**

A summary of qualifications applied to the sample results are noted below for the fractions validated. Specific details regarding qualification of the data are addressed in the Specific Evaluation section of this narrative. If an issue is not addressed there were no actions required based on unmet quality criteria. When more than one qualifier is associated with a compound/analyte the validator has chosen the qualifier that best indicates possible bias in the results and flagged the data accordingly. However, information regarding all quality control issues is provided in the body of the report and on the qualification summary page. Please note that when a compound or analyte is flagged due to blank contamination the BL qualifier code takes precedence over all other qualifier codes except a code that explains rejected data.

**VOA**

No qualifications to the data were required.
SVOA

No qualifications to the data were required.

GRO

No qualifications to the data were required.

TPH

The field duplicate pair exhibited a non-compliant RPD (>20%) between the native sample and the field duplicate. The reported results were qualified as estimated J.

Select Filtered Metals

The laboratory did not analyze a CRI standard for the analyte manganese as required. The analyte was flagged as estimated for reported concentrations <2X RL. This resulted in the qualification of only one non-detect result as estimated UJ.

The laboratory did not perform a matrix spike or a serial dilution in this SDG. These QC samples are required by Region II. Qualifications were required.

Specific Evaluation of Data

Data Completeness

The SDG was received complete and intact. Resubmissions were not required.

Technical Holding Times

According to chain of custody records, sampling was performed on 11/8-10/11 and samples were received at the laboratory 11/9-12/11. All sample preparation and analysis was performed within Region II and/or method holding time requirements.

CRI Standards

Select Metals

The laboratory did not analyze a CRI standard for the analyte manganese. All positive results were above the action level of 2X the reporting limit. The reported non-detect result for manganese in sample VWAI-MW07-1111 was qualified as estimated UJ with a qualifier code of OT.
Matrix Spike

Select Filtered Metals

The laboratory did not perform a matrix spike sample on a sample from this SDG. Region II required that all positive results be qualified as estimated J because of this. Therefore, the reported positive results for iron and manganese were qualified as estimated J with a qualifier code of OT.

Serial Dilution

Select Filtered Metals

The laboratory did not perform a serial dilution sample on a sample from this SDG. Region II required that all positive results be qualified as estimated J because of this. Therefore, the reported positive results for iron and manganese were qualified as estimated J with a qualifier code of OT.

Field Duplicates

TPH

The field duplicate pair of samples VWAI-MW05-1111 and VWAI-MW05P-1111 exhibited a RPD >20% (27%) for Oil Range Organics (ORO). The reported positive results for ORO in the two samples were qualified as estimated J with a qualifier code of FD.

A summary of qualifications required is provided on the following page. Please do not hesitate to contact DataQual ES with any questions regarding this validation report.

Sincerely,

Jacqueline Cleveland
Vice President
### Summary of Data Qualifications

**VOA**

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<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
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<tr>
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**SVOA**

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**GRO**

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**TPH**

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<td>FD</td>
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### Select Filtered Metals

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<td>iron, manganese</td>
<td>+</td>
<td>J</td>
<td>OT</td>
</tr>
</tbody>
</table>
Glossary of Qualification Flags and Abbreviations

Qualification Flags (Q-Flags)

U  not detected above the reported sample quantitation limit
J  estimated value
UJ reported quantitation limit is qualified as estimated
N  analyte has been tentatively identified
JN analyte has been tentatively identified, estimated value
R  result is rejected; the presence or absence of the analyte cannot be verified

Method/Preparation/Field QC Blank Qualification Flags (Q-Flags)

Organic Methods

NA  The sample result for the blank contaminant is greater than the LOQ (2X sample LOQ for common laboratory contaminants) when the blank value is less than the LOQ. The sample result for the blank contaminant is not qualified with any blank qualifiers.

LOQ The sample result for the blank contaminant is less than the LOQ (2X sample LOQ for common laboratory contaminants) but greater than the MDL when the blank value is less than the LOQ. The sample result for the blank contaminant is changed to the LOQ and qualified as non-detect U.

Inorganic Methods

ICB/CCB/PB Action:

No Action - The sample result is greater than the LOQ and greater than ten times (10X) the blank value.

U - The sample result is greater than or equal to the MDL but less than or equal to the LOQ, result is reported as non-detect at the LOQ, when the ICB/CCB/PB result is less or greater than the LOQ.
Glossary of Qualification Flags and Abbreviations, continued

R - Sample result is greater than the LOQ and less than the ICB/CCB/PB value when the ICB/CCB/PB value is greater than the LOQ.

J - Sample result is greater than the ICB/CCB/PB value but less than 10X the ICB/CCB/PB value when ICB/CCB/PB value is greater than the LOQ.

J/UJ - Sample result is less than 10X LOQ when blank result is below the negative LOQ.

Field QC Blank action:

Note – Use field blanks to qualify data only if field blank results are greater than prep blank results.
Do not use rinsate blank associated with soils to qualify water samples and vice versa.

No Action - The sample result is greater than the LOQ and greater than ten times (10X) the blank value.

U - The sample result is greater than or equal to the MDL but less than or equal to the LOQ, result is reported as non-detect at the LOQ, when the FB result is less or greater than the LOQ.

R - Sample result is greater than the LOQ and less than the FB value when the FB value is greater than the LOQ.

J - Sample result is greater than the FB value but less than 10X the FB value when FB value is greater than the LOQ.

General Abbreviations

RL reporting limit
MDL method detection limit
IDL instrument detection limit
LOD Level of Detection
LOQ Level of Quantitation
+ positive result
- non-detect result
### QUALIFIER CODE REFERENCE

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<tr>
<th>Qualifier</th>
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<tbody>
<tr>
<td>TN</td>
<td>Tune</td>
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<tr>
<td>BSL</td>
<td>Blank Spike/LCS - High Recovery</td>
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<tr>
<td>BSH</td>
<td>Blank Spike/LCS - Low Recovery</td>
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<td>BD</td>
<td>Blank Spike/Blank Spike Duplicate (LCS/LCSD) Precision</td>
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<td>Matrix interference obscuring the raw data</td>
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<td>Matrix Spike/Matrix Spike Duplicate Precision</td>
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<td>Second Source - Bad reproducibility between tandem detectors</td>
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<tr>
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<td>Concentration Exceeds Linear Range</td>
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<td>Redundant Result - due to Dilution</td>
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<td>Other - explained in data validation report</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624
Soil Extract Volume: (ul)
Purge Volume: 5.0 (mL)

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<th>CAS NO.</th>
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<th>CONCENTRATION: ug/l</th>
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<td>78-87-5</td>
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<td>5.0</td>
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</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/Low/MED) LOW  
% Moisture: not doc.  
GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0  
Soil Extract Volume: ___________________ (uL) Soil Aliquot Volume: ___________________ (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
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</thead>
<tbody>
<tr>
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<td>0.61</td>
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</table>
Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: ______________________ (uL) Soil Aliquot Volume: ______________________ (uL)
Purge Volume: 5.0 (mL)

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<th>CAS NO.</th>
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<td>78-07-5</td>
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<td>U</td>
<td>0.33</td>
<td>0.50</td>
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<tr>
<td>78-07-5</td>
<td>1,2-Dichloropropane</td>
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<td>CAS NO.</td>
<td>COMPOUND</td>
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<td>0.41</td>
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<td>Benzene</td>
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<td>78-87-5</td>
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<td>1.0 ug/L</td>
<td>0</td>
<td>0.61</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITREM          Case No.: K2359
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
GC Column: DB-624      ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume:     (uL) Soil Aliquot Volume:     (uL)
Purge Volume: 5.0  (mL)

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<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
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<tr>
<td>78-87-5</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  Contract: 
Lab Code: MITKEM  Case No.: K2359  Mod. Ref No.: 
Matrix: (SOIL/SED/WATER)  WATER  Lab Sample ID: K2359-18A 
Sample wt/vol:  5.00 (g/mL) ML  Lab File ID: V0A7785.0 
Level: (TRACE/LOW/MED)  LOW  Date Received: 11/12/2011 
% Moisture: not dec.  Date Analyzed: 11/10/2011 
GC Column: DH-624  ID: 0.25 (mm) Dilution Factor: 1.0 
Soil Extract Volume:  5.0 (uL) Soil Aliquot Volume:  
Purge Volume:  5.0 (mL) 

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
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<th>DL</th>
<th>LOD</th>
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IA - FORM I VOA-1
VOLATILE ORGANICS ANALYSIS DATA SHEET

CLIENT SAMPLE NO. VVAT-MW04-1111
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<th>CAS NO.</th>
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<td>1,2-Dichloropropane</td>
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<td>CAS NO.</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  Contract:  
Lab Code: MITKEM  Case No.: K2359  Mod. Ref No.:  
Matrix: (SOIL/SED/WATER) WATER  SDG No.: SK2359  
Sample wt/vol: 1000 (g/mL) ML  
Level: (LOH/NED) LOW  
% Moisture:  
Concentrated Extract Volume: 1000 (µL)  
Injection Volume: 1.0 (µL) GPC Factor: 1.00  
GPC Cleanup: (Y/N) N  pH:  
Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
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<tbody>
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<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
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<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>2.0 ug/L</td>
</tr>
</tbody>
</table>
### Lab Name: SPECTRUM ANALYTICAL, INC.  
### Lab Code: MLTKEM  
### Case No.: K2359  
### Matrix: (SOIL/SED/WATER) WATER  
### Sample wt/vol: 1000 (g/mL) ML  
### Level: (LOW/MED) LOW  
### % Moisture: Decanted: (Y/N)  
### Concentrated Extract Volume: 1000 (ul)  
### Injection Volume: 1.0 (ul)  
### GPC Cleanup: (Y/N) N  
### pH:  
### Extraction: (Type) SEP  
### Date Received: 11/09/2011  
### Date Extracted: 11/14/2011  
### Date Analyzed: 12/02/2011  
### Dilution Factor: 1.0

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<td>U</td>
<td>1.3</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: K2359  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 1000 (g/mL) ML  
Level: (LOW/MED) LOW  
% Moisture: Decanted: (Y/N)  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL) GPC Factor: 1.00  
GPC Cleanup: (Y/N) N  
P H:  
Dilution Factor: 1.0  

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<td>117-81-7</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  Contract:
Lab Code: MITHEM  Case No.: K2359  Mod. Ref No.:
Matrix: (SOIL/SED/WATER)  WATER  Lab Sample ID: K2359-15A
Sample wt/vol: 1000 (g/mL) ML  Lab File ID: S3H7597.D
Level: (LOW/MED) LOW  Extraction: (Type) SEP
% Moisture: Decanted: (Y/N) Date Received: 11/11/2011
Concentrated Extract Volume: 1000 (uL) Date Extracted: 11/14/2011
Injection Volume: 1.0 (uL) GPC Factor: 1.00 Date Analyzed: 12/03/2011
GPC Cleanup: (Y/N) N  pH:  Dilution Factor: 1.0

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Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM    Case No.: K2359
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) mL
Level: (LOW/MEU) LOW

% Moisture: _______ Decanted: (Y/N) _______ Date Received: 11/11/2011
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00 Date Extracted: 11/14/2011

GPC Cleanup: (Y/N) N pH: _______ Date Analyzed: 12/03/2011
Dilution Factor: 1.0

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<td>5.0</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  Contract:  
Lab Code: MITKEM  Case No.: K2359  Mod. Ref No.:  
Matrix: (SOIL/SED/WATER)  WATER  Lab Sample ID: K2359-22E  
Sample wt/vol: 1000 (g/mL) ML  Lab File ID: S3H7603.D  
Level: (LOW/MED)  LOW  Extraction: (Type)  SEFF  
% Moisture:  Decanted: (Y/N)  Date Received: 11/12/2011  
Concentrated Extract Volume: 1000 (uL)  Date Extracted: 11/14/2011  
Injection Volume: 1.0 (uL) GPC Factor: 1.00  Date Analyzed: 12/03/2011  
GPC Cleanup: (Y/N)  N  pH:  
Dilution Factor: 1.0  

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Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITEFM
Case No.: K2359
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N pH: 
Dilution Factor: 1.0

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Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: K2359  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 1000 (g/mL) ML  
Level: (LOW/MED) LOW  
% Moisture:  
Decanted: (Y/N)  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL) GPC Factor: 1.00  
GPC Cleanup: (Y/N) N  
ph:  
Dilution Factor: 1.0  

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### Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

**Client:** CH2M-Hill, Inc.

**Client Sample ID:** VWAI-TB01-110811

**Lab ID:** K2359-01

**Project:** CTO-0083 Vieques AOC I

**Collection Date:** 11/08/11 9:30

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**Qualifiers:**

- ND - Not Detected at the Reporting Limit
- J - Analyte detected below quantitation limits
- B - Analyte detected in the associated Method Blank
- DF - Dilution Factor
- S - Spike Recovery outside accepted recovery limits
- R - RPD outside accepted recovery limits
- E - Value above quantitation range
- RL - Reporting Limit
**Analyses**

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**Qualifiers:**
- ND - Not Detected at the Reporting Limit
- J - Analyte detected below quantitation limits
- B - Analyte detected in the associated Method Blank
- DF - Dilution Factor
- S - Spike Recovery outside accepted recovery limits
- R - RPD outside accepted recovery limits
- E - Value above quantitation range
- RL - Reporting Limit
# Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

**Client:** CH2M-Hill, Inc.

**Client Sample ID:** VWAI-MW05P-1111

**Lab ID:** K2359-83

**Project:** CTO-0083 Vieques AOC I

**Collection Date:** 11/08/11 9:40

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## Analyses

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<td><strong>Gasoline Range Organics</strong></td>
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<td>100 ug/L</td>
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**Qualifiers:**

- **ND** - Not Detected at the Reporting Limit
- **J** - Analyte detected below quantitation limits
- **B** - Analyte detected in the associated Method Blank
- **DF** - Dilution Factor
- **S** - Spike Recovery outside accepted recovery limits
- **R** - RPD outside accepted recovery limits
- **E** - Value above quantitation range
- **RL** - Reporting Limit
### Analyses

**SW846 8015D GRO -- Gasoline Range Organic (GRO) by GC-FID**

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**Qualifiers:**
- ND - Not Detected at the Reporting Limit
- J - Analyte detected below quantitation limits
- B - Analyte detected in the associated Method Blank
- DF - Dilution Factor
- S - Spike Recovery outside accepted recovery limits
- R - RPD outside accepted recovery limits
- E - Value above quantitation range
- RL - Reporting Limit
**Analyses**

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<th>DF Date Analyzed</th>
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<tbody>
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**Qualifiers:**
- ND: Not Detected at the Reporting Limit
- J: Analyte detected below quantitation limits
- B: Analyte detected in the associated Method Blank
- DF: Dilution Factor
- S: Spike Recovery outside accepted recovery limits
- R: BPD outside accepted recovery limits
- E: Value above quantitation range
- RL: Reporting Limit
### Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

**Client:** CH2M-Hill, Inc.

**Client Sample ID:** VWAI-MW03-1111

**Lab ID:** K2359-11

**Project:** CTO-0083 Vieques AOC I

**Collection Date:** 11/09/11 11:15

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### Analyses

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<tr>
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<tr>
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<tr>
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**Qualifiers:**

- ND - Not Detected at the Reporting Limit
- J - Analyte detected below quantitation limits
- B - Analyte detected in the associated Method Blank
- DF - Dilution Factor
- S - Spike Recovery outside accepted recovery limits
- R - RPD outside accepted recovery limits
- E - Value above quantitation range
- RL - Reporting Limit

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**Page 56 of 138**
## SWD48 8015D GRO — Gasoline Range Organic (GRO) by GC-FID

<table>
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<tr>
<th>Analyses</th>
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**Qualifiers:**
- **ND** - Not Detected at the Reporting Limit
- **J** - Analyte detected below quantitation limits
- **B** - Analyte detected in the associated Method Blank
- **DF** - Dilution Factor
- **S** - Spike Recovery outside accepted recovery limits
- **R** - RPD outside accepted recovery limits
- **E** - Value above quantitation range
- **RL** - Reporting Limit

**Client:** CH2M-Hill, Inc.
**Client Sample ID:** VWA1-TB01-110911
**Lab ID:** K2359-14
**Project:** CTO-0083 Vinques AOC I
**Collection Date:** 11/09/11 7:00
**Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division**

Client: CH2M-Hill, Inc.
Client Sample ID: VWAI-TB01-111011
Lab ID: K2359-17

Project: CTO.0083 Vieques AOC I
Collection Date: 11/10/11 7:00

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<th>RL</th>
<th>Units</th>
<th>DF Date Analyzed</th>
<th>Batch ID</th>
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<td>SW846 8016D GRO -- Gasoline Range Organic (GRO) by GC-FID</td>
<td>ND</td>
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<td>ug/L</td>
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Qualifiers: ND - Not Detected at the Reporting Limit
J - Analyte detected below quantitation limits
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S - Spike Recovery outside accepted recovery limits
R - RPD outside accepted recovery limits
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**Analyses**

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<th>Qual</th>
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- RL - Reporting Limit

**Client:** CH2M-Hill, Inc.  
**Client Sample ID:** VWAI-MW02-1111  
**Lab ID:** K2359-20  
**Project:** CTO-0083 Vieques AOC I  
**Collection Date:** 11/10/11 9:50  

**Page 61 of 138**
**Spectrum Analytical, Inc. Featuring Hanibal Technology – Rhode Island Division**

Client: CH2M-Hill, Inc.
Client Sample ID: VWA1-EB01-111011
Lab ID: K2359-22

Project: CTO-0083 Vieques AOC I
Collection Date: 11/10/11 11:05

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**Qualifiers:**
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- **J** - Analyte detected below quantitation limits
- **B** - Analyte detected in the associated Method Blank
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- **S** - Spike Recovery outside accepted recovery limits
- **R** - RPD outside accepted recovery limits
- **E** - Value above quantitation range
- **RL** - Reporting Limit
### Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

**Client:** CH2M-Hill, Inc.  
**Client Sample ID:** VWAI-MW05-1111  
**Lab ID:** K2359-02  
**Project:** CTO-0083 Visques AOC 1  
**Collection Date:** 11/08/11 9:35

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## Analyses

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Page 104 of 138
### Analyses

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Page 105 of 138
### Analyses

<table>
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**Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division**

**Client:** CH2M-Hill, Inc.

**Client Sample ID:** VWAI-EB01-111011

**Lab ID:** K2359-22

**Project:** CTO-0083 Vieques AOC I

**Collection Date:** 11/10/11 11:05

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**Additional Notes:**
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- R - RPD outside accepted recovery limits
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- LOD - Limit of Detection
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Comments:

[Signature]

11/12/2011
**U.S. EPA - CLP**

**INORGANIC ANALYSIS DATA SHEET**

Lab Name: Spectrum Analytical, Inc.  
Contract: 933562, N62

Lab Code: MITKEM  
Case No.:  
SAS No.:  
SDG No.: SK2359

Matrix (soil/water): WATER  
Level (low/med): NED

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): ug/L

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<th>C</th>
<th>Q</th>
<th>M</th>
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Comments:

[Signature: JC]

[Date: 11/12/12]
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% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): ug/L
### Concentration Units

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<td>7439-89-6</td>
<td>Iron</td>
<td>54.2</td>
<td>( \frac{J}{OT} )</td>
<td>( \frac{J}{OT} )</td>
<td>31.0</td>
<td>50.0</td>
<td>200</td>
<td></td>
</tr>
<tr>
<td>7439-96-5</td>
<td>Manganese</td>
<td>1280</td>
<td>( \frac{J}{OT} )</td>
<td>( \frac{J}{OT} )</td>
<td>10.0</td>
<td>15.0</td>
<td>50.0</td>
<td></td>
</tr>
</tbody>
</table>

**Comments:**

---

For the given laboratory analysis, the concentration of Iron is 54.2 ug/L, and the concentration of Manganese is 1280 ug/L.
**U.S. EPA - CLP**

**INORGANIC ANALYSIS DATA SHEET**

Lab Name: Spectrum Analytical, Inc.
Lab Code: MITKEM
Matrix (soil/water): WATER
Level (low/med): MED
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
<th>MDL</th>
<th>LOD</th>
<th>FQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>7439-89-6</td>
<td>Iron</td>
<td>50</td>
<td>U</td>
<td>P</td>
<td></td>
<td>31.0</td>
<td>50.0</td>
<td>200</td>
</tr>
<tr>
<td>7439-96-5</td>
<td>Manganese</td>
<td>15</td>
<td>M</td>
<td></td>
<td>P</td>
<td>10.0</td>
<td>15.0</td>
<td>50.0</td>
</tr>
</tbody>
</table>

Comments:

---

EPA SAMPLE NO. VWA1-MX07-1111
Contract: 933562, N62
SDG No.: SK2359
Lab Sample ID: X2359-09
Date Received: 11/11/2011
### Chain of Custody Record

**Project No.:** JZ435, ET FK  
**Site Name:** ACC  
**Location:** WORCE  
**State:** RI 
**Sampler(s):** DEMATSAKER / C. VERA

---

#### Special Handling:
- **TAT- Indicate Date Needed:** 
- All TATs subject to laboratory approval. Min. 24-hour notification needed for rush. Samples disposed of after 30 days unless otherwise instructed.

---

#### Laboratory Analysis Details:

<table>
<thead>
<tr>
<th>Lab Id</th>
<th>Sample Id</th>
<th>Date</th>
<th>Time</th>
<th>Type</th>
<th>VOA Vials</th>
<th>Amber Glass</th>
<th>Clear Glass</th>
<th>Plastic</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>VNAI-N 535</td>
<td>11-03-11</td>
<td>09:30</td>
<td>X5</td>
<td>X1</td>
<td>4</td>
<td>2</td>
<td>2</td>
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<tr>
<td>C2</td>
<td>VNAI-N 535</td>
<td>11-03-11</td>
<td>09:30</td>
<td>X4</td>
<td>Gw</td>
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<td>4</td>
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<td>VNAI-N 535</td>
<td>11-03-11</td>
<td>09:30</td>
<td>X4</td>
<td>Gw</td>
<td>7</td>
<td>4</td>
<td>2</td>
</tr>
</tbody>
</table>

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#### Requisition Details:

- **Issuer:** [redacted]  
- **Recipient:** [redacted]  
- **Date:** 11-05-11  
- **Time:** 12:30

---

**Condition upon receipt:**
- [ ] Feed  
- [ ] Ambient  
- [ ] 0°C  
- [X] 4°C

---

**Notes:**

**QA/QC Reporting Level:**  
- [ ] Level I  
- [ ] Level II  
- [ ] Level III  
- [ ] Level IV  
- [ ] Other

**State specific reporting standards:**

---

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<table>
<thead>
<tr>
<th>Lab Id</th>
<th>Sample Id</th>
<th>Date</th>
<th>Time</th>
<th>Type</th>
<th># of VOA Vials</th>
<th># of Amber Glass</th>
<th># of Clear Glass</th>
<th># of Plastic</th>
</tr>
</thead>
<tbody>
<tr>
<td>03</td>
<td>VHAT-ΝΑΟΕ5P14d</td>
<td>11-02-11</td>
<td>07:40</td>
<td>G</td>
<td>4</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>04</td>
<td>VHAT-ΕΙΒ1-ΝΟΑ11</td>
<td>11-02-11</td>
<td>13:00</td>
<td>G</td>
<td>4</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
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</table>

QA/QC Reporting Level:
- Level I
- Level II
- Level III
- Level IV
- Other

State specific reporting standards:

Condition upon receipt: 
- Cold 
- Ambient  

Relinquished by:  
- Name: [Signature]  
- Date: 11-08-11  
- Time: 13:00

Received by:  
- Name: [Signature]  
- Date: 11-09-11  
- Time: 10:30
### Chain of Custody Record

**Report To:** siegel.brand@ch2m.com  
**Invoice To:**  
**Project No.:** 392415-61/FK  
**Site Name:** ADCI Viegues  
**Location:** Viegues  
**State:** PR  
**Sampler(s):** DW Water/16, Viegues

**Preservative Code:**  1=Na2S2O3  2=HCl  3=H2SO4  4=HNO3  5=NaOH  6=Ascorbic Acid  7=CH3OH  8=NaHSO4  9=H3PO4  10=  11=  

| Lab Id | Sample Id | Date | Time | Type | Matrix | # of VOA Vials | # of Amber Glass | # of Clear Glass | # of Plastic | TPC (ppb) | TPH (ppb) | Lead | Copper | Zinc | Cadmium | T.C. | Equipment Blank | Trip Blank |
|--------|-----------|------|------|------|--------|---------------|-----------------|-----------------|--------------|-------------|-----------|-----------|-------|--------|-------|---------|-----|----------------|-----------|
| 5      | VNAT-MNO5B-1111 | 11/20/11 | 0900 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 6      | VNAT-MNO5B-111A | 11/20 | 0900 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 7      | VNAT-MNO5B-111A | 11/20 | 0905 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 8      | VNAT-MNO5B-111A | 11/20 | 0905 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 9      | VNAT-MNO7-111 | 11/20 | 0920 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 10     | VNAT-MNO7-111A | 11/20 | 0920 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 11     | VNAT-MNO7-111A | 11/20 | 0920 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 12     | VNAT-MNO3-1111A | 11/20 | 1105 | G  | GW | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 13     | VNAT-MNO3-1111B | 11/20 | 1110 | G  | X14 | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  
| 14     | VNAT-MNO3-1111B | 11/20 | 1100 | G  | X14 | 2 | 2 | 2 | 2 | 2 | 1 | 1 | 3 | 2 | 2 | 2 | equipment blank |  

**Special Handling:**
- TAT- Indicate Date Needed:
- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rush orders.
- Samples disposed of after 30 days unless otherwise instructed.

**QA/QC Reporting Level**
- [ ] Level I
- [ ] Level II
- [ ] Level III
- [ ] Level IV
- [ ] Other

**State Specific Reporting Standards:**

**Condition upon receipt:** [ ] Fed [ ] Ambient [ ] C

**Relinquished by:** [Signature]

**Received by:** [Signature]

**Date:** [11/01/11]

**Time:** [13:30]

**E-mail to EDD Format:**

**Condition upon receipt:** [ ] Fed [ ] Ambient [ ] C

---

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# Chain of Custody Record

**Special Handling:**
- **FAT:** Indicate Date Needed: ____________
- All FATs subject to laboratory approval.
- Min. 24-hour notification is needed for rush.
- Samples disposed of after 30 days unless otherwise instructed.

**Report To:**

**Invoice To:**

**Project No.:** 232-83-9566

**Project Mgr.:**

**P.O. No.:**

**RQN:**

**Site Name:**

**Location:**

**State:**

**Sample(s):**

**Project: 7.5-28.5-6.5 Fls.**

**Containers:**

<table>
<thead>
<tr>
<th>No.</th>
<th>VOA Vials</th>
<th># of Amber Glass</th>
<th>Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>2</td>
<td>GW</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>2</td>
<td>X1</td>
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**Tests:**

<table>
<thead>
<tr>
<th>VOA No.</th>
<th>Date/Time</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10/09/99</td>
<td>09:20</td>
</tr>
<tr>
<td>2</td>
<td>10/09/99</td>
<td>11:20</td>
</tr>
</tbody>
</table>

- **QA/QC Reporting Level:**
  - Level I
  - Level II
  - Level III
  - Level IV
  - Other ____________

- **State Specific Reporting Standards:**

**Condition upon receipt:**
- **Ice:**
- **Ambient:**

**Relinquished by:**

**Received by:**

**Date:**

**Time:**

**Notes:**
**CHAIN OF CUSTODY RECORD**

Report To: 

Invoice To: 

Project No.: 9248S.FI.FK 

Site Name: 

Location: Vieques 

State: Puerto Rico 

Project Mgr.: Stephen Brand 

P.O. No.: 

RQN: 

Special Handling: TAT- Indicate Date Needed: 
- All TATs subject to laboratory approval. 
- Min. 24-hour notification needed for rushes. 
- Samples disposed of after 30 days unless otherwise instructed. 

Project(s): D. Whitaker 

List preservative code below: 

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
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</thead>
<tbody>
<tr>
<td>2</td>
<td>Water</td>
</tr>
<tr>
<td>6</td>
<td>Soil</td>
</tr>
<tr>
<td>11</td>
<td>Sludge</td>
</tr>
<tr>
<td>9</td>
<td>Air</td>
</tr>
</tbody>
</table>

QA/QC Reporting Level: 
- Level I 
- Level II 
- Level III 
- Level IV 
- Other 

State specific reporting standards: 

E-mail to: 

EDD Format: 

Relinquished by: 

Received by: 

Date: 11/14/11 

Time: 13:30 

Condition upon receipt: 
- Ice 
- Ambient 
- FedEx 

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### Chain of Custody Record

**Project No.:** 392485.F.E.FK  
**Site Name:** Vieques AOC  
**Location:** Vieques  
**State:** Puerto Rico  
**Sampler(s):** 

---

**Preservative Code:**  
- 1 = Na₂S₂O₃  
- 2 = HCl  
- 3 = H₂SO₄  
- 4 = HNO₃  
- 5 = NaOH  
- 6 = Ascorbic Acid  
- 7 = CH₃OH  

**Containers:**  

<table>
<thead>
<tr>
<th>Lab Id</th>
<th>Sample Id</th>
<th>Date</th>
<th>Time</th>
<th>Type</th>
<th>Matrix</th>
<th># of VOA Vials</th>
<th># of Amber Glass</th>
<th># of Clear Glass</th>
<th># of Plastic</th>
<th>QA/QC Reporting Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>VMAI-MW04-111</td>
<td>11/01/11</td>
<td>0920</td>
<td>G</td>
<td>GW</td>
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<tr>
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<td>VMAI-MW02-111MS</td>
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<tr>
<td>20</td>
<td>VMAI-MW02-111SD</td>
<td>11/01/11</td>
<td>0950</td>
<td>G</td>
<td>GW</td>
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<td>0950</td>
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<td>GW</td>
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**Relinquished by:**  
**Received by:**  
- **Date:** 11/01/11  
- **Time:** 1330

**Condition upon receipt:**  
- E-mail to 
- EDD Format

---

**Special Handling:**  
- All TATs subject to laboratory approval.  
- Min. 24-hour notification needed for rush.  
- Samples disposed of after 30 days unless otherwise instructed.
### Chain of Custody Record

**Special Handling:**
- TAT: Indicate Date Needed.
- All TATs subject to laboratory approval. Min. 24-hour notification needed for rush.
- Samples disposed of after 30 days unless otherwise instructed.

#### Laboratory Information
- **Project Mgr.:** Stephen Brand
- **P.O. No.:** 
- **RQN.:** 

#### List of Acid Names
1. Na₂SO₃
2. HCl
3. H₂SO₄
4. HNO₃
5. NaOH
6. Ascorbic Acid
7. CH₃OH
8. NaHSO₄
9. 
10. 
11. 

#### Sample Information

<table>
<thead>
<tr>
<th>Lab Id</th>
<th>Sample Id</th>
<th>Date</th>
<th>Time</th>
<th>Type</th>
<th>Matrix</th>
<th>QAP Grade</th>
<th>CO Grade</th>
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</thead>
<tbody>
<tr>
<td>15</td>
<td>WWI-NN084-111</td>
<td>1/10/11</td>
<td>09:20</td>
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<td>GW</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>20</td>
<td>WWI-NN02-111MS</td>
<td>1/10/11</td>
<td>09:50</td>
<td>G</td>
<td>GW</td>
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<tr>
<td>20</td>
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<td>2</td>
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<tr>
<td>20</td>
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<td>1/10/11</td>
<td>09:50</td>
<td>G</td>
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<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

#### Relinquished by
- **Relinquished by:** [Signature]

#### Received by
- **Received by:** [Signature]

#### Date & Time
- **Date:** 1/10/11
- **Time:** 13:30

#### Condition upon receipt
- [ ] Fedex
- [ ] Ambient
- **Temp.:** 56°F

---

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<table>
<thead>
<tr>
<th>Remarks: (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Custody Seal(s)</td>
</tr>
<tr>
<td>2. Custody Seal Nos.</td>
</tr>
<tr>
<td>3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists</td>
</tr>
<tr>
<td>4. Airbill</td>
</tr>
<tr>
<td>5. Airbill No.</td>
</tr>
<tr>
<td>6. Sample Tags</td>
</tr>
<tr>
<td>Sample Tag Numbers</td>
</tr>
<tr>
<td>7. Sample Condition</td>
</tr>
<tr>
<td>8. Cooler Temperature Indicator Bottle</td>
</tr>
<tr>
<td>9. Cooler Temperature</td>
</tr>
<tr>
<td>10. Does information on TR/COCs and sample tags agree?</td>
</tr>
<tr>
<td>11. Date Received at Laboratory</td>
</tr>
<tr>
<td>12. Time Received</td>
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</tbody>
</table>

### Sample Transfer

<table>
<thead>
<tr>
<th>Fraction (1)</th>
<th>VOA/VOA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area #</td>
<td></td>
</tr>
<tr>
<td>By</td>
<td></td>
</tr>
<tr>
<td>On</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>IR Temp Gun ID: MT-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coolant/Condition: ICE</td>
</tr>
<tr>
<td>Preservative Name/Lot No:</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Lab Sample ID</th>
<th>Preservation (pH)</th>
<th>VOA Matrix</th>
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</thead>
<tbody>
<tr>
<td>K2359-01</td>
<td>H2SO4</td>
<td>H</td>
</tr>
<tr>
<td>K2359-02</td>
<td>&lt;2</td>
<td>H</td>
</tr>
<tr>
<td>K2359-03</td>
<td>H</td>
<td></td>
</tr>
<tr>
<td>K2359-04</td>
<td>H</td>
<td></td>
</tr>
</tbody>
</table>

### VOA Matrix Key:
- US = Unpreserved Soil
- A = Air
- UA = Unpreserved Aqueous
- H = HCl
- M = MeOH
- E = Encore
- N = NaHCO3
- F = Freeze

### Sample Condition Form

- Rad OK: Yes / No
### Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

**Received By:**

**Reviewed By:**

**Work Order:** K2359  
**Client Name:** CH2M Hill Inc.

**Project Name/Event:** CTO-0083 Vieques AOC I / AOC-I

**Remarks:** (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.

1. **Custody Seal(s)**
   - Present/Absent
   - Intact/Broken
   - Lab Sample ID: K2359-05  
     - Preservation (pH): H
     - Soil Headspace or Air Bubble: Present
   - K2359-06
   - K2359-07
   - K2359-08
   - K2359-09
   - K2359-10
   - K2359-11
   - K2359-12
   - K2359-13
   - K2359-14

2. **Custody Seal Nos.**
   - N/A

3. **Traffic Reports/Chain of Custody Records (TR/CCDs) or Packing Lists**
   - Present/Absent
   - Lab Sample ID: K2359-15

4. **Airbill**
   - AirBill/Sticker
   - Present/Absent
   - FedEx 8762 4395 5649

5. **Airbill No.**
   - FedEx 8762 4395 5649

6. **Sample Tags**
   - Present/Absent
   - Sample Tag Numbers
     - Not Listed on Chain-of-Custody

7. **Sample Condition**
   - Intact/Broken/Leaking

8. **Cooler Temperature**
   - Indicator Bottle
   - Present/Absent

9. **Cooler Temperature**
   - 5 °C

10. **Does information on TR/CCDs and sample tags agree?**
    - Yes/No

11. **Date Received at Laboratory**
    - 11/11/2011

12. **Time Received**
    - 09:05

**Sample Transfer**

<table>
<thead>
<tr>
<th>Fraction (1)</th>
<th>VOA/VOA</th>
<th>Fraction (2)</th>
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</thead>
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<td>Area #</td>
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<td></td>
</tr>
<tr>
<td>By</td>
<td>By</td>
<td></td>
<td></td>
</tr>
<tr>
<td>On</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**IR Temp Gun ID:** MT-1

**Coolant Condition:** ICE

**Preservative Name/Lot No.:**

**VOA Matrix Key:**
- US = Unpreserved Soil
- UA = Unpreserved Aqueous
- H = HCl
- M = MeOH
- N = NaHSO4
- F = Freeze

**See Sample Condition Notification/Corrective Action Form:** Yes / No

**Rad OK:** Yes / No
Spectrum Analytical, Inc. Featuring Hanibal Technology -- Rhode Island Division

Received By: [Signature]
Reviewed By: [Signature]
Work Order: K2359
Client Name: CR2M Hill Inc.
Project Name/Event: CTO-0083 Vieques AOC I / AOC-I
Log-in Date: 11/12/2011

Remarks: (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.

<table>
<thead>
<tr>
<th>Lab Sample ID</th>
<th>Preservation (pH)</th>
<th>VOA Matrix</th>
<th>Soil Headspace or Air Bubble &gt; or equal to 1/4&quot;</th>
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</thead>
<tbody>
<tr>
<td>K2359-17</td>
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</tr>
<tr>
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<td>&lt;2</td>
<td>H</td>
<td></td>
</tr>
<tr>
<td>K2359-19</td>
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<td></td>
<td></td>
</tr>
<tr>
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<td>&lt;2</td>
<td>H</td>
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<tr>
<td>K2359-21</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>K2359-22</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4. Airbill
AirBill/Trcker: Present/Absent

5. Airbill No.
FedEx 8762 4395 5844

6. Sample Tags
Sample Tag Numbers: Present/Absent

7. Sample Condition
Intact/Broken/Leaking

8. Cooler Temperature Indicator Bottle
Present/Absent

9. Cooler Temperature
5 °C

10. Does Information on TR/COCs and sample tags agree?
Yes/No

11. Date Received at Laboratory
11/12/2011

12. Time Received
09:00

Sample Transfer

Fraction (1) TVOA/VOA
Fraction (2) BVOA/BREST/AB0

Area #
By
On

IR Temp Gun ID: MT-1
Coolant Condition: ICE
Preservative Name/Act No.

VOA Matrix Key:
US = Unpreserved Soil
A = Air
UA = Unpreserved Aqueous
H = HCl
M = MeOH
E = Encore
N = NaHSO4
F = Freeze

See Sample Condition Notification/Corrective Action Form
Yes/No

Rad OK
Yes/No
Spectrum Analytical, Inc. RL Division Sample Condition Notification

Project#: 2359  
Client: CH2M  
Client project #: WEF03  
Date of Receipt: 11-09-11  
Received By: Dan M.  

Unusual Occurance Description:  
VWAI - MW05 - W11 = 1L amber rec's broken
3L left  

Client Contacted:  
Contacted via: Phone/Fax/E-mail  
Date: 11/9  
Time:  
Contacted By:  
Name of person contacted:  

Client Response:  
Responded via: Phone/Fax/E-mail  
Date:  
Name of person responding:  
Responding to:  

Action Taken:  see attached email

Form ID: QAF.0005
Edward Lawler [Warwick]

From: Anita.Dodson@CH2M.com
Sent: Friday, November 11, 2011 10:41 AM
To: Hillary.Ott@CH2M.com; Edward Lawler [Warwick]; Stephen.Brand@CH2M.com
Cc: John.Swenfurth@CH2M.com
Subject: RE: another FW: Minor correction for FW: Samples shipped from AOC I today 11/10/11 from Vieques to Mitkem

Hillary - Thanks for the update.

Stephen – Since the cooler with FEDEX airbill 5866 is unaccounted for, I think it best that we go ahead and assume those samples will need to be recollected. IF FEDEX can find this cooler and get it to Mitkem tomorrow we will be ok, but barring a miracle, that’s likely NOT to happen. Please go ahead and plan on recollecting those samples. I will call your cell phone tomorrow if the cooler arrives at Mitkem.

Ed – Would it be possible to get an update from Mitkem on the status of cooler receipt tomorrow? I will be checking email and can also be reached on my cell at 757-284-9208.

Thanks,
Anita

From: Ott, Hillary/WDC
Sent: Friday, November 11, 2011 10:37 AM
To: Edward Lawler [Warwick]; Brand, Stephen/VBO; Dodson, Anita/VBO
Cc: Swenfurth, John/TPA
Subject: RE: another FW: Minor correction for FW: Samples shipped from AOC I today 11/10/11 from Vieques to Mitkem

Hello Everyone,

I just got off the phone with FEDEX. Two coolers ending in 5844, 5855 are still in Memphis, TN and should arrive tomorrow for Saturday delivery. The customer service representative placed an expedited shipping note to those two coolers. FEDEX has also put an inquiry into tracking down the third cooler, 5866. At this point no one is quite sure where this cooler is. They will be calling me with updates when they have located the package.

Please remember to be very careful when filling out the Air Bills. It looks like the address for Mitkem was incorrect.

Thanks,

Hillary Ott
Environmental Information Specialist
CH2M Hill
15010 Conference Center Drive
Chantilly, VA 20151

From: Edward Lawler [Warwick] [mailto:elawler@mitkem.com]
Sent: Friday, November 11, 2011 10:17 AM
To: Brand, Stephen/VBO; Ott, Hillary/WDC; Dodson, Anita/VBO
Cc: Swenfurth, John/TPA
Subject: another FW: Minor correction for FW: Samples shipped from AOC I today 11/10/11 from Vieques to Mitkem

Actually, the second cooler we received today was shipped two days ago.

So in summary, we have received both coolers that were shipped on 11/9, and none of the coolers shipped yesterday.

Our understanding is that the third cooler shipped yesterday, with the incorrect deliver-to address (airbill ending in 5866), needs someone from CH2M-Hill to contact FedEx and change the deliver-to address. We will be here until noon on Saturday to receive samples.

Thanks.
--Ed

From: Edward Lawler [Warwick]
Sent: Friday, November 11, 2011 10:12 AM
To: 'Stephen.Brand@CH2M.com'; Hillary.Ott@CH2M.com; Anita.Dodson@CH2M.com
Cc: John.Swenfurth@CH2M.com
Subject: Minor correction for FW: Samples shipped from AOC I today 11/10/11 from Vieques to Mitkem

There is a minor correction to the information below. We have received ONE cooler that was shipped yesterday. The airbill ending in 5650, the COC with SVOCs and DRO. The rest of the information remains the same.

--Ed

From: Edward Lawler [Warwick]
Sent: Friday, November 11, 2011 9:43 AM
To: 'Stephen.Brand@CH2M.com'; Hillary.Ott@CH2M.com; Anita.Dodson@CH2M.com
Cc: John.Swenfurth@CH2M.com
Subject: RE: Samples shipped from AOC I today 11/10/11 from Vieques to Mitkem

Good morning—

As of 9:40am on 11/11/11, we received the cooler that was supposed to be here yesterday. Samples are in the lab, and there is a good chance we will have the NO3 analyzed within the holding time. Cooler arrived at 5 degrees C.

However, we have NOT received the 3 coolers that were shipped yesterday. I tracked these, and two of them say they will be here by 10:30 (which I believe to be fiction, because we typically get all FedEx deliveries at the same time of the day, and there were no additional coolers on the truck that left here a short while ago). The third cooler (tracking number ending in 5866) they couldn't seem to find. I think the airbill was not completed correctly.

We will be here tomorrow to receive any samples that arrive on Saturday.

--Ed

From: Stephen.Brand@CH2M.com [mailto:Stephen.Brand@CH2M.com]
Sent: Thursday, November 10, 2011 3:39 PM
To: Edward Lawler [Warwick]; Hillary.Ott@CH2M.com; Anita.Dodson@CH2M.com
Cc: John.Swenfurth@CH2M.com
Subject: Samples shipped from AOC I today 11/10/11 from Vieques to Mitkem

Here are the chains, notes, and FedEx forms for today's shipment. Three coolers. Let me know if
there are any problems.

Stephen Brand P.G.
Hydrogeologist
5700 Cleveland Street, Suite 101
Virginia Beach, VA 23462
Direct - 757.671.6211
Fax - 703.376.9970
Mobile - 757.285.7685
www.ch2mhill.com
Target Zero!

Please consider the environment before printing this email.
Thanks Ed. Because the persulfate concentration in each well (immediately prior to sampling) was less than a cutoff point, we didn’t need to collect the ascorbic acid-preserved VOCs. The VOCs should have been HCl-preserved. Therefore, I can cancel the analysis of AA-VOCs samples (sample IDs end in “A”) as long as there is a corresponding HCl-preserved analysis for the same sample. Would you please cancel analysis for the attached samples and let me know if anything seems out-of-the-ordinary?

Thanks,
Mike Z.

From: Edward Lawler [Warwick] [mailto:elawler@mitkem.com]
Sent: Tuesday, November 15, 2011 8:04 AM
To: Zamboni, Michael/WDC
Cc: Brand, Stephen/VBO
Subject: RE: Vieques AOC I Groundwater Samples

Mike—

These were going to be analyzed this morning. I have put them "on-hold" so they won’t be run. If you decide to have these analyzed (I think the 7-day hold time goes out tomorrow), please let me know ASAP.

Thanks

–Ed

From: Michael.Zamboni@CH2M.com [mailto:Michael.Zamboni@CH2M.com]
Sent: Monday, November 14, 2011 5:27 PM
To: Edward Lawler [Warwick]
Cc: Michael.Zamboni@CH2M.com; Stephen.Brand@CH2M.com
Subject: Vieques AOC I Groundwater Samples

Hi Ed,

I was just wondering if you’ve analyzed any of the Vieques AOC I Groundwater Samples yet for VOCs from ascorbic acid-preserved vials. These are the sample IDs which end in "A". We were contemplating if we could cancel these analyses, but it may be a moot point if you’ve already analyzed them.

Thanks,
Mike Z.
<table>
<thead>
<tr>
<th>Sample ID to Cancel</th>
<th>Date/Time</th>
<th>Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW02-1111A</td>
<td>11/10/2011 9:50</td>
<td>VOCs (AA)</td>
</tr>
<tr>
<td>VWAI-MW02-11111MSA</td>
<td>11/10/2011 9:50</td>
<td>VOCs (AA)</td>
</tr>
<tr>
<td>VWAI-MW02-11111SDA</td>
<td>11/10/2011 9:50</td>
<td>VOCs (AA)</td>
</tr>
<tr>
<td>VWAI-MW05B-1111A</td>
<td>11/9/2011 9:00</td>
<td>VOCs (AA)</td>
</tr>
<tr>
<td>VWAI-MW05BP-1111A</td>
<td>11/9/2011 9:05</td>
<td>VOCs (AA)</td>
</tr>
</tbody>
</table>
Edward Lawler [Warwick]

From: Hillary.Ott@CH2M.com
Sent: Tuesday, November 15, 2011 3:50 PM
To: Edward Lawler [Warwick]
Cc: Michael.Zamboni@CH2M.com
Subject: Vieques AOCl - Login Revisions
Attachments: Vieques-AOCl-CTF.pdf

Hi Ed,

After reviewing the Login for Vieques AOC 1, I found a couple mistakes that will need to be updated. I have attached a Corrections to File Memo documenting the changes that need to be made. Also, I noticed on the login for VWAI-MW05-1111 and VWAI-MW05P-1111 you have them logged in for SW8260 but we cancelled those analyses last week.

Can you please update and send me the revised login?

Thanks,

Hillary Ott
Environmental Information Specialist
CH2M Hill
15010 Conference Center Drive
Chantilly, VA 20151
MEMORANDUM

CORRECTIONS TO COCS

TO: Ed Lawler, Mitkem.

COPIES: File
Data Validation Package
Laboratory Package SDG: K2359

FROM: Hillary Ott
Project Data Manager

DATE: November 15, 2011

This memo is to document corrections made to entries on the Chains of Custody (COC) and Logins for Vieques, AOCl.

The corrections include changes to the sample time on the Login:

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Date Collected</th>
<th>Incorrect Time Collected</th>
<th>Correct Time Collected</th>
<th>SDG</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-TB01-111011</td>
<td>11/10/2011</td>
<td>9:00</td>
<td>7:00</td>
<td>K2359</td>
</tr>
</tbody>
</table>

The corrections include changes to the sample date on the Login:

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Incorrect Date Collected</th>
<th>Correct Date Collected</th>
<th>Time Collected</th>
<th>SDG</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-EB01-111011</td>
<td>1/10/2011</td>
<td>11/10/2011</td>
<td>7:00</td>
<td>K2359</td>
</tr>
</tbody>
</table>

The corrections include cancellation of analyses:

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Date Collected</th>
<th>Time Collected</th>
<th>Requested Analyses</th>
<th>Analyses to Cancel</th>
<th>SDG</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW05-1111</td>
<td>11/8/2011</td>
<td>9:35</td>
<td>List 1 VOCs, SVOCs, GRO, DRO, ORO, Field Filtered Iron &amp; Manganese, Sulfate, Nitrate, TOC.</td>
<td>List 1 VOCs</td>
<td>K2359</td>
</tr>
<tr>
<td>VWAI-MW05P-1111</td>
<td>11/8/2011</td>
<td>9:40</td>
<td>List 1 VOCs, SVOCs, GRO, DRO, ORO, Field Filtered Iron &amp; Manganese, Sulfate, Nitrate, TOC.</td>
<td>List 1 VOCs</td>
<td>K2359</td>
</tr>
</tbody>
</table>
Edward Lawler [Warwick]

From: Hillary.Ott@CH2M.com
Sent: Wednesday, November 16, 2011 9:14 AM
To: Edward Lawler [Warwick]
Cc: Michael.Zamboni@CH2M.com
Subject: RE: Vieques AOCI - Login Revisions

Hi Ed,

Please cancel the 2 samples for VOC. The VWAI-MW05B-1111 and VWAI-MW05BP-1111 you received on 11/9 were the recollected samples for VOC.

Thanks,

Hillary Ott
Environmental Information Specialist
CH2M Hill
15010 Conference Center Drive
Chantilly, VA 20151

From: Edward Lawler [Warwick] [mailto:elawler@mitkem.com]
Sent: Wednesday, November 16, 2011 7:45 AM
To: Ott, Hillary/WDC
Subject: RE: Vieques AOCI - Login Revisions

Hi Hillary—

I recall Anita saying that those 2 VOC samples would be cancelled and recollected, but we never received the recollected samples. So I didn't cancel the original analyses. (with so much other confusion about this project, I may have missed another communication).

Can you please confirm that you don't want these 2 samples analyzed for VOCs? (we still have them scheduled for GRO analysis).

Thanks

- Ed

From: Hillary.Ott@CH2M.com [mailto:Hillary.Ott@CH2M.com]
Sent: Tuesday, November 15, 2011 3:50 PM
To: Edward Lawler [Warwick]
Cc: Michael.Zamboni@CH2M.com
Subject: Vieques AOCI - Login Revisions

Hi Ed,

After reviewing the Login for Vieques AOC 1, I found a couple mistakes that will need to be updated. I have attached a Corrections to File Memo documenting the changes that need to be made. Also, I noticed on the login for VWAI-MW05-1111 and VWAI-MW05P-1111 you have them logged in for SW8260 but we cancelled those analyses last week.

Can you please update and send me the revised login?

11/16/2011
I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: K2359  
LAB: Spectrum Analytical

SITE NAME: Vieques CTO-083 AOC I (limited compd list)

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent?  
ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, and/or cover letter signed release present?  
ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.2 Are case number and SDG number(s) contained in the narrative or cover letter?

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, and/or Chain of Custodies from the field samplers present for all samples sign release present?  
ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)?  

1.3 Sample Conditions/Problems
1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

[ ] \[ \]

ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects non"UJ".

Sampled 11/8-10/11

Analyzed 11/15-18/11

Temp 4-5°C

2.0 Holding Times

Read 11/9-12/11

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

[ ] \[ \]

The maximum holding time for aqueous samples is 14 days.

The maximum holding time for soils non aqueous samples is 14 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with HCL acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. For non-aqueous samples for volatile components that are frozen (less than 7°C) or are properly cooled (4°C ± 2°C) and preserved with NaHSO4, the maximum holding time is 14 days from sample collection. If
uncertain about preservation, contact the laboratory /sampling team to determine whether or not samples were preserved.

ACTION: Qualify sample results according to Table 1:

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Preserved</th>
<th>Criteria</th>
<th>Action</th>
<th>Detected Associated Compounds</th>
<th>Non-Detected Associated Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aqueous</td>
<td>No</td>
<td>≤7 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>No</td>
<td>&gt; 7 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>&gt; 14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td>Non Aqueous</td>
<td>No</td>
<td>≤14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes/No</td>
<td>&gt; 14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
</tbody>
</table>

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Water

b. Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:

a. Water

b. Soil

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data.
Assessments and contact the laboratory/project officer/appropriate official for an explanation/resubmittal, make any necessary corrections and document effect in the Data Assessment.

3.3 Were the surrogate recovery limits followed per Table 2. If Table 2 criteria were not followed, the laboratory may use in-house performance criteria (per SW-846, Method 8000C, section 9.7). Other compounds may be used as surrogates, depending upon the analysis requirements.

Table 2. Surrogate Spike Recovery Limits for Water and Soil/Sediments

<table>
<thead>
<tr>
<th>DMC</th>
<th>Recovery Limits (% Water)</th>
<th>Recovery Limits Soil/Sediment</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Bromofluorobenzene</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dibromofluoromethane</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Toluene-d₄</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dichloroethane-d₄</td>
<td>80-120</td>
<td>70-130</td>
</tr>
</tbody>
</table>

Note: Use above table if laboratory did not provide in house recovery criteria.

Note: Other compounds may be used as surrogated depending upon the analysis requirements.

3.4 Were outliers marked correctly with an asterisk?

ACTION: Circle all outliers with a red pencil.

3.5 Were one or more volatile surrogate recoveries out of specification for any sample or method blank. Table 2.

If yes, were samples reanalyzed?

Were method blanks reanalyzed?
ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects, but qualify positive results as estimated "J".

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. If one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose the blank problem to be an isolated occurrence.

3.6 Are there any transcription/calculation errors between raw data and reported data? [ ] [ ] [ ]

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Sample (Form III/Equivalent)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG? [ ] [ ]
Note: LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation/resubmittals. Make note in the data assessment.

4.2 Were the Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

A. Water
   [ ] [ ] [ ]

B. Soil
   [ ] [ ] [ ]

C. Med Soil
   [ ] [ ] [ ]

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

4.3 Have in house LCS recovery limits been developed (Method 8000C, Sect 9.7).

   [ ] [ ] [ ]

4.4 If in house limits are not developed, are LCS acceptance recovery limits between 70 - 130% (Method 8000c Sect 9.5)? [ ] [ ]

4.5 Were one or more of the volatile LCS recoveries outside the in house laboratory recovery criteria for spiked analytes? If in house limits are not present use 70 - 130% recovery limits [ ] [ ]
## Table 3. LCS Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Action</th>
<th>Detected Spiked Compounds</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td></td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td></td>
<td>UJ</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td></td>
<td>No Qualifications</td>
</tr>
</tbody>
</table>

### 5.0 Matrix Spikes (Form III or equivalent)

#### 5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix? [ ] [ ]

**NOTE:** The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, Method 8260B, Sect 8.4.2).

#### 5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III? [ ] [ ]

**ACTION:** If any data are missing take action as specified in section 3.2 above.

#### 5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples...
of similar matrix or concentration level. Laboratories analyzing
one to ten samples per month are required to analyze at least one
MS per month \[\text{page 8000C, section 9.5.} \] )

a. Water

b. Waste

c. Soil/Solid

Note: The LCS is spiked with the same analytes at the same
concentrations as the matrix spike (SW-846 8000C, Section
9.5). If different make note in data assessment.
Matrix/LCS spiking standards should be prepared from
volatile organic compounds which are representative of the
compounds being investigating. At a minimum, the matrix
spike should include 1,1-dichloroethene, trichloroethene,
chlorobenzene, toluene, and benzene. The concentration of
the LCS should be determined as described SW-Method 8000C
Section 9.5.

ACTION: If any MS/MD, MS/MSD or replicate data are
missing, take the action specified in 3.2 above.

5.4 Have in house MS recovery limits been developed (Method 8000C,
Sect 9.7) for each matrix.

5.5 Were one or more of the volatile MS/MSD recoveries
outside of the in-house laboratory recovery criteria
for spiked analytes? If none are present, then use 70-130%
recovery as per SW-846, 8000C, Sect. 9.5.4.

ACTION: Circle all outliers with a red pencil.

NOTE: If any individual % recovery in the MS (or MSD) falls
outside the designated range for recovery the reviewer
should determine if there is a matrix effect. A matrix
effect is indicated if the LCS data are within limits but
the MS data exceeds the limits.
NOTE: No qualification of data is necessary on MS and MSD data alone. However, using informed professional judgement, the data reviewer may use MS and MSD results in conjunction with other QC criteria to determine the need for some qualification.

Note: The data reviewer should first try to determine to what extent the results of the MS and MSD affect the associated data. This determination should be made with regard to the MS and MSD sample itself, as well as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affect all associated samples, and the reviewer must use professional judgement to qualify the data from all associated samples.

Note: The reviewer must use professional judgement to determine the need for qualification of non-spiked compounds.

ACTION: Follow criteria in Table 4 when professional judgement deems qualification of sample.

Table 4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Detected Spiked Compounds</td>
</tr>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td>No Qualifications</td>
</tr>
</tbody>
</table>

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6.0 **Blank (CIP Form IV Equivalent)**

6.1 Is the Method Blank Summary form present?  

6.2 Frequency of Analysis: Has a method blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch?  

6.3 Has a method blank been analyzed for each GC/MS system used?  

**ACTION:** If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.  

Is the chromatographic performance (baseline stability) for each instrument acceptable for volatile organic compounds?  

7.0 **Contamination**

**NOTE:** "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.
7.2 Do any field/rinse blanks have positive volatile organic compound results? _  __

**ACTION:** Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

**NOTE:** All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surge or calibration QC problems.

**ACTION:** Follow the directions in Table 5 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

```
VWA-1-TP01-110811-MOG
VWA-1-TP01-110911-MOG
VWA-1-TP01-111011-MOG
VWA-1-EP01-110811-MOG
VWA-1-EP01-110911-MOG
VWA-1-EP01-111011-MOG
```

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Table 5. Volatile Organic Analysis Blank Contamination Criteria

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td>No qualification</td>
<td></td>
</tr>
<tr>
<td>&lt; CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>&gt; CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report the concentration for the sample with a U, or qualify the data as unusable R</td>
<td></td>
</tr>
<tr>
<td>= CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
</tr>
<tr>
<td>Gross contamination</td>
<td>Detects</td>
<td>Qualify results as unusable R</td>
<td></td>
</tr>
</tbody>
</table>

* 2x the CRQL for methylene chloride, 2-butane, and acetone
** Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 µg/L.

**NOTE:** If gross blank contamination exists (e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected volatile organic target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
7.3 Are there field/rinse/equipment blanks associated with every sample?

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.

NOTE: For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgment to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?

9.3 Has an instrument performance check solution (BFB)
been analyzed for every twelve hours of sample analysis per instrument?(see Table 4, SW-846, page 8260B-36)

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS GC/MS tuning data are available.

ACTION: If the laboratory/project officer cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable, "R".

9.4 Have the ion abundances been normalized to m/z 95?

9.5 Have the ion abundance criteria been met for each instrument used?

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.)

9.7 Have the appropriate number of significant figures (two) been reported?

ACTION: If large errors exist, take action as specified in section 3.2.

9.8 Are the spectra of the mass calibration compounds acceptable.

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.
10.0 Target Analytes (CLP Form I Equivalent)

10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate
   
   ![Checkmark]

b. Matrix spikes and matrix spike duplicates
   
   ![Checkmark]

c. Blanks
   
   ![Checkmark]

d. Laboratory Control Samples
   
   ![Checkmark]

10.2 Are the reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

a. Samples and/or fractions as appropriate
   
   ![Checkmark]

b. Matrix spikes and matrix spike duplicates (Mass spectra not required)
   
   ![Checkmark]

c. Blanks
   
   ![Checkmark]

d. Laboratory Control Samples
   
   ![Checkmark]

ACTION: If any data are missing, take action specified in 3.2 above.

10.3 Is chromatographic performance acceptable with respect to:

Baseline stability?

   ![Checkmark]
Resolution?  YES  NO  N/A
Peak shape?  YES  NO  N/A
Full-scale graph (attenuation)?  YES  NO  N/A
Other: ____________________________

ACTION: Use professional judgement to determine the acceptability of the data.

10.4 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample?

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, contact the lab for missing spectra.

10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

10.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

10.7 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be
positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

11.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

11.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier? [ ] [ ] [ ]

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

11.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate [ ] [ ] [ ]

b. Blanks [ ] [ ] [ ]

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a CAS#.

NOTE: If TICs are present in the associated blanks take action as specified in section 3.2 above.
11.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)?

ACTION: 1. Flag with "R" any target compound listed as a TIC.

2. Make sure all rejected compounds are properly reported if they are target compounds.

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

11.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO₂(M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be
reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL's adjusted to reflect sample dilutions and, for soils, sample moisture? [ ] [ ] [ ]

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC accedence dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration? [ ] [ ] [ ]

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above.

14.0 GC/MS Initial Calibration (CLP Form VI Equivalent)
14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction?  

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

ACTION: If the percent relative standard deviation (% RSD) is > 20%, (8000C-39) qualify positive results for that analyte "J". When % RSD > 90%, qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.2 Are all average RRFs > 0.050?

NOTE: (Method Requirement) For SFCC compounds, the individual RRF values must be ≥ the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

Chloromethane 0.10
1,1-Dichloroethane 0.10
Bromoform 0.10
Chlorobenzene 0.30
1,1,2,2-Tetrachloroethane 0.30

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, or for the requirements for the 5 compounds in 14.2 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration.

NOTE: (Method Requirement) For the following CCC compounds, the % RSD values must be ≤ 30.0%. If % RSD values reported are > 30.0% document in the Data Assessment.
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 20.0%, or > 30% for the 6 compounds in 14.3 above, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF?  [ ]

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.) [ ]

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

15.0 GC/MS Calibration Verification (CLP Form VII Equivalent)
15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest? [ ] [ ] [ ]

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument? [X] [ ] [ ]

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

____________________________________________________________________________________

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

15.3 Was the % D determined from the calibration verification determined using RRF or CF? [X] [ ] [ ]

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.

15.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2). [X] [ ] [ ]

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be < 20.0%. If %D values reported are > 20.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride
ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

15.5 Do any volatile compounds have a RRF < 0.05? [ ] V

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

<table>
<thead>
<tr>
<th>Compound</th>
<th>RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloromethane</td>
<td>0.10</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>0.10</td>
</tr>
<tr>
<td>Bromoform</td>
<td>0.10</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>0.30</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>0.30</td>
</tr>
</tbody>
</table>

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, or < the requirements for the 5 compounds in section 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard (IS) areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to + 100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)? [ ] V

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ACTION:  If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION:  List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area Lower Limit</th>
<th>Area Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when the associated IS are counts area > + 100%.

3. If the IS area is below the lower limit (< -50%), qualify all associated non-detects (U-values) "J".

4. If extremely low area counts are reported (< -25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

ACTION:  Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.
17.0 Field Duplicates

17.1 Were any field duplicates submitted for volatile analysis?

[Blank]

**ACTION:** Compare the reported results for field duplicates and calculate the relative percent difference.

**ACTION:** Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.
### Initial Calibration Date:

11/14/2011

#### RRF and %RSD Calculations:

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>1,2-dichloroethane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>0.2720</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>1251031</td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>1151324</td>
</tr>
<tr>
<td>Conc. of Internal STD</td>
<td>50</td>
</tr>
<tr>
<td>Conc. of Compound</td>
<td>200</td>
</tr>
<tr>
<td>Calculated RRF</td>
<td>0.272</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>benzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>12.90</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>RRF of STD 1</td>
<td>0.9940</td>
</tr>
<tr>
<td>RRF of STD 2</td>
<td>0.9550</td>
</tr>
<tr>
<td>RRF of STD 3</td>
<td>0.8120</td>
</tr>
<tr>
<td>RRF of STD 4</td>
<td>0.8100</td>
</tr>
<tr>
<td>RRF of STD 5</td>
<td>0.7160</td>
</tr>
<tr>
<td>RRF of STD 6</td>
<td>0.9750</td>
</tr>
<tr>
<td>Calculated % RSD</td>
<td>12.90</td>
</tr>
</tbody>
</table>

### Continuing Calibration File ID:

11/15/2011

#### RRF and %D Calculations:

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>1,2-dichloroethane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>0.289</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>377827</td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>1309593</td>
</tr>
<tr>
<td>Conc. of Internal STD</td>
<td>50</td>
</tr>
<tr>
<td>Conc. of Compound</td>
<td>50</td>
</tr>
<tr>
<td>Calculated RRF</td>
<td>0.289</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>1,2-dichloropropane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value</td>
<td>1.5</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Average RRF</td>
<td>0.252</td>
</tr>
<tr>
<td>Calibration Check RRF</td>
<td>0.248</td>
</tr>
<tr>
<td>Calculated % D</td>
<td>1.59</td>
</tr>
</tbody>
</table>
**Sample Calculation**

**Sample ID:** WWAI-MW07-1111  
**Standard ID:** 11/15/2011  
**Compound:** benzene  
**Concentration:** 5.3 ug/L

<table>
<thead>
<tr>
<th></th>
<th>Water (ug/L)</th>
<th>Soil (ug/Kg)</th>
<th>Soil ug/Kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>120597</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>1298350</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conc. of Internal (ng)</td>
<td>250</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>RRF of Compound</td>
<td>0.877</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dilution Factor</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Weight of Sample</td>
<td>NA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of Sample</td>
<td>5</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>% Moisture</td>
<td>NA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aliquot of sample</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Concentration</td>
<td>5.30</td>
<td>#DIV/0!</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>RT of Internal STD</th>
<th>RT of Compound</th>
<th>RRT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td>5.057</td>
<td>4.832</td>
<td>0.956</td>
</tr>
<tr>
<td>Standard</td>
<td>5.057</td>
<td>4.832</td>
<td>0.956</td>
</tr>
</tbody>
</table>
REPORT NARRATIVE

Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.

Client: CH2M-Hill, Inc.

Project: CTO-0083 Vieques AOC I

Laboratory Workorder / SDG #: K2359

SW846 8260C, VOC by GC-MS

I. SAMPLE RECEIPT

Several communications with the client regarding samples to analyze and/or cancel are included in the Sample Transmittal section of this report.

II. HOLDING TIMES

A. Sample Preparation:

   All samples were prepared within the method-specified holding times.

B. Sample Analysis:

   All samples were analyzed within the method-specified holding times.

III. METHODS

   Samples were analyzed for a select list of volatile compounds following procedures in laboratory test code: SW846 8260C.

IV. PREPARATION

   Aqueous Samples were prepared following procedures in laboratory test code: SW5030.

V. INSTRUMENTATION

   The following instrumentation was used:
Instrument Code: V10
Instrument Type: GCMS-VOA
Description: HP7890A
Manufacturer: Agilent
Model: 7890A / 5975C
GC Column used: 30 m X 0.25 mm ID [1.40 um thickness] DB-624 capillary column.

Instrument Code: V6
Instrument Type: GCMS-VOA
Description: HP6890 / HP5973
Manufacturer: Hewlett-Packard
Model: 6890 / 5973
GC Column used: 30 m X 0.25 mm ID [1.40 um thickness] DB-624 capillary column.

VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Surrogates:

Surrogate standard percent recoveries were within the QC limits.

D. Laboratory Control Spikes (LCS):

Percent recoveries for lab control samples were within the QC limits.

E. Internal Standards:

Internal standard peak areas were within the QC limits.

F. Dilutions:

No sample in this SDG required analysis at dilution.

G. Samples:

No other unusual occurrences were noted during sample analysis.
I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: 

Date: __________ 12/9/11

Page 10 of 138
E - The concentration of this analyte exceeds the calibration range of the instrument.

A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: K2354

SITE NAME: Vieques CTO-083 AOC I

LAB: Spectrum Analytical
(limited compd list)

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format?

ACTION: If not, note the effect on review of the data in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present?

2.2 Are case number and SDG number(s) contained in the narrative or cover letter?
II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples?

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all non-detects data are qualified as unusable (R), and detects are flagged "J".

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".  
Sampled 11/8-10/11  Extr. 11/14/11
Temp 4.5°C

2.0 Holding Times

2.1 Have any semivolatile technical holding times, determined from date of collection to date of extraction, been exceeded?

Continuous extraction of water samples for semivolatile analysis must be started within 7 days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within
40 days of the date of extraction.

Table of Holding Time Violations

(See Traffic Report)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Matrix</th>
<th>Date Sampled</th>
<th>Date Lab Received</th>
<th>Date Extracted</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).
3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water
   
   b. Low/Med Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water

b. Low/Med Soil

ACTION: If CLP deliverables are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk?  

ACTION: Circle all outliers in red.

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank (Reviewer should use lab in house recovery limits. Use surrogate recovery limits from USEPA National Functional Guidelines January 2005 page 130, if in house limits are not available. See Method 8000B-43 or 80000C-24).

Note: Examine lab in house limits for reasonableness.

If yes, were samples re-analyzed?
**2H - FORM II SV-2**

**WATER SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY**

<table>
<thead>
<tr>
<th>CLIENT SAMPLE NO.</th>
<th>SDMC1 (NBZ) #</th>
<th>SDMC2 (FBP) #</th>
<th>SDMC3 (TPH) #</th>
<th>TOT OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td>01 MB-63017</td>
<td>102</td>
<td>99</td>
<td>126</td>
<td>0</td>
</tr>
<tr>
<td>02 VWAJ-MW05-11</td>
<td>93</td>
<td>92</td>
<td>51</td>
<td>0</td>
</tr>
<tr>
<td>03 VWAJ-MW05P-1</td>
<td>93</td>
<td>90</td>
<td>47</td>
<td>1</td>
</tr>
<tr>
<td>04 VWAJ-EB01-11</td>
<td>89</td>
<td>86</td>
<td>99</td>
<td>0</td>
</tr>
<tr>
<td>05 VWAJ-MW03-11</td>
<td>78</td>
<td>80</td>
<td>61</td>
<td>0</td>
</tr>
<tr>
<td>06 VWAJ-MW07-11</td>
<td>70</td>
<td>54</td>
<td>49 *</td>
<td>1</td>
</tr>
<tr>
<td>07 VWAJ-EB01-11</td>
<td>94</td>
<td>87</td>
<td>87</td>
<td>0</td>
</tr>
<tr>
<td>08 VWAJ-MW04-11</td>
<td>85</td>
<td>85</td>
<td>71</td>
<td>0</td>
</tr>
<tr>
<td>09 VWAJ-MW02-11</td>
<td>91</td>
<td>88</td>
<td>72</td>
<td>0</td>
</tr>
<tr>
<td>10 VWAJ-MW02-11</td>
<td>94</td>
<td>95</td>
<td>72</td>
<td>0</td>
</tr>
<tr>
<td>11 VWAJ-MW02-11</td>
<td>95</td>
<td>91</td>
<td>78</td>
<td>0</td>
</tr>
<tr>
<td>12 VWAJ-EB01-11</td>
<td>90</td>
<td>85</td>
<td>97</td>
<td>0</td>
</tr>
<tr>
<td>13 LCS-63017</td>
<td>93</td>
<td>92</td>
<td>121</td>
<td>0</td>
</tr>
<tr>
<td>14 LCSD-63017</td>
<td>88</td>
<td>83</td>
<td>116</td>
<td>0</td>
</tr>
</tbody>
</table>

**QC LIMITS**

SDMC1 (NBZ) = Nitrobenzene-d5  
SDMC2 (FBP) = 2-Fluorobiphenyl  
SDMC3 (TPH) = Terphenyl-d14

# Column to be used to flag recovery values

* Values outside of contract required QC limits

D DMC diluted out
Were method blanks re-analyzed?

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

1. Flag all positive results as estimated ("J").

2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.

3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".

2. Non-detects for that fraction should be qualified as unusable (R).

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II?

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document
effect in data assessments.

4.0 **Matrix Spikes (Form III/Equivalent)**

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate/or duplicate unsiked Sample recoveries been listed on the Recovery Form (Form III)?

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**NOTE:** Method 3500B/page 4 states the spiking compounds:

- **Base/neutrals**
  - 1,2,4-Trichlorobenzene
  - Acenaphthene
  - 2,4-Dinitrotoluene
  - Pyrene
  - N-Nitroso-di-n-propylamine
  - 1,4-Dichlorobenzene

- **Acids**
  - Pentachlorophenol
  - Phenol
  - 2-Chlorophenol
  - 4-Chloro-3-methylphenol
  - 4-Nitrophenol

**Note:** Some projects may require the spiking of specific compounds of interest.

**Note:** See Method 8270D-sec 8.4.2 for deciding on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate. If samples are expected to contain target analytes, then laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratory should use a matrix spike and matrix spike duplicate pair.

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Low Water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>b. Low Solid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>c. Med Solid</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. It may be necessary to contact the lab to obtain the required data.

NOTE: If the data has not been reported on CLP equivalent form, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

4.3 Were matrix spikes performed at concentration equal to 100µg/L for acid compounds, and 200µg/l for base compounds (Method 3500B-4), or those specified in project plan. [Yes/No] [ ]

4.4 How many semivolatile spike recoveries are outside Laboratory in house MS/MSD recovery limits (use recovery limits values in Method 8270D-43644 Table 6 if in house values not available).

Water

\[ \frac{0}{6} \] out of \[ 6 \]

Solids

\[ \frac{0}{6} \] out of \[ 6 \]
4.5 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

<table>
<thead>
<tr>
<th>Water</th>
<th>Solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 out of 3</td>
<td>___ out of ___</td>
</tr>
</tbody>
</table>

**ACTION:** Circle all outliers with red pencil.

**ACTION:** No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a Laboratory Control Sample (LCS) analyzed with each analytical batch?

14 ___ ___

**NOTE:** When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 **Blanks (Form IV/Equivalent)**

5.1 Is the Method Blank Summary (Form IV) present?

1 ___ ___

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch?

1 ___ ___

5.3 Has a method blank been analyzed either after
the calibration standard or at any other time
during the analytical shift for each GC/MS system
used?

ACTION: If any method blank data are missing, call
lab for explanation/resubmittal. If not
available, use professional judgement to
determine if the associated sample data
should be qualified.

5.4 Chromatography: review the blank raw data-
chromatograms (RICs), quant reports or data system
printouts and spectra.

Is the chromatographic performance (baseline
stability) for each instrument acceptable for
the semivolatiles?

ACTION: Use professional judgement to determine the
effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled
water blanks" are validated like any other
sample and are not used to qualify the data.
Do not confuse them with the other QC blanks
discussed below.

6.1 Do any method/instrument/reagent blanks have
positive results for target analytes and/or TICs?
When applied as described below, the contaminant
concentration in these blanks are multiplied by
the sample dilution factor and corrected for
percent moisture where necessary.

6.2 Do any field/rinse/ blanks have positive results
for target analytes and/or TICs (if required,
see section 10 below)?
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

\[ VWAI-EB01-110811 - N0\]  
\[ VWAI-EB01-110911 - N0\]  
\[ VWAI-EB01-111011 - N0\]
Blank Action for Semivolatile Analyses

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>&lt; CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>= CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>&gt; CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and &lt; blank contamination</td>
<td></td>
<td>Report concentration of sample with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and ≥ blank contamination</td>
<td></td>
<td>No qualification required</td>
</tr>
</tbody>
</table>

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

NOTE: If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required.

6.3 Are there field/rinse/equipment blanks associated with every sample? [✓] Yes

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

6.4 Was a instrument blank analyzed after each sample/dilution which contained a target compound
that exceeded the initial calibration range.  

6.5 Does the instrument blank have positive results for target analytes and/or TICs?  

Note: Use professional judgement to determine if carryover occurred and qualify analytes accordingly.

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270D? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column.

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (Form V/Equivalent)

8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)?

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5
page 8270D-12).

8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift?

8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

<table>
<thead>
<tr>
<th>DATE</th>
<th>TIME</th>
<th>INSTRUMENT</th>
<th>SAMPLE NUMBERS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
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</tr>
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<td></td>
</tr>
</tbody>
</table>

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

8.4 Have the ion abundances been normalized to m/z 198?

8.5 Have the ion abundance criteria been met for each instrument used?

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).
ACTION: If ion abundance criteria are not met, take action specified in section 3.2

8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)

8.7 Have the appropriate number of significant figures (two) been reported?

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

8.8 Are the spectra of the mass calibration compound acceptable?

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

9.0 Target Analytes

9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate

b. Matrix spikes and matrix spike duplicates

c. Blanks

9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270D-14)?
ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

a. Samples and/or fractions as appropriate

b. Matrix spikes and matrix spike duplicates (Mass spectra not required)

c. Blanks

ACTION: If any data are missing, take action specified in 3.2 above.

9.4 Are the response factors shown in the Quant Report?

9.5 Is chromatographic performance acceptable with respect to:

Baseline stability?

Resolution?

Peak shape?

Full-scale graph (attenuation)?

Other: ________________________

ACTION: Use professional judgement to determine the acceptability of the data.

9.6 Are the lab-generated standard mass spectra of identified semivolatile compounds present for
each sample?

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

9.8 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

9.9 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.7, 9.8, and 9.9.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.
10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form Is, Part B present; and do listed TICS include scan number or retention time, estimated concentration and "JN" qualifier?

NOTE: Review sampling reports to determine if the lab was required to identify non target analytes (refer to section 7.6.2, page 8270D-21).

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate

b. Blanks

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)?

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the
sample mass spectrum?

10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R."

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and, for soils, sample moisture?
ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration?

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI/Equivalent)

13.1 Is the Initial Calibration Form (Form VI/Equivalent) present and complete for the semivolatile fraction?

ACTION: If any calibration forms or standard row data are missing, take action specified in 3.2 above.

13.2 Are all base neutral or acid RRFs > 0.050?
Check the average RRFs of the four System Performance Check Compounds (SPCCs): N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. These compounds must have average RRFs greater than or equal to 0.05 before running samples and should not show any peak tailing.

**ACTION:** Circle all outliers in red.

**ACTION:** For any target analyte with average RRF < 0.05

1. "R" all non-detects;
2. "J" all positive results.

13.3 Are response factors for base neutral or acid target analytes stable over the concentration range of the calibration (% Relative standard deviation [% RSD] < 20.0%)?

**NOTE:** The % RSD for each individual Calibration Check Compound (CCC, Method 8270D-40 see Table 4) must be less than 30% before analysis can begin. If greater 30%, the lab must clean and recalibrate the instrument.

**CALIBRATION CHECK COMPOUNDS**

<table>
<thead>
<tr>
<th>Base/Neutral Fraction</th>
<th>Acid Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acenaphthene</td>
<td>4-Chloro-3-methylphenol</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>2,4-Dichlorophenol</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>2-Nitrophenol</td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>Phenol</td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>2,4,6-Trichlorophenol</td>
</tr>
</tbody>
</table>

- 25 -
Benzo(a)pyrene

ACTION: If the %RSD for any CCC >30% and no corrective action taken, then "J" qualify all positive hits and "UJ" qualify all non-detects.

ACTION: Circle all outliers in red.

ACTION: If the % RSD is ≥ 20.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detect results for that analyte "R," unusable. Alternatively, the lab should calculate first or second order regression fit of the calibration curve and select the fit which introduces the least amount of error.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

13.4 Did the laboratory calculate the calibration curve by the least squares regression fit? [ ] [ ]

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.) [ ] [ ]

ACTION: Circle Errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.5 Do the target compounds for this SDG include Pesticides? [ ] [ ]
13.6 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%? 

ACTION: If DDT percent breakdown exceeds 20%:

i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

14.0 GC/MS Calibration Verification (Form VII/Equivalent)

14.1 Are the Calibration Verification Forms (Form VII) present and complete for all compounds of interest? 

14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument? 

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis,
call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

14.3 Do any of the SPCCs have an RRF < 0.05? [ ] [ ]

If YES, make a note in data assessment if the lab did not take corrective action specified in section 7.4.4, page 8270D-18. [ ] [ ]

14.4 Do any of the CCCs have a %D between the initial and continuing RRF which exceeds 20.0%? [ ] [ ]

ACTION: If yes, make a note in data assessment.

14.5 Do any semivolatile compounds have a % Difference (%D) between the initial and continuing RRF which exceeds 20.0%? [ ] [ ]

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is above 90%, qualify all non-detects for that analyte as "R", unusable.

14.6 Do any semivolatile compounds have a RRF < 0.05? [ ] [ ]

ACTION: Circle all outliers in red.

ACTION: If RRF < 0.05, qualify as unusable ("R") associated non-detects and "J" associated positive values.

14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more). [ ] [ ]
ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area</th>
<th>LowerLimit</th>
<th>Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

Note: Check Table 5, 8270D-41 for associated analytes.

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.
iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard?  

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Laboratory Control Samples (LCS)

16.1 Were any LCS samples run in order to verify analytes which failed criteria for spike recovery?  

16.2 Did the lab spike LCS sample spiked with the same analytes and the same concentrations as the matrix spike?  

16.3 Were the mean and standard deviation of all analytes within the QC acceptance ranges as shown in Table 6, 8270D-43?  

ACTION: If the recovery of any analyte falls out of the designated range, the analytical results for that compound is suspect and should be qualified "J" in the unspiked samples.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for semivolatile analysis?
ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.
**DataQual**

**Initial Calibration Date:** 12/1/2011

**RRF and %RSD Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>naphthalene</td>
<td>1.155</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>621062</td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>268811</td>
</tr>
<tr>
<td>Conc. of Internal STD</td>
<td>40</td>
</tr>
<tr>
<td>Conc. of Compound</td>
<td>80</td>
</tr>
<tr>
<td>Calculated RRF</td>
<td>1.155</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-methylnaphthalene</td>
<td>7.7</td>
</tr>
</tbody>
</table>

| RRF of STD 1 | 0.696 |
| RRF of STD 2 | 0.74  |
| RRF of STD 3 | 0.747 |
| RRF of STD 4 | 0.744 |
| RRF of STD 5 | 0.799 |
| RRF of STD 6 | 0.879 |
| RRF of STD 7 | 0.741 |
| Calculated % RSD | 7.7   |

**Continuing Calibration File ID:** 12/5/2011

**RRF and %D Calculations:**

<table>
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<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
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<tbody>
<tr>
<td>bis(2-ethylhexyl)phthalate</td>
<td>0.689</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>217885</td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>505725</td>
</tr>
<tr>
<td>Conc. of Internal STD</td>
<td>40</td>
</tr>
<tr>
<td>Conc. of Compound</td>
<td>25</td>
</tr>
<tr>
<td>Calculated RRF</td>
<td>0.689</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>naphthalene</td>
<td>6.8</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Average RRF</td>
<td>1.022</td>
</tr>
<tr>
<td>Calibration Check RRF</td>
<td>0.953</td>
</tr>
<tr>
<td>Calculated % D</td>
<td>6.8</td>
</tr>
</tbody>
</table>
SAMPLE CALCULATION

Sample ID: VWAI-MW05-1111
Standard ID: 12/2/2011
Compound: 2-methylnaphthalene
Concentration: 11 µg/L

<table>
<thead>
<tr>
<th></th>
<th>Water (µg/L)</th>
<th>Soil (µg/Kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>41944</td>
<td></td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>193580</td>
<td></td>
</tr>
<tr>
<td>Conc. of Internal (ng)</td>
<td>40</td>
<td>2</td>
</tr>
<tr>
<td>RRF of Compound</td>
<td>0.764</td>
<td></td>
</tr>
<tr>
<td>Final Volume</td>
<td>1000</td>
<td>1000</td>
</tr>
<tr>
<td>Dilution Factor</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>GPC Factor</td>
<td>NA</td>
<td>1</td>
</tr>
<tr>
<td>Injection Volume</td>
<td>1</td>
<td>1</td>
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<tr>
<td>Weight of Sample</td>
<td>NA</td>
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</tr>
<tr>
<td>Initial Volume of Sample</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td>% Moisture</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Concentration</td>
<td>11.34</td>
<td>#DIV/0!</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>RT of Internal STD</th>
<th>RT of Compound</th>
<th>RRT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td>5.32</td>
<td>6.655</td>
<td>1.251</td>
</tr>
<tr>
<td>Standard</td>
<td>5.319</td>
<td>6.655</td>
<td>1.251</td>
</tr>
</tbody>
</table>
FIELD DUPLICATE SAMPLE SUMMARY

Sample ID: VWAI-MW05-111
Duplicate Sample ID: VWAI-MW05P-111

Water: RPD>75%
Soil: RPD>100%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-methylnaphthalene</td>
<td>11</td>
<td>11</td>
<td>0</td>
</tr>
<tr>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>

* one values below LOD
only values above LOD listed

COMMENTS: No qualifications required.
REPORT NARRATIVE

Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.

Client: CH2M-Hill, Inc.

Project: CTO-0083 Vieques AOC I

Laboratory Workorder / SDG #: K2359

SW846 8270D, SVOA by GC-MS

I. SAMPLE RECEIPT

Several communications with the client regarding samples to analyze and/or cancel are included in the Sample Transmittal section of this report.

II. HOLDING TIMES

A. Sample Preparation:

All samples were prepared within the method-specified holding times.

B. Sample Analysis:

All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed for select semivolatile organic compounds following procedures in laboratory test code: SW846 8270D.

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW3510.

V. INSTRUMENTATION

The following instrumentation was used:
VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Surrogates:

Surrogate standard percent recoveries were within the QC limits with the following exceptions. Please note that the QC acceptance criteria generally allow one surrogate recovery outside of the QC limits per fraction.

VWAI-MW05P-1111 (K2359-03B), recovery is below criteria for Terphenyl-d14 at 47% with criteria of (50-135).

VWAI-MW07-1111 (K2359-15A), recovery is below criteria for Terphenyl-d14 at 49% with criteria of (50-135).

D. Spikes:

1. Laboratory Control Spikes (LCS):

Percent recoveries for lab control samples were within the QC limits.

2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):

Matrix spikes were performed on samples: VWAI-MW02-1111 (K2359-20EMS) and VWAI-MW02-1111 (K2359-20EMSD).

Percent recoveries and replicate RPDs were within the QC limits.

E. Internal Standards:

Internal standard peak areas were within the QC limits.
F. **Dilutions:**

No sample in this SDG required analysis at dilution.

G. **Samples:**

No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: [Signature]

Date: [Date] 12/12/11
This SDG contains GRO results SW-846 method 8015B. Region II validation guidelines were used as applicable, however, the Region has not developed an SOP for this method so these worksheets are used as an alternative.

Laboratory: Spectrum Analytical

**Holding Times**

| Sampling Date: | 11/8-10/11 | 14-day sample holding time was applied based on SW-846 recommendations |
| Received Date: | 11/9-12/11 |
| Analysis Dates: | 11/12/11 |
| Cooler Temp: | 4-5°C |

All sample analysis holding time requirements were met.

**Calibrations**

A five-point calibration curve was analyzed for both the target compound and the surrogate compound. The RFs and %RSDs were calculated and met criteria for both the target compound and the surrogate compound. Continuing calibration standards were analyzed per the method. All %Ds were within QC limits.

**Blank Summary**

Blank qualification guidelines:

- No action is taken if a compound is found in the blank but not in the sample.
- Sample weight, volume or dilution factor must be taken into consideration when applying criteria.
- Apply the same data validation guidelines to any associated method, trip, rinse and field blanks and all associated samples.
- Qualification/Action codes:
  - **U** - The blank contamination concentration is ≤ RL or > RL and sample result is < RL. Result is qualified as U at the RL.
  - **U** - The blank contamination concentration is > RL and sample result is either is > RL but < blank contamination concentration. Result is qualified as U at reported concentration.
  - **NA** - The sample is greater than the RL when the blank contamination concentration is < RL or the sample result is greater than the blank contamination concentration when the blank contamination concentration is > RL.

No contamination was exhibited in the method - no qualifications required. All QC blanks exhibited no contamination.

**Surrogate Recoveries Summary**

All criteria met with.

**Laboratory Control Spike**

All criteria met.
Matrix Spike/Matrix Spike Duplicate Summary

An MS/MSD was submitted for sample VWAI-MW02-1111 - ALL CRITERIA WERE MET.

Field Duplicate Sample Summary

A field duplicate was not submitted for this data package.

Sample ID: VWAI-MW05-1111  Duplicate Sample ID: VWAI-MW05P-1111

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Duplicate Conc.</th>
<th>RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>GRO</td>
<td>0</td>
<td>0</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>

Comments: No qualifications required.

Specific Comments:

Raw data was verified.

Validator Signature: [Signature]

Date: 1/2/12

Vieques CTO-083 AOC I
SDG# K2359
Page 2 of 2
**DataQual**

**Initial Calibration Date:** 5/17/2011

**RF and %RSD Calculations:**

<table>
<thead>
<tr>
<th>Compound Name:</th>
<th>GRO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value:</td>
<td>4.692 E4</td>
</tr>
<tr>
<td>Area of Compound</td>
<td>93849135</td>
</tr>
<tr>
<td>Conc of Compd</td>
<td>2000</td>
</tr>
<tr>
<td>Calculated RRF</td>
<td>46924.57</td>
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</table>

<table>
<thead>
<tr>
<th>Compound Name:</th>
<th>GRO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value:</td>
<td>18.17</td>
</tr>
<tr>
<td>RRF of STD 1</td>
<td>4.670</td>
</tr>
<tr>
<td>RRF of STD 2</td>
<td>4.787</td>
</tr>
<tr>
<td>RRF of STD 3</td>
<td>4.694</td>
</tr>
<tr>
<td>RRF of STD 4</td>
<td>4.692</td>
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<tr>
<td>RRF of STD 5</td>
<td>6.791</td>
</tr>
<tr>
<td>Calculated % RSD</td>
<td>18.17</td>
</tr>
</tbody>
</table>

**Continuing Calibration File ID:** 11/22/2011

**RF and %D Calculations:**

<table>
<thead>
<tr>
<th>Compound Name:</th>
<th>GRO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Value CF:</td>
<td>43.665 E3</td>
</tr>
<tr>
<td>Lab Value %D:</td>
<td>14.8</td>
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<tr>
<td>Area of compound</td>
<td>21832321</td>
</tr>
<tr>
<td>Concentration</td>
<td>500</td>
</tr>
<tr>
<td>Calculated CF</td>
<td>43664.6</td>
</tr>
<tr>
<td>Average CF</td>
<td>51.268</td>
</tr>
<tr>
<td>Calibration Check CF</td>
<td>43.665</td>
</tr>
<tr>
<td>Calculated % D</td>
<td>14.83</td>
</tr>
</tbody>
</table>
REPORT NARRATIVE
Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.
Client: CH2M-Hill, Inc.

Project: CTO-0083 Vieques AOC I

Laboratory Workorder / SDG #: K2359
SW846 8015D GRO, Gasoline Range Organic (GRO) by GC-FID

I. SAMPLE RECEIPT

Several communications with the client regarding samples to analyze and/or cancel are included in the Sample Transmittal section of this report.

II. HOLDING TIMES

A. Sample Preparation:

All samples were prepared within the method-specified holding times.

B. Sample Analysis:

All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 8015D GRO.

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW5030.

V. INSTRUMENTATION

The following instrumentation was used:

Instrument Code: V4
VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

Gasoline Range Organics are calibrated using the average response factor from a GRO component spike. This GRO component spike includes compounds from MTBE through Naphthalene. Samples are integrated from the retention times of MTBE through Naphthalene range inclusive. The laboratory control sample spikes are performed using a gasoline product spike.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Surrogates:

Surrogate standard percent recoveries were within the QC limits.

D. Spikes:

1. Laboratory Control Spikes (LCS):

   Percent recoveries for lab control samples were within the QC limits.

2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):

   Matrix spikes were performed on samples: VWAI-MW02-1111 (K2359-20AMS) and VWAI-MW02-1111 (K2359-20AMSD).

   Percent recoveries and replicate RPDs were within the QC limits.

E. Dilutions:

No sample in this SDG required analysis at dilution.

F. Samples:

No other unusual occurrences were noted during sample analysis.
I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed:______

Date:______12/12/11___________
DataQual

Worksheets – DRO BY 8015D

This SDG contains DRO results SW-846 method 8015D. Region II validation guidelines were used as applicable, however, the Region has not developed an SOP for this method so these worksheets are used as an alternative.

Holding Times

Sampling Date: 11/8-10/11
Received Date: 11/9/11; 11/11/11
Preparation Date: 11/12/11
Analysis Dates: 11/15/11

7-day water or 14 day soil sample holding time was applied based on SW-846 recommendation, cooler temps were/were not acceptable. Appropriate preservation was/was not used.

All sample extraction and analysis holding time requirements were/were not met for all samples in this SDG. Qualifications needed: NONE

Calibrations

6-point calibration curves were analyzed for both the target and the surrogate compound on the instrument(s) used to analyze these samples. The CFs and %RSD’s were calculated and did/did not meet criteria for both the target and surrogate compound. Continuing calibration standards were/were not analyzed per the method. All %Ds were/were not within QC limits in all CCVs. These samples were analyzed on instrument(s) and sequence(s). Qualifications needed:

Blank Summary

Blank qualification guidelines:

• No action is taken if a compound is found in the blank but not in the sample.
• Sample weight, volume or dilution factor must be taken into consideration when applying criteria.
• Apply the same data validation guidelines to any associated method, trip, rinse and field blanks and all associated samples.
• Qualification/Action codes:
  - U - The blank contamination concentration is ≤ RL or > RL and sample result is < RL. Result is qualified as U at the RL.
  - U - The blank contamination concentration is > RL and sample result > RL but < blank contamination concentration. Result is qualified as U at reported concentration.
  - NA The sample is greater than the RL when the blank contamination concentration is < RL or the sample result is greater than the blank contamination concentration when the blank contamination concentration is > RL.

Please note: the LOD = RL for this project.

Blank Contamination and Qualification Summaries

<table>
<thead>
<tr>
<th>Blank ID</th>
<th>Compound</th>
<th>Concentration</th>
<th>LOD</th>
</tr>
</thead>
<tbody>
<tr>
<td>no contamination noted</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Associated samples and required qualifications are noted in the following table.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Q Flag</th>
</tr>
</thead>
<tbody>
<tr>
<td>no qualifications were required</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Surrogate Recoveries Summary

All surrogate recoveries were/were not acceptable in these samples. Qualifications needed: NONE

VIEQUES, CTO-83 AOC I SOILS
SDG SK2359
Page 1 of 2
Matrix Spike/Matrix Spike Duplicate Summary
The MS/MSD pair submitted in this SDG exhibited acceptable/unacceptable recoveries and RPDs. LCS recoveries were not acceptable. Qualifications needed: NONE

Field Duplicate Sample Summary
Sample ID: VWAI-MW05-1111 Duplicate Sample ID: VWAI-MW05P-1111

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Duplicate Conc.</th>
<th>RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>ETPH</td>
<td>1.9</td>
<td>1.9</td>
<td>0%</td>
</tr>
<tr>
<td>ORO</td>
<td>0.54</td>
<td>0.71</td>
<td>27%</td>
</tr>
</tbody>
</table>

Comments: Flag ORO as estimated in both samples (>20% RPD).

Sample Result Verification

Specific Comments:
The samples required manual integration for the target compounds due to the nature of the compounds (multi-component). Positive results were reported in the field water samples. Raw data and quantitation calculations were verified.

Reviewer: [Signature]

Date: 1-10-12

VIEQUES, CTO-83 AOC 1 SOILS
SDG SK2359
**DataQual**

**Initial Calibration Date:** 11/22/11 on F1.1  
**RRF and %RSD Calculations:**

| Compound Name:        | dro C10 TO C28 in Level 2  
| Lab Value:            | 0.9190  
| Area of Compound      | 54809086  
| Conc. of Compound     | 200  
| Area of Internal Standard | 11929317  
| Conc. of Internal Standard | 40  
| Calculated RF         | 0.9189  

| Compound Name:        | DRO (C10-C28)  
| Lab Value:            | 3.83  
| RF of STD 1           | 0.8560  
| RF of STD 2           | 0.9190  
| RF of STD 3           | 0.9280  
| RF of STD 4           | 0.9650  
| RF of STD 5           | 0.9180  
| RF of STD 6           | 0.9430  
| RF of STD 7           |  
| Calculated % RSD      | 3.97  

*DIFERENCES ARE DUE TO ROUNCING OF RESPONSE FACTORS TO 3 SD BY THE LAB. NO IMPACT ON RESULTS.*

**Continuing Calibration File ID:**  
TPH F CCAL L5 100 PPM, 11/29/11, 2024  
**RRF and %D Calculations:**

| Compound Name:        | ORO - C29 TO C40  
| Lab Value:            | 0.922  
| Area of Compound      | 430286026  
| Conc. of Compound     | 1400  
| Area of Internal Standard | 13327694  
| Conc. of Internal Standard | 40  
| Calculated RF         | 0.9224  

| Compound Name:        | ORO  
| Lab Value:            | 1.2  
| Average RF            | 0.9330  
| Calibration Check RF  | 0.9220  
| Calculated % D        | 1.2  


**SAMPLE CALCULATION**

Sample ID: VWAI-MW05-1111  
Standard ID: ICAL, 11/29/11, F1  
Compound: DRO  
Concentration: 1.9 mg/L

<table>
<thead>
<tr>
<th></th>
<th>Water (mg/L)</th>
<th>Soil (mg/Kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>562472983</td>
<td></td>
</tr>
<tr>
<td>RF of Compound</td>
<td>0.928</td>
<td></td>
</tr>
<tr>
<td>Area of Internal Standard</td>
<td>12678236</td>
<td></td>
</tr>
<tr>
<td>Conc. Of Internal Standard</td>
<td>40</td>
<td></td>
</tr>
<tr>
<td>Final Volume</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Dilution Factor</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>GPC Factor</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Injection Volume</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Weight of Sample</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Initial Volume of Sample</td>
<td>1000</td>
<td>NA</td>
</tr>
<tr>
<td>% Solids Factor</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Concentration</td>
<td>.91</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>
REPORT NARRATIVE

Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.

Client: CH2M-Hill, Inc.

Project: CTO-0083 Vieques AOC I

Laboratory Workorder / SDG #: K2359

SW846 8015D TPH, Total Petroleum Hydrocarbons (TPH) by GC-FID

I. SAMPLE RECEIPT

Several communications with the client regarding samples to analyze and/or cancel are included in the Sample Transmittal section of this report.

II. HOLDING TIMES

A. Sample Preparation:

   All samples were prepared within the method-specified holding times.

B. Sample Analysis:

   All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 8015D TPH.

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW3510.

V. INSTRUMENTATION

The following instrumentation was used:

Instrument Code: F1
Instrument Type: GC-FID
VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

Calibrations met the Method/SOP acceptance criteria. Diesel Range Organics (DRO) are quantified using the average response factor from C9 - C28 hydrocarbon standards. Oil range organics (ORO) are quantified using the average response factor from C29 - C40 hydrocarbon standards. Continuing calibration verifications are evaluated by comparison of the average response for the individual C9 through C28 peaks (for DRO, or C29 - C 40 for ORO) to the average from the initial calibration. Samples are integrated from the retention time of C9 through C28 (for DRO or C29 - C40 for ORO) inclusive. The laboratory control sample spikes are performed using a diesel fuel product spike.

Please note that the analyte DRO (C9 - C28 range hydrocarbons) are reported as “Extractable Total Petroleum Hydrocarbons” on the data sheets, while ORO (C29 - C40) are reported as “Oil Range Organics” on the data sheets. These results are as described above, with “Extractable Total Petroleum Hydrocarbons” including only the C9 - C28 range organics.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Surrogates:

Surrogate standard percent recoveries were within the QC limits.

D. Spikes:

1. Laboratory Control Spikes (LCS):

Percent recoveries for lab control samples were within the QC limits. Please note that laboratory QC spikes appear in the DRO range only, not in the ORO range.

2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):
Matrix spikes were performed on samples: VWAI-MW02-1111 (K2359-20EMS) and VWAI-MW02-1111 (K2359-20EMSD).

Percent recoveries were within the QC limits.

Replicate RPDs were within the advisory QC limits.

E. Dilutions:

No sample in this SDG required analysis at dilution.

F. Samples:

No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: [Signature]

Date: [Date] 12/9/11
### Standard Operating Procedure

**USEPA Region 2**

**Evaluation of Metals Data for the Contract Laboratory Program**

**Data Assessment and Contract Compliance Review**

<table>
<thead>
<tr>
<th>SOP: HW-2</th>
<th>Revision 13</th>
<th>Appendix A.1</th>
<th>Sept. 2006</th>
</tr>
</thead>
</table>

#### A.1.1 Contract Compliance Screening Report
- Present?
- **ACTION:** If no, contact RSCC/PO.

#### A.1.2 Record of Communication (from RSCC)
- Present?
- **ACTION:** If no, request from the RSCC.

#### A.1.3 Sampling Trip Report
- Present and complete?
- **ACTION:** If no, contact RSCC/PO.

#### A.1.4 Chain of Custody/Sample Traffic Report
- Present?
- **ACTION:** If no, contact RSCC/WAM/PO.

#### A.1.5 Cover Page
- Present?
- Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?
- Do the sample identification numbers on the Cover Page agree with sample identification numbers on:
  - **(a) Traffic Report Sheet?**

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Standard Operating Procedure
USEPA Region 2
Evaluation of Metals Data for the Contract Laboratory Program
Data Assessment and Contract Compliance Review

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(b) Form 1's?

Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?

[ ] [ ] [ ]

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 **SDG Narrative, DC-1 & DC-2 Form**

Is the SDG Narrative present?

[ ] [ ] [ ]

Is Sample Log-In Sheet (Form DC-1) present and complete?

[ ] [ ] [ ]

Is Complete SDG inventory Sheet (Form DC-2) present and complete?

[ ] [ ] [ ]

**ACTION:**
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

A.1.7 **Form I to XV**

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name?

[ ] [ ] [ ]

Laboratory Code?

[ ] [ ] [ ]

RAS/Non-RAS Case No.?

[ ] [ ] [ ]

SDG No.

[ ] [ ] [ ]
REPORT NARRATIVE

Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.

Client : CH2M-Hill, Inc.

Project: CTO-0083 Vieques AOC I

Laboratory Workorder / SDG #: K2359

SW846 6010C

I. SAMPLE RECEIPT

Several communications with the client regarding samples to analyze and/or cancel are included in the Sample Transmittal section of this report.

II. HOLDING TIMES

A. Sample Preparation:

All samples were prepared within the method-specified holding times.

B. Sample Analysis:

All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed for Iron and Manganese only following procedures in laboratory test code: SW846 6010C

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW3005A

V. INSTRUMENTATION

The following instrumentation was used to perform
Instrument Code: OPTIMA3
Instrument Type: ICP
Description: Optima ICP-OES
Manufacturer: Perkin-Elmer
Model: 4300 DV

VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Laboratory Control Spikes (LCS):

Percent recoveries for laboratory control samples were within the QC limits.

D. Samples:

No unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

[Signature]

Signed: ________________

Date: ___12/9/11________________
Contract No.?

**ACTION:**
If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

A.1.7.2 After comparing values on Forms I-IX against the raw data, do any computation/transcription errors exceed 10% of the reported values on the Forms for:

(a) all analytes analyzed by ICP-AES? [ 

(b) all analytes analyzed by ICP-MS? [ 

(c) Mercury? [ 

(d) Cyanide? [ 

**ACTION:**
If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 **Raw Data**
Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 **Digestion/Distillation Log**

Digestion Log for ICP-AES (Form XII) present? [ 

Digestion Log for ICP-MS (Form XII) present? [ 

Digestion Log for mercury (Form XII) present? [ 

Distillation Log for cyanide (Form XII) present? [ 

Are pH values for metals and...
cyanide reported for each aqueous sample?

Are percent solids calculations present for soils/sediments?

Are preparation dates present on the sample preparation logs/bench sheets?

**NOTE:**
Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.

A.1.8.2 Is the analytical instrument real-time printouts present for:

ICP-AES?

ICP-MS?

Mercury?

Cyanide?

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:

Legible?

Properly labeled?

Are all field samples, QC samples and field QC samples present on:

Digestion/Distillation log?

Instrument Printouts?

**ACTION:**
If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.
A.1.9 Technical Holding Times: (Aqueous and soil samples)
(Examine sample Traffic Reports and digestion/distillation logs to determine the holding time from the sample collection date to the sample preparation data.)

A.1.9.1 Cyanide distillation (14 days) exceeded?  
Mercury analysis (28 days) exceeded?  
Other Metals analysis (180 days) exceeded?  

ACTION:  
If yes, reject (R) and red-line non-detects and flag as estimated (J) results ≥ MDL even if sample(s) was preserved properly.

NOTE:  
In addition to qualifying the data, a list of all samples and analytes which exceeded the holding times must be prepared. Report for each sample the number of days that were exceeded. (Subtract the sample collection date from the sample preparation date). Attach this list to the data review narrative.

A.1.9.2 Is pH of aqueous samples for:
Metals Analysis ≤ 2?  
Cyanide Analysis ≥ 12?

ACTION:  
If no for any of the above, flag non-detects as "R" and detects as "J".

A.1.9.3 Is the cooler temperature ≤ 10°C?

ACTION:  
If cooler temperature is >10°C, flag non-detects as "UJ" and detects as "J".

A.1.10 Final Data Correctness - Form I
A.1.10.1 Are Form I's for all samples
SAMPLE CALCULATION

EPA SAMPLE ID: VWAI-MW03-1111
COMPOUND: Manganese
CONCENTRATION: 1350 µg/L
%Solids – NA

Raw Data result: 1.3545 mg/L

1.3545 mg/L (1000 µg/1 mg) = 1354.5 µg/L

FIELD DUPLICATE SAMPLE SUMMARY

Note: All reported results are noted in the table below because the client requested that the MDL be used as reporting limit instead of the RL for this project. RPDs or absolute differences were calculated based on Region II guidelines: if results are >5X RL RPD is calculated, if results are <5X RL the absolute difference is calculated. Flags are applied to field duplicate pair only as follows: For RPD values - RPD ≥ 35% but <120% results are J, RPD >120%, results are R. For absolute difference values - ≥/+ 2X RL results are J, ≥/+ 4X RL results are R.

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>none</th>
<th>Duplicate Sample ID:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Analyte</td>
<td>Sample Conc.</td>
<td>Duplicate Conc.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments: No qualifications required.

<table>
<thead>
<tr>
<th>Sample ID:</th>
<th>none</th>
<th>Duplicate Sample ID:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Analyte</td>
<td>Sample Conc.</td>
<td>Duplicate Conc.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments: No qualifications required.

Reviewer: ___________________________ Date: 1/14/12
A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

Is the calculation error less than 10% of the correct result? [ ]

Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)? [ ]

Are results on Form I's reported by correct significant figures? [ ]

Are soil sample results on Form I's corrected for percent solids? [ ]

Are all "less than MDL" values reported by the CRQLs and coded with "U"? [ ]

Are values less than the CRQLs but greater than or equal to the MDLs flagged with "J"? [ ]

Are appropriate contractual quality control and Method qualifiers used?

ACTION:
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3 Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data? [ ]

Was a brief physical description
of the samples before and after digestion given on the Form I's?

Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I?

**ACTION:**
If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11 **Initial Calibration**

A.1.11.1 Is a record of at least 2 point (A blank and a standard) calibration present for ICP-AES analysis?

A.1.11.2 Is a record of at least 2 point (a blank and a standard) calibration present for ICP-MS analysis?

A.1.11.3 Is a record of at least 5 point calibration (a blank & 4 standards) present for Hg analysis?

A.1.11.4 Is a record of at least 4 point calibration (a blank & 4 standards) present for cyanide?

**ACTION:**
If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).

Is one initial calibration standard at the CRQL level for cyanide and mercury?

**ACTION:**
If no, write in the Contract Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11.2 Is the curve correlation coefficient ≥ 0.995 for:
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Mercury Analysis? [ ] [ ] [ ]

Cyanide Analysis? [ ] [ ] [ ]

ICP-AES (more than 2 point Calib.)? [ ] [ ] [ ]

ICP-MS (more than 2 point calib.)? [ ] [ ] [ ]

**ACTION:**
If no, qualify the associated sample results ≥ MDL as estimated “J” and
non-detects as “UJ”.

**NOTE:**
The correlation coefficient shall be calculated by the data validator
using standard concentrations and the corresponding instrument response (e.g.
absorbance, peak area, peak height, etc.).

A.1.12 Initial and Continuing Calibration Verification- Form IIA

A.1.12.1 Present and complete for every metal and cyanide? [ ] [ ] [ ]

Present and complete for ICP-AES and ICP-MS when both these methods
were used for the same analyte? [ ] [ ] [ ]

**ACTION:**
If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO
for re-submittal from the laboratory.

A.1.12.2 Was a Continuing Calibration Verification performed every 10 samples or every 2 hours
whichever is more frequent? [ ] [ ] [ ]

**ACTION:**
If no for any of the above, write in the Contract-Problem/Non-Compliance
Section of the Data Review Narrative.

A.1.12.3 Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples? [ ] [ ] [ ]
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**ACTION:**
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative and qualify results \( \geq \) MDL as estimated (J).

A.1.12.2
Circle on each Form II A all percent recoveries that are outside the contract windows.

Are ICV/CCVs within control limits for:

- Metals - 90-110%R?
  - [ ]
  - [ ]
  - [ ]

- Hg - 80-120%R?
  - [ ]
  - [ ]
  - [ ]

- Cyanide - 85-115%R?
  - [ ]
  - [ ]
  - [ ]

**ACTION:**
If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89%(65-79% for Hg; 70-84% for CN). Qualify only positive results (\( \geq \) MDL) as "J" if the ICV/CCV %R is between 111-125%(121-135% for Hg; 116-130% for CN). Reject (R) and red-line only detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detects) if the recovery is less than 75%(65% for Hg; 70% for CN).

**NOTE:**
For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

A.1.12.3
Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)?
  - [ ]
  - [ ]
  - [ ]

**ACTION:**
If no, Qualify all cyanide results \( \geq \) MDL as "J".

A.1.13  **CRQI Standard Analysis - Form IIB**

A.1.13.1  For each ICP-AES run, was a CRI

**-22-**
(CRQL or MDL when MDL > CRQL) standard analyzed?
(Note: CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)

For each ICP-MS run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis?

For each mercury run, was a CRQL standard analyzed?

For each cyanide run, was a CRQL standard analyzed?

ACTION:
If no for any of the above, write this deficiency in the Contract Problems/Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL) as J and non-detects UJ.

The affected ranges are:
- ICP-AES Analysis - *True Value ± CRQL
- ICP-MS Analysis - *True Value ± CRQL
- Mercury Analysis - *True Value ± CRQL
- Cyanide Analysis - *True Value ± CRQL
* True value of the CRQL Standard

A.1.13.2 Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis?

ACTION:
If no, write in the Contract Problem/Non-Compliance Section of the "Data Review Narrative".

A.1.13.3 Circle on each Form IIB all percent recoveries that are outside the acceptance windows.
Is the CRQL standard within control limits for:

Metals (ICP-AES/ICP-MS) - 70 - 130%?

Mercury - 70 - 130%?

Cyanide - 70 - 130%?

**ACTION:**
If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-59%. Flag (J) only detects <2xCRQL if the recovery is between 131% and ≤180%. If the recovery is less than 150%, reject (R) and red-line non-detects and detects < 2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detects <2xCRQL and flag (J) detects > 2xCRQL but < ICV/CCV if the recovery is > 180%.

**NOTE:**
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard.
2. Flag (J) or reject (R) only the final sample results on Form I's when Sample raw data are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

A.1.14 *Initial and Continuing Calibration Blanks - Form III*

A.1.14.1 Present and complete for all the instruments used for the metals and cyanide analyses?

- [ ] Yes
- [ ] No
- [ ] Not Applicable

Was an initial Calibration Blank analyzed after ICV?

- [ ] Yes
- [ ] No
- [ ] Not Applicable

Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?

- [ ] Yes
- [ ] No
- [ ] Not Applicable

Were the IC8 & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by Lab did not run CRQL std. of...
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using MDLs from direct analysis (Preparation Method "NP1")?
(Check Form III against the raw data)

**ACTION:**
If no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".

A.1.14.2 Circle with red pencil on each Form III all Calib. Blank values that are:

- > MDL but ≤ CRQL
- > CRQL

A.1.14.2.1 When MDL < CRQL, is any Calib. Blank value > MDL but ≤ CRQL?

**ACTION:**
If yes, change sample results > MDL but ≤ CRQL to the CRQL with a "U". Do not qualify non-detects.

A.1.14.2.2 When MDL < CRQL, is any Calib. Blank value > CRQL?

**ACTION:**
If yes, reject (R) and red line the associated sample results > CRQL but < ICB/CCB Blank Result. Flag as "J" detects > ICB/CCB blank value but < 10xICB/CCB value. Change the sample results > MDL but ≤ the CRQL to CRQL with a "U".

A.1.14.2.3 Is any Calibration Blank value below the negative CRQL?

**ACTION:**
If yes, flag (J) as estimated all associated sample results > CRQL but <10xCRQL.

**NOTE:**
1. For ICB that does not meet the technical QC Criteria, apply the action to all samples.
2. For CCBs that do not meet the technical QC criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

A.1.15 Preparation Blank - FORM III

NOTE: The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1 Was one Preparation Blank prepared with and analyzed for:

Each Sample Delivery Group (SDG)? [✓] ___ ___

Each batch of the SDG samples digested/distilled? [✓] ___ ___

Each matrix type? [✓] ___ ___

All instruments used for metals and cyanide analyses? [✓] ___ ___

ACTION:
If no for any of the above, flag as estimated (J) all the associated positive data <10xMDL for which the Preparation Blank was not analyzed.

NOTE:
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated (J), but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

≥ MDL but < CRQL, and

> CRQL

A.1.15.2.1 When MDL < CRQL, is any preparation blank value ≥ MDL but < CRQL?

[✓] ___

ACTION:
If yes, change sample result ≥ MDL
but ≤ CRQL to CRQL with a "U".

A.1.15.2.2 When the MDL ≤ CRQL, is any Preparation Blank value greater than its CRQL?  

If yes, is the Prep. Blank value greater than the value of the associated Field Blank collected and analyzed with the SDG samples?

If yes, is the lowest concentration of that analyte in the associated samples less than 10 times the Preparation Blank value?

**ACTION:**
If yes, reject (R) and red-line all associated sample results greater than the CRQL but less than the Prep.Blank value. Flag as "J" detects > Prep. Blank value but <10xPrep.Blank. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.

If the Prep. Blank value is less than the same analyte value in the Field Blank, do not qualify the sample results due to the Prep. Blank criteria.

**NOTE:**
Convert soil sample result to mg/Kg on wet weight basis to compare with the soil Prep. Blank result on Form III.

A.1.15.2.3 Is the Prep. Blank concentration below the negative CRQL?

**ACTION:**
If yes, flag (J) all associated sample results less than 10xCRQL. Qualify non-detects as estimated (UJ).

A.1.15.2.4 When the MDL is greater than the CRQL, is the preparation blank concentration on Form III greater than two times the MDL?

**ACTION:**
If yes, reject (\(\checkmark\)) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.

A.1.16 ICP-AES/ICP-MS Interference Check Sample (ICS) - Form IV
NOTE: Not required for CN, Hg, Al, Ca, Fe and Mg.

A.1.16.1 Present and complete?

Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples?

Was ICS analyzed at the beginning of the ICP-MS analytical run?

**ACTION:**
If no, flag as estimated (J) all sample results.

A.1.16.2 ICP-AES Method

A.1.16.2.1 ICSA Solution:
For ICP-AES, are the ICSA "Found" analyte values within the control limits \(\pm 1\) of CRQL of the true/established mean value?

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV?

**ACTION:**
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results \(\geq\) MDL
for which the ICSA “Found” value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA “Found” value is less than (True value-CRQL), flag non-detects as “UJ” and detects as “J”.

A.1.16.2.3 ICSAB Solution
For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value? [ ]  [ ]  [ ]

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (μg/L or mg/kg) greater than or equal to its respective concentration in the ICSAB Solution on Form IV? [ ]  [ ]  [ ]

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79%, qualify sample results ≥ MDL as “J” and non-detects as “UJ”. Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.

A.1.16.3 ICP-MS Method

A.1.16.3.1 ICSA Solution:
For ICP-MS, are the ICSA “Found” analyte values within the control limits of ±CRQL of the true/established mean value? [ ]  [ ]  [ ]

ACTION:
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated only sample results ≥ MDL if the ICSA “Found” value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA “Found” value is less than (True value-CRQL), flag the associated sample detects as “J” and non-detects as “UJ”.

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A.1.16.3.3 ICSAB Solution
For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater? [ ] [ ] [X]

**ACTION:**
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but < 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only detects (≥ MDL).

A.1.17 Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A
**Note:** Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only)

A.1.17.1 Was Matrix Spike analysis performed:

For each matrix type? [ ] [X] [ ]

For each SDG? [ ] [X] [ ]

On one of the SDG samples? [ ] [X] [ ]

For each concentration range (i.e., low, med., high)? [ ] [X] [ ]

For each analytical Method (ICP-AES, ICP-MS, Hg, CN) used? [ ] [X] [ ]

Was a spiked sample prepared and analyzed with the SDG samples? [ ] [X] [ ]

**ACTION:**
If no for any of the above, flag as estimated (J) all the positive data for which a spiked sample was not analyzed.

**NOTE:**
If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.

**Lab did not perform MSI/MSD (client did not request). Tall + results for both Fe & Mn.**
A.1.17.2 Was a field blank or PE sample used for the spiked sample analysis?

ACTION:
If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.

A.1.17.3 Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.

Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?

NOTE:
Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.

Are results outside the control limits (75-125%) flagged with Lab Qualifier "N" on Form I's and Form VA?

A.1.17.4 Aqueous

Are any spike recoveries:

(a) less than 30%?
(b) between 30-74%?
(c) between 126-150%?
(d) greater than 150%?

ACTION:
If the matrix spike recovery is less than 30%, reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as "J" and non-detects
as "UJ". If between 126-150%, flag (J) all data ≥ MDL as "J". If greater than 150%, reject (R) and red-line all associated data ≥ MDL.

(NOTE: Replace "N" with "J", "R" as appropriate.)

A.1.17.5 Soil/Sediment

Are any spike recoveries:

(a) less than 10%? ___ [ ]
(b) between 10-74%? ___ [ ]
(c) between 126-200%? ___ [ ]
(d) greater than 200%? ___ [ ]

ACTION:
If yes for any of the above, proceed as follows:

If the matrix spike recovery is less than 10%, reject (R) and red-line all associated data (detects & non-detects);
if between 10-74%, qualify all associated data ≥ MDL as "J" and non-detects as "UJ";
if between 126-200%, flag (J) all associated data ≥ MDL as "J" If greater than 200%, reject (R) and red-line all associated data ≥ MDL.

(NOTE: Replace "N" with "J" or "R" as appropriate.)

A.1.18 Lab Duplicates) - Form VI

A.1.18.1 Was the lab duplicate analysis performed:

For each SDG? [ ]
On one of the SDG samples? [ ]
For each matrix type? [ ]
For each concentration range (low or med.)? [ ]
For each analytical Method (ICP-AES/ICP-MS, Hg, CN) Used? [ ]
Was a lab duplicate prepared and analyzed with the SDG samples? [ ]

Client didn't request
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YES NO N/A

ACTION:
If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.

NOTE:
If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

A.1.18.2 Was a Field Blank or PE sample used for the Lab Duplicate analysis? [ ]

ACTION:
If yes, flag as estimated (J) all SDG sample results (detects & non-detects) for which Field Blank or PE sample was used for duplicate analysis.

A.1.18.3 Circle on each Form VI all values that are:

RPD > 20%, or

Absolute Difference > CRQL

Are all values within control limits (RPD ≤ 20% or absolute difference ≤ ±CRQL)? [ ]

If no, are all results outside the control limits flagged with an "**" (Lab Qualifier) on Form VI and on all Form I’s? [ ]

ACTION:
If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.

NOTE:
The laboratory is not required to report on Form VI the RPD when both values are non-detects.

A.1.18.4 Aqueous

A.1.18.4.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL).
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is any RPD > 20% but < 100%?  ___  [___]  ___

is any RPD ≥ 100%?  ___  [___]  ___

ACTION:
If the RPD is > 20% but < 100%,
flag (J) as estimated the associated
sample data ≥ CRQL. If the RPD is
≥ 100%, reject (R) and red-line the
associated sample data ≥ CRQL.

(NOTE: Replace “*” with “J” or “R” as appropriate.)

A.1.18.4.2 When the sample and/or duplicate value
<5xCRL (substitute MDL for CRQL when MDL > CRQL),
is the absolute difference between sample
and duplicate values:

> ± CRQL?  ___  [___]  ___

> ± 2xCRQL?  ___  [___]  ___

ACTION:
If the absolute difference is > CRQL,
flag as estimated all the associated
sample results ≥ MDL but < 5xCRQL as “J”
and non-detects as “UJ”. If the absolute
difference is > 2xCRQL, reject (R) and
red-line all the associated non-detects
and detects ≥ MDL but < 5xCRQL.

NOTE:
1. Replace “*” with “J”, “UJ” or “R” as appropriate.
2. If one value is >CRQL and the other value is non-detect,
calculate the absolute difference between the value > CRQL
and the MDL, and use this difference to qualify sample results.

A.1.18.5 Soil/Sediment

A.1.18.5.1 When sample and duplicate values
are both ≥ 5xCRQL (substitute MDL for
CRQL when MDL > CRQL),
is any RPD ≥ 35% but < 120%?  ___  [___]  ___

is any RPD ≥ 120%?  ___  [___]  ___

ACTION:
If the RPD is ≥ 35% and < 120%, flag
(J) as estimated the associated sample
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YES NO N/A

data > CRQL. If the RPD is ≥ 120%, reject
(R) and red-line the associated sample
data ≥ CRQL.

A.1.18.5.2 When the sample and/or duplicate value
<5xCRQL(substitute MDL for CRQL when MDL > CRQL),
is the absolute difference between sample
and duplicate:

> ± 2 x CRQL?

> ± 4 x CRQL

ACTION:
If the absolute difference is > 2 x CRQL,
flag all the associated sample results ≥ MDL
but < 5xCRQL as "J" and non-detects as "UJ".
If the absolute difference is > 4xCRQL, reject
(R) and red-line all the associated non-detects
and detects ≥ MDL but < 5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.
2. If one value is >CRQL and the other value is non-detect,
calculate the absolute difference between the value ≥ CRQL
and the MDL, and use this difference to qualify sample results.

A.1.19

Field Duplicates

Aqueous Field Duplicates

A.1.19.1 Was an aqueous Field Duplicate pair
collected and analyzed?
(Check Sampling Trip Report)

ACTION:
If yes, prepare a Form (Appendix A.4) for each
aqueous Field Duplicate pair. Report the sample
and Field Duplicate results on Appendix A.4 from
their respective Form I's. Calculate and report RPD
on Appendix A.4 when sample and its Field Duplicate
values are both > 5xCRQL. Calculate and report the
absolute difference on Appendix A.4 when at least one
value (sample or duplicate) is <5xCRQL. Evaluate the
aqueous Field Duplicate analysis in accordance with the
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QC criteria stated in Sections A.1.19.2 and A.1.19.3.

NOTE:
1. Do not transfer "" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

RPD ≥ 20%  or

Difference > ± CRQL

When sample and duplicate values are both >5xCRQL (substitute MDL for CRQL when MDL > CRQL),

- is any RPD ≥ 20%? ___ [___] ✓
- is any RPD ≥ 100%? ___ [___] ✓

ACTION:
If the RPD is >20% but < 100%, flag (J) only the associated sample and its Field Duplicate results ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line only the associated sample and its Field Duplicate result ≥ CRQL.

A.1.19.3 When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

- > ± CRQL? ___ [___] ✓
- > ± 2 x CRQL? ___ [___] ✓

ACTION:
If the absolute difference is > CRQL, flag detects ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the difference is > 2xCRQL, reject (R) and red-line non-detects.
and results > MDL but <5xCRQL of the sample and its Field Duplicate.

**Soil/Sediment Field Duplicates**

A.1.19.4 Was a soil field duplicate pair collected and analyzed? (Check Sampling Trip Report)  

[___] [___] [___]

**ACTION:**

If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than 5xCRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is < 5xCRQL. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

**NOTE:**

1. Do not transfer "**" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and apply the criteria to qualify the results.

A.1.19.5 Circle on each Appendix A.4 all values that have:

RPD ≥ 35%, or Difference > ± 2xCRQL

When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?  

[___] [___] [___]

is any RPD > 120%?  

[___] [___] [___]

**ACTION:**

If the RPD is ≥ 35% but < 120%,
flag only the associated sample
and its Field Duplicate results
≥ CRQL as "J". If the RPD is ≥ 120%,
reject (R) and red-line only the sample
and its Field Duplicate results ≥ CRQL.

A.1.19.6 When the sample and/or duplicate value(s)
<5xCRQL (substitute MDL for CRQL when MDL > CRQL),
is the absolute difference between sample
and Field Duplicate:

> ± 2 x CRQL?

> ± 4 x CRQL?

ACTION:
If the absolute difference is > 2xCRQL, flag
Sample and its Field Duplicate results ≥ MDL
but <5xCRQL as "J" and non-detects as "UJ".
If the difference is >4xCRQL, reject (R) and
red-line non-detects and detects ≥ MDL but
<5xCRQL of the sample and its Field Duplicate.

A.1.20 Laboratory Control Sample (LCS)- Form VII

A.1.20.1 Was one LCS prepared and analyzed for:

Each SDG?

Each matrix type?

Each batch samples digested/distilled?
For each Method(ICP-AES, ICP-MS, Hg, CN)
used?

Was an LCS prepared and analyzed with
the samples?

ACTION:
If no for any of the above, prepare
Telephone Record Log and contact
CLP PO or TOPO for submittal of the
LCS results. Flag (J) as estimated all
the data for which an LCS was not
analyzed.

NOTE:
If only one LCS was analyzed for
A.1.20.2 Aqueous LCS

Circle on each Form VII the LCS percent recoveries outside control limits 80-120%.

NOTE: 1. Use digested ICV as LCS for aqueous mercury
      2. Use distilled ICV as LCS for aqueous cyanide

Is any LCS recovery:

Less than 50%?  __  [ ]  ___
Between 50% and 79%?  __  [ ]  ___
Between 121% and 150%?  __  [ ]  ___
Greater than 150%?  __  [ ]  ___

ACTION:
If the LCS recovery is less than 50%, reject (R) and red-line all associated sample data (detects & non-detects); for a recovery between 50-79%, flag detects as "J" all non-detects as "UJ". If the LCS recovery is between 121-150%, flag only detects as "J". If the recovery is greater than 150%, reject (R) and red-line all detects.

A.1.20.3 Solid LCS

If an analyte's MDL is equal to or greater than the true value of LCS, disregard the "Action" below for that analyte even though the LCS is out of control limits.

Is the LCS "Found" value greater than the Upper Control Limit reported on Form VII?  __  [ ]  ___

ACTION:
If yes, flag (J) all the associated
detects ≥ MDL as estimated (J).

Is the LCS "Found" value lower
than the Lower Control Limit
reported on Form VII?

ACTION:
If yes, flag detects as "J" and
non-detects as "UJ".

A.1.21 ICP-AES/ICP-MS Serial Dilution - Form VIII
NOTE: Serial dilution analysis is required only
when the initial concentration is equal to or
greater than 50 x MDL.

A.1.21.1 Was a Serial Dilution analysis
performed:

For each SDG?

On one of the SDG samples?

For each matrix type?

For each concentration range
(low or med.)?

Was a Serial Dilution sample
analyzed with the SDG samples?

ACTION:
If no for any of the above, flag
as estimated (J) detects ≥ MDL of
all the SDG samples for which the
ICP Serial Dilution Analysis was
not performed.

A.1.21.2 Was a Field Blank or PE sample used
for the Serial Dilution Analysis?

ACTION:
If yes, flag as estimated (J) detects
≥ MDL of all the SDG samples

A.1.21.3 Circle on Form VIII the Percent Differences
(%) between sample results and its dilution
results that are outside the control limits ± 10%
when initial concentrations ≥ 50 x MDLs.

Are results outside the control limits flagged with an "E" (Lab Qualifier) on Form VIII and all Form I's?  

ACTION:
If no, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.21.4 Are any %D values:
> 10%?  
≥ 100%?  

ACTION:
If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose raw data > MDL; if the %D is ≥ 100%, reject (R) and red-line all associated samples with raw data ≥ MDL.

(NOTE: Replace "E" with "J" or "R" as appropriate.)

A.1.22 Total/Dissolved or Inorganic/Total Analytes

A.1.22.1 Were any analyses performed for dissolved as well as total analytes on the same sample(s)?  
Were any analyses performed for inorganic as well as total analytes on the same sample(s)?

ACTION:
If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic) and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:

(1) The dissolved (or inorganic) concentration is greater than total concentration, and
(2) greater than or equal to 5xMDL.

A.1.22.2 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%?
A.1.22.3 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 50%?

ACTION:
If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23 Field Blank - Form I

NOTE: Designate "Field Blank" as such on Form I

A.1.23.1 Was a Field/Rinsate Bank collected and analyzed with the SDG samples?

If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL (or 2xMDL when MDL > CRQL)?

If yes, circle the Field Blank value on Form I that is greater than the CRQL, (or 2 x MDL when MDL > CRQL).

Is any Field Blank value greater than CRQL also greater than the Preparation Blank value?

If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria?

ACTION:
If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results) greater than the CRQL but less than the Field Blank value. Reject on Form I’s the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as “J” detects between the Field Blank value and 10xField Blank value. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.

If the Field Blank value is less than the
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Prep. Blank value, do not qualify the sample results due to the Field Blank criteria.

NOTE:
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

A.1.24  Verification of Instrumental Parameters - Form IX, XA, XB, XI

A.1.24.1 Is verification report present for:

Method Detection Limits (Form IX - Annually)?

ICP-AES Interelement Correction Factors (Form XA & XB - Quarterly)?

ICP-AES & ICP-MS Linear Ranges (Form XI - Quarterly)?

ACTION:
If no, contact CLP PO/TOPO for submittal from the laboratory.

A.1.24.2 Method Detection Limits - Form IX

A.1.24.2.1 Are MDLs present on Form IX for:

All the analytes?

All the instruments used?

Digested and undigested samples and Calib.Blanks?

ICP-AES and ICP-MS when both instruments are used for the same analyte?

ACTION:
If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than % CRQL.

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A.1.24.2.2 Is MDL greater than the CRQL for any analyte?

[YES] [NO] [N/A]

If yes, is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?

[___] [___] [___]

ACTION:
If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.

A.1.24.3 Linear Ranges - Form XI

A.1.24.3.1 Was any sample result higher than the high linear range for ICP-AES or ICP-MS?

[___] [___] [___]

Was any sample result higher than the highest calibration standard for mercury or cyanide?

[___] [___] [___]

If yes for any of the above, was the sample diluted to obtain the result reported on Form I?

[___] [___] [___]

ACTION:
If no, flag (J) as estimated the affected detects (≥ MDL) reported on Form I.

A.1.25 ICP-MS Tune Analysis - Form XIV

A.1.25.1 Was the ICP-MS instrument tuned prior to calibration?

[___] [___] [___]

ACTION:
If no, reject (R) and red-line all sample data for which tuning was not performed.

A.1.25.2 Was the tuning solution analyzed or scanned at least five times consecutively?

[___] [___] [___]

Were all the required isotopes spanning the analytical range present in the tuning solution?

[___] [___] [___]

Was the mass resolution within 185...
<table>
<thead>
<tr>
<th>SOP: HW-2 Revision 13</th>
<th>Appendix A.1</th>
<th>Sept. 2006</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
</table>

0.1 amu for each isotope in the tuning solution? 

Was %RSD less than 5% for each isotope of each analyte in the tuning solution? 

**ACTION:**
If no for any of the above, qualify all results ≥ MDL associated with that Tune as estimated “J”, and all non-detects associated with that Tune as “UJ”.

---

<table>
<thead>
<tr>
<th>A.1.26 ICP-MS Internal Standards - Form XV</th>
</tr>
</thead>
</table>

Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)? 

Were all the target analyze masses bracketed by the masses of the five internal standards? 

**ACTION:**
If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.

---

<table>
<thead>
<tr>
<th>A.1.26.2 Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank?</th>
</tr>
</thead>
</table>

If no, was the original sample diluted two fold. Internal Standard added and the sample re-analyzed? 

Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)? 

**ACTION:**
If no for any of the above, flag detects as “J” and non-detects “UJ” of all the analytes with atomic masses between the atomic mass of the internal standard lighter...
than the affected internal standard, and the atomic mass of the internal standard heavier than the affected internal standard.

A.1.27 Percent Solids of Sediments

A.1.27.1 Are percent solids in sediment(s):

< 50%? __ [___] ___

ACTION:
If yes, qualify as estimated (J) all detects and non-detects of a sample that has percent solids less than 50% (i.e., moisture content greater than 50%).

NOTE:
Flag(J) only the sample results that were not previously flagged due to other QC criteria.

Inorganic Data Review Narrative

Case# __________ Site: __________ Matrix: Soil ___
SDG# __________ Lab: __________ Water ___
Sampling Team: __________ Reviewer: __________ Other ___

A.2.1 Data Validation Flags:
The following flags may have been applied in red by the data validator and must be considered by the data user.

J - This flag indicates the result qualified as estimated

R and Red-Line - A red-line drawn through a sample result indicates unusable value. The red-lined data are known to contain significant errors based on documented information and must not be used by the data user.

U - This data validation qualifier is applied to sample results > MDL when associated blank is contaminated

Fully Usable Data - The results that do not carry "J" or "red-line" are fully usable.

A.2.2 Laboratory Qualifiers:
The CLP laboratory applies a contractual qualifier on all
July 27, 2012
SDG# LI093, Spectrum Analytical, Inc.
Vieques Island, Puerto Rico-CTO-083

Dear Ms. Ott,

The following Data Validation report is provided as requested for the parameters noted in the table below for SDG # LI093. The data validation was performed in accordance with the SW-846 methods utilized by the laboratory, the Region II Standard Operating Procedures for the Validation of Organic Data Acquired Using SW-846 Methods (8260B-Rev 2, August 2008- SOP #HW-24 and 8270D-Rev 4, August 2008-SOP #HW-22), and professional judgment. Region II has not developed a validation checklist SOP for the methods used to assess the metals in this SDG (SW-846 method 6010C). The Region II Standard Operating Procedure for the Evaluation of Metals Data for the CLP was used as applicable for the metals data. Region II flagging conventions were used. All areas of concern are discussed in the body of the report and a summary of data qualifications is provided.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Lab ID</th>
<th>Matrix</th>
<th>VOA</th>
<th>SVOA</th>
<th>Fe, Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW05-0512</td>
<td>L1093-01</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-TB01-052212</td>
<td>L1093-02</td>
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<td></td>
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<tr>
<td>VWAI-EB01-052312</td>
<td>L1093-03</td>
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<tr>
<td>VWAI-TB01-052312</td>
<td>L1093-04</td>
<td>water</td>
<td>X</td>
<td></td>
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</tr>
<tr>
<td>VWAI-MW04-0512</td>
<td>L1093-05</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>VWAI-MW07-0512</td>
<td>L1093-06</td>
<td>water</td>
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<tr>
<td>VWAI-MW07P-0512</td>
<td>L1093-07</td>
<td>water</td>
<td>X</td>
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<td></td>
</tr>
<tr>
<td>VWAI-MW05-0512 MS</td>
<td>L1093-01MS</td>
<td>water</td>
<td>X</td>
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<tr>
<td>VWAI-MW05-0512 MSD</td>
<td>L1093-01MSD</td>
<td>water</td>
<td>X</td>
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</tr>
</tbody>
</table>

The following quality control samples were provided with this SDG: samples VWAI-TB01-052212 and VWAI-TB01-052312-trip blanks; sample VWAI-EB01-052312-equipment blank; and sample VWAI-MW07P-0512-field duplicate of sample VWAI-MW07-0512.

The samples were evaluated based on the following criteria:

- Data Completeness
- Sample Condition
- Technical Holding Times
- GC/MS Tuning
- GC Performance
Overall Evaluation of Data/Potential Usability Issues

A summary of qualifications applied to the sample results are noted below for the fractions validated. Specific details regarding qualification of the data are addressed in the Specific Evaluation section of this narrative. If an issue is not addressed there were no actions required based on unmet quality criteria. When more than one qualifier is associated with a compound/analyte the validator has chosen the qualifier that best indicates possible bias in the results and flagged the data accordingly. However, information regarding all quality control issues is provided in the body of the report and on the qualification summary page. Please note that when a compound or analyte is flagged due to blank contamination the BL qualifier code takes precedence over all other qualifier codes except a code that explains rejected data.

VOA

No qualifications to the data were required.

SVOA

The associated matrix spike and matrix spike duplicate exhibited non-compliant recoveries that required qualifications to the data.

Select Filtered Metals

The laboratory did not perform a matrix spike, matrix duplicate or a serial dilution in this SDG. These QC samples are required by Region II. Qualifications were required.
Specific Evaluation of Data

Data Completeness

The SDG was received complete and intact. Resubmissions were not required.

Technical Holding Times

According to chain of custody records, sampling was performed on 5/22-23/12 and samples were received at the laboratory 5/23-24/12. All sample preparation and analysis was performed within Region II and/or method holding time requirements.

Matrix Spike/Matrix Duplicates

SVOA

The matrix spike and matrix spike duplicate associated with sample VWAI-MW05-0512 exhibited low recoveries for bis(2-ethylhexyl) phthalate at 37% and 30% (QC limit 40-125%). Therefore the non-detected result in the associated sample was qualified as estimated IU, qualifier code: MSL.

Select Filtered Metals

The laboratory did not perform a matrix spike/matrix duplicate on a sample from this SDG. Region II required that all positive and non-detect results be qualified as estimated J because of this. Therefore, the reported positive and non-detect results for iron and manganese were qualified as estimated J/UL with a qualifier code of OT.

Serial Dilution

Select Filtered Metals

The laboratory did not perform a serial dilution sample on a sample from this SDG. Region II required that all positive results be qualified as estimated J because of this. Therefore, the reported positive results for iron and manganese were qualified as estimated J with a qualifier code of OT.

A summary of qualifications required is provided on the following page. Please do not hesitate to contact DataQual ES with any questions regarding this validation report.

Sincerely,

Jacqueline Cleveland
Vice President
Summary of Data Qualifications

### VOA

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>No qualifications</td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tbody>
</table>

### SVOA

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW05-0512</td>
<td>bis(2-ethylhexyl) phthalate</td>
<td>-</td>
<td>UJ</td>
<td>MSL</td>
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</table>

### Select Filtered Metals

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Analyte</th>
<th>Results</th>
<th>Q flag</th>
<th>Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>all samples</td>
<td>iron, manganese</td>
<td>+/-</td>
<td>J/UJ</td>
<td>OT</td>
</tr>
</tbody>
</table>
Glossary of Qualification Flags and Abbreviations

Qualification Flags (Q-Flags)

U  not detected above the reported sample quantitation limit
J  estimated value
UJ reported quantitation limit is qualified as estimated
N  analyte has been tentatively identified
JN analyte has been tentatively identified, estimated value
R  result is rejected; the presence or absence of the analyte cannot be verified

Method/Preparation/Field QC Blank Qualification Flags (Q-Flags)

Organic Methods

NA The sample result for the blank contaminant is greater than the
LOQ (2X sample LOQ for common laboratory contaminants) when the blank value is less than the LOQ. The sample result for the blank contaminant is not qualified with any blank qualifiers.

LOQ The sample result for the blank contaminant is less than the LOQ (2X sample LOQ for common laboratory contaminants) but greater than the MDL when the blank value is less than the LOQ. The sample result for the blank contaminant is changed to the LOQ and qualified as non-detect U.

Inorganic Methods

ICB/CCB/PB Action:

No Action - The sample result is greater than the LOQ and greater than ten times (10X) the blank value.

U - The sample result is greater than or equal to the MDL but less than or equal to the LOQ, result is reported as non-detect at the LOQ, when the ICB/CCB/PB result is less or greater than the LOQ.
Glossary of Qualification Flags and Abbreviations, continued

R - Sample result is greater than the LOQ and less than the ICB/CCB/PB value when the ICB/CCB/PB value is greater than the LOQ.

J - Sample result is greater than the ICB/CCB/PB value but less than 10X the ICB/CCB/PB value when ICB/CCB/PB value is greater than the LOQ.

J/UJ - Sample result is less than 10X LOQ when blank result is below the negative LOQ.

Field QC Blank action:

Note – Use field blanks to qualify data only if field blank results are greater than prep blank results.
Do not use rinsate blank associated with soils to qualify water samples and vice versa.

No Action - The sample result is greater than the LOQ and greater than ten times (10X) the blank value.

U - The sample result is greater than or equal to the MDL but less than or equal to the LOQ, result is reported as non-detect at the LOQ, when the FB result is less or greater than the LOQ.

R - Sample result is greater than the LOQ and less than the FB value when the FB value is greater than the LOQ.

J - Sample result is greater than the FB value but less than 10X the FB value when FB value is greater than the LOQ.

General Abbreviations

RL reporting limit
MDL method detection limit
IDL instrument detection limit
LOD Level of Detection
LOQ Level of Quantitation
+ positive result
- non-detect result
## QUALIFIER CODE REFERENCE

<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>TN</td>
<td>Tune</td>
</tr>
<tr>
<td>BSL</td>
<td>Blank Spike/LCS - High Recovery</td>
</tr>
<tr>
<td>BSH</td>
<td>Blank Spike/LCS - Low Recovery</td>
</tr>
<tr>
<td>BD</td>
<td>Blank Spike/Blank Spike Duplicate (LCS/LCSD) Precision</td>
</tr>
<tr>
<td>BRL</td>
<td>Below Reporting Limit</td>
</tr>
<tr>
<td>ISL</td>
<td>Internal Standard - Low Recovery</td>
</tr>
<tr>
<td>ISH</td>
<td>Internal Standard - High Recovery</td>
</tr>
<tr>
<td>MSL</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - Low Recovery</td>
</tr>
<tr>
<td>MSH</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - High Recovery</td>
</tr>
<tr>
<td>M1</td>
<td>Matrix interference obscuring the raw data</td>
</tr>
<tr>
<td>MDP</td>
<td>Matrix Spike/Matrix Spike Duplicate Precision</td>
</tr>
<tr>
<td>2S</td>
<td>Second Source - Bad reproducibility between tandem detectors</td>
</tr>
<tr>
<td>SSL</td>
<td>Spiked Surrogate - Low Recovery</td>
</tr>
<tr>
<td>SSH</td>
<td>Spiked Surrogate - High Recovery</td>
</tr>
<tr>
<td>SD</td>
<td>Serial Dilution Reproducibility</td>
</tr>
<tr>
<td>ICL</td>
<td>Initial Calibration - Low Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICH</td>
<td>Initial Calibration - High Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICB</td>
<td>Initial Calibration - Bad Linearity or Curve Function</td>
</tr>
<tr>
<td>CCL</td>
<td>Continuing Calibration - Low Recovery or %Difference</td>
</tr>
<tr>
<td>CCH</td>
<td>Continuing Calibration - High Recovery or %Difference</td>
</tr>
<tr>
<td>LD</td>
<td>Lab Duplicate Reproducibility</td>
</tr>
<tr>
<td>HT</td>
<td>Holding Time</td>
</tr>
<tr>
<td>PD</td>
<td>Pesticide Degradation</td>
</tr>
<tr>
<td>2C</td>
<td>Second Column - Poor Dual Column Reproducibility</td>
</tr>
<tr>
<td>LR</td>
<td>Concentration Exceeds Linear Range</td>
</tr>
<tr>
<td>MBL, EBL, FBL or TBL</td>
<td>Blank Contamination</td>
</tr>
<tr>
<td>RE</td>
<td>Redundant Result - due to Re-analysis or Re-extraction</td>
</tr>
<tr>
<td>DL</td>
<td>Redundant Result - due to Dilution</td>
</tr>
<tr>
<td>FD</td>
<td>Field Duplicate</td>
</tr>
<tr>
<td>OT</td>
<td>Other - explained in data validation report</td>
</tr>
<tr>
<td>%SOL</td>
<td>High moisture content</td>
</tr>
<tr>
<td>CAS NO.</td>
<td>COMPOUND</td>
</tr>
<tr>
<td>---------</td>
<td>------------------------</td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
</tr>
<tr>
<td>CAS NO.</td>
<td>COMPOUND</td>
</tr>
<tr>
<td>--------</td>
<td>-------------------</td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Case No.: L1093
Mod. Ref No.: ...
SDG No.: SL1093
Matrix: (SOIL/SED/WATER) WATER
Lab Sample ID: L1093-03A
Sample wt/vol: 5.00 (g/mL) ML
Lab File ID: V6I7300.D
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
Date Received: 05/24/2012
Date Analyzed: 05/25/2012
GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50 U</td>
<td>U</td>
<td></td>
<td>0.41</td>
<td>0.50</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>0.50 U</td>
<td>U</td>
<td></td>
<td>0.33</td>
<td>0.50</td>
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<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0 U</td>
<td>U</td>
<td></td>
<td>0.61</td>
<td>1.0</td>
</tr>
</tbody>
</table>
VOLATILE ORGANICS ANALYSIS DATA SHEET

Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L1093  
Contract:  
Mod. Ref No.:  
SDG No.: SL1093

Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Lab Sample ID: L1093-04A  
Lab Code: MITKEM  
Lab File ID: V6I7297.D

Level: (TRACE/LOW/MED) LOW  
Date Received: 05/24/2012  
% Moisture: not dec.  
Date Analyzed: 05/25/2012

GC Column: DB-624  
ID: 0.25 (mm)  
Dilution Factor: 1.0

Soil Extract Volume:  
Soil Aliquot Volume:  
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50</td>
<td>U</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>0.50</td>
<td>U</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0</td>
<td>U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Matrix: WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: LOW
% Moisture: not dec.
GC Column: DB-624 ID: 0.25 (mm)
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50</td>
<td>U</td>
<td>0.41</td>
<td>0.50</td>
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<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>2.6</td>
<td>J</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0</td>
<td>U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  Contract: 
Lab Code: MITKEM  Case No.: L1093  Mod. Ref No.:  SDG No.: SL1093  
Matrix: (SOIL/SED/WATER) WATER  Lab Sample ID: L1093-06A  
Sample wt/vol: 5.00 (g/mL) ML  Lab File ID: V6I7302.D  
Level: (TRACE/LOW/MED) LOW  Date Received: 05/24/2012  
% Moisture: not dec.  Date Analyzed: 05/25/2012  
GC Column: DB-624  ID: 0.25 (mm)  Dilution Factor: 1.0  
Soil Extract Volume: (uL)  Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50 U</td>
<td></td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
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<td>Benzene</td>
<td>2.9 J</td>
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<td>0.50</td>
<td>5.0</td>
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<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0 U</td>
<td></td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Soil Extract Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50</td>
<td>U</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
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<td>0.50</td>
<td>5.0</td>
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<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0</td>
<td>U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L1093  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Dilution Factor: 1.0  
Soil Extract Volume: (uL)  
Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: ug/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>54</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
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</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>52</td>
<td>0.33</td>
<td>0.50</td>
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</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>51</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Contract:  
Case No.: L1093  
Mod. Ref No.: SDG No.: SL1093  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Lab Sample ID: L1093-01AMSD  
Lab File ID: V6I7273.D  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
Date Received: 05/23/2012  
Date Analyzed: 05/24/2012  
GC Column: DB-624  
ID: 0.25 (mm)  
Dilution Factor: 1.0  
Soil Extract Volume: (uL)  
Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>55</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>54</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>78-97-5</td>
<td>1,2-Dichloropropane</td>
<td>54</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
<td></td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Case No.: L1093
Mod. Ref No.: __________
SDG No.: SL1093
Matrix: (SOIL/SED/WATER) WATER
Lab Sample ID: L1093-01B
Sample wt/vol: 1000 (g/mL) ML
Lab File ID: S6A9050.D
Level: (LOW/MED) LOW
Extraction: (Type) SEP
% Moisture: Decanted: (Y/N) __________
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N pH: __________
Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>1.3</td>
<td>J</td>
<td>0.96</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>11</td>
<td></td>
<td>0.94</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>2.0</td>
<td></td>
<td>1.3</td>
<td>2.0</td>
<td>5.0</td>
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</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Case No.: L1093
Mod. Ref No.: SDG No.: S10093
Matrix: (SOIL/SED/WATER) WATER
Lab Sample ID: L1093-038
Sample wt/vol: 1000 (g/mL) ML
Lab File ID: S6A9046.D
Level: (LOW/MED) LOW
Extraction: (Type) SEP
% Moisture: Decanted: (Y/N)
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.00
GPC Cleanup: (Y/N) N
pH: Dilution Factor: 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>2.0</td>
<td>0</td>
<td>0.96</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>2.0</td>
<td>0</td>
<td>0.94</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>2.0</td>
<td>0</td>
<td>1.3</td>
<td>2.0</td>
<td>5.0</td>
</tr>
<tr>
<td>CAS NO.</td>
<td>COMPOUND</td>
<td>CONCENTRATION: UG/L</td>
<td>Q</td>
<td>DL</td>
<td>LOD</td>
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</tr>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>2.2</td>
<td>0.96</td>
<td>2.0</td>
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<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>2.0</td>
<td>0.94</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>111-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>2.0</td>
<td>1.3</td>
<td>2.0</td>
<td>2.0</td>
<td>5.0</td>
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</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L1093  
Mod. Ref No.:  
SDG No.: SL1093  
Lab Sample ID: L1093-06B  
Lab File ID: S6A9048.D  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 1000 (g/mL) ML  
Level: (LOW/MED) LOW  
% Moisture:  
Decanted: (Y/N)  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL) GPC Factor: 1.00  
GPC Cleanup: (Y/N) N  
ph:  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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</thead>
<tbody>
<tr>
<td>91-20-3</td>
<td>Naphthalene</td>
<td>3.3</td>
<td>0.96</td>
<td>2.0</td>
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<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>3.4</td>
<td>0.94</td>
<td>2.0</td>
<td>2.0</td>
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<tr>
<td>117-61-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>2.0</td>
<td>U</td>
<td>1.3</td>
<td>2.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Case No.: L1093
Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 1000 (g/mL) ML
Level: (LOW/MED) LOW
% Moisture: Decanted: (Y/N) N
Concentrated Extract Volume: 1000 (uL)
Injection Volume: 1.0 (uL) GPC Factor: 1.0
GPC Cleanup: (Y/N) N

CAS NO. | COMPOUND                      | CONCENTRATION: UG/L | Q  | DL | LOD | LOQ |
--------|--------------------------------|---------------------|----|----|-----|-----|
91-20-3 | Naphthalene                    | 3.2                 |    | 0.96| 2.0 | 2.0 |
91-57-6 | 2-Methylnaphthalene           | 3.3                 |    | 0.94| 2.0 | 2.0 |
117-81-7| Bis(2-ethylhexyl)phthalate     | 2.0                 |    | 1.3 | 2.0 | 5.0 |
**Lab Name:** SPECTRUM ANALYTICAL, INC.  
**Lab Code:** MITKEM  
**Case No.:** L1093  
**Matrix:** (SOIL/SED/WATER)  
**Sample wt/vol:** 1000 (g/mL) ML  
**Level:** (LOW/MED) LOW  
**% Moisture:**  
**Concentrated Extract Volume:** 1000 (uL)  
**Injection Volume:** 1.0 (uL)  
**GPC Clean-up:** (Y/N) N  
**pH:**  
**CAS NO.** | **COMPounder** | **CONCENTRATION:**  
<table>
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<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
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<td>0.94</td>
<td>2.0</td>
<td>2.0</td>
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<td></td>
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<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>18</td>
<td>1.3</td>
<td>2.0</td>
<td>5.0</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
**Lab Name:** SPECTRUM ANALYTICAL, INC.  
**Lab Code:** MITKEN  
**Case No.:** L1093  
**Mod. Ref No.:**  
**SDG No.:** SL1093  

**Matrix:** WATER  
**Sample wt/vol:** 1000 (g/mL) ML  
**Lab Sample ID:** L1093-01BMSD  
**Lab File ID:** S6A9052.D  
**Level:** LOW  
**Extraction:** SEPF  
**% Moisture:**  
**Decanted:** (Y/N)  
**Concentrated Extract Volume:** 1000 (uL)  
**Injection Volume:** 1.0 (uL)  
**GPC Factor:** 1.00  
**GPC Cleanup:** (Y/N)  
**pH:**  
**Dilution Factor:** 1.0

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tr>
<td>91-57-6</td>
<td>2-Methylnaphthalene</td>
<td>49</td>
<td>0.94</td>
<td>2.0</td>
<td>2.0</td>
<td></td>
</tr>
<tr>
<td>117-81-7</td>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>15</td>
<td>1.3</td>
<td>2.0</td>
<td>5.0</td>
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</tr>
</tbody>
</table>
**U.S. EPA - CLP**

**INORGANIC ANALYSIS DATA SHEET**

| Lab Name: Spectrum Analytical, Inc. | Contract: 933562, N62 |
| Lab Code: MITKEM | Case No.: | SAS No.: | SDG No.: SL1093 |
| Matrix (soil/water): WATER | Lab Sample ID: L1093-05 |
| Level (low/med): MED | Date Received: 05/24/2012 |
| % Solids: 0.0 |

Concentration Units (ug/L or mg/kg dry weight): ug/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
<th>MDL</th>
<th>LOD</th>
<th>PQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>7439-89-6 Iron</td>
<td>50</td>
<td>OT</td>
<td>P</td>
<td>31.0</td>
<td>50.0</td>
<td>200</td>
<td></td>
<td></td>
</tr>
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<td>7439-96-5 Manganese</td>
<td>712</td>
<td>OT</td>
<td>P</td>
<td>10.0</td>
<td>15.0</td>
<td>50.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments:

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FORM I - IN SW846

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Em1.12.12.A
U.S. EPA - CLP
1 INORGANIC ANALYSIS DATA SHEET

Lab Name: Spectrum Analytical, Inc. Contract: 933562, N62
Lab Code: MITKEM Case No.: ________ SAS No.: ________ SDG No.: SL1093
Matrix (soil/water): WATER Lab Sample ID: L1093-01
Level (low/med): MED Date Received: 05/23/2012
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): ug/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>Q</th>
<th>M</th>
<th>MDL</th>
<th>LOD</th>
<th>PQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>7439-89-6</td>
<td>Iron</td>
<td>107</td>
<td>J</td>
<td>OT</td>
<td>P</td>
<td>31.0</td>
<td>50.0</td>
<td>200</td>
</tr>
<tr>
<td>7439-96-5</td>
<td>Manganese</td>
<td>1230</td>
<td>J</td>
<td>OT</td>
<td>P</td>
<td>10.0</td>
<td>15.0</td>
<td>50.0</td>
</tr>
</tbody>
</table>

Comments:
Inorganic Analysis Data Sheet

Lab Name: Spectrum Analytical, Inc.  
Lab Code: MITKEM  
Matrix (soil/water): WATER  
Level (low/med): MED  
% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): ug/L

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Analyte</th>
<th>Concentration</th>
<th>C</th>
<th>M</th>
<th>MDL</th>
<th>LOD</th>
<th>PQL</th>
</tr>
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<tbody>
<tr>
<td>7439-89-6</td>
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<td>P</td>
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<td>31.0</td>
<td>50.0</td>
<td>200</td>
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<tr>
<td>7439-96-5</td>
<td>Manganese</td>
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<td>P</td>
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<td>10.0</td>
<td>15.0</td>
<td>50.0</td>
</tr>
</tbody>
</table>

Comments:

Signature: [Signature]
Date: [Date]

Form I - IN  
Sw846
REPORT NARRATIVE
Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.

Client: CH2M-Hill, Inc.

Project: CTO-0083 Vieques AOC I

Laboratory Workorder / SDG #: L1093
SW846 8260C, VOC by GC-MS

I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:

All samples were prepared within the method-specified holding times.

B. Sample Analysis:

All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 8260C

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW5030

V. INSTRUMENTATION
The following instrumentation was used

Instrument Code: V6
Instrument Type: GCMS-VOA
Description: HP6890 / HP5973
Manufacturer: Hewlett-Packard
Model: 6890 / 5973
GC Column used: 30 m X 0.25 mm ID [1.40 um thickness] DB-624 capillary column.

VI. ANALYSIS

A. Calibration:

   Calibrations met the method/SOP acceptance criteria.

B. Blanks:

   All method blanks were within the acceptance criteria.

C. Surrogates:

   Surrogate standard percent recoveries were within the QC limits.

D. Spikes:

   1. Laboratory Control Spikes (LCS):

      Percent recoveries for lab control samples were within the QC limits.

   2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):

      Matrix spikes were performed on samples: VWAI-MW05-0512 (L1093-01AMS) and VWAI-MW05-0512 (L1093-01AMSD).

      Percent recoveries were within the QC limits.

      Replicate RPDs were within the advisory QC limits.

E. Internal Standards:

   Internal standard peak areas were within the QC limits.

F. Dilutions:
No sample in this SDG required analysis at dilution.

G. Samples:

No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: 

Date: 6/14/2012
REPORT NARRATIVE
Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.
Client: CH2M-Hill, Inc.
Project: CTO-0083 Vieques AOC I
Laboratory Workorder / SDG #: L1093
SW846 8270D, SVOA by GC-MS

I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:
   All samples were prepared within the method-specified holding times.

B. Sample Analysis:
   All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code:
SW846 8270D

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW3510

V. INSTRUMENTATION

The following instrumentation was used
VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Surrogates:

Surrogate standard percent recoveries were within the QC limits with the following exceptions. Please note that the acceptance criteria allow one surrogate recovery outside of the QC limits per fraction.

VWAI-MW05-0512 (L1093-01B), recovery is below criteria for Terphenyl-d14 at 22% with criteria of (50-135).

VWAI-MW05-0512 (L1093-01BMS), recovery is below criteria for Terphenyl-d14 at 18% with criteria of (50-135).

VWAI-MW05-0512 (L1093-01BMSD), recovery is below criteria for Terphenyl-d14 at 17% with criteria of (50-135).

VWAI-EB01-052312 (L1093-03B), recovery is below criteria for Terphenyl-d14 at 46% with criteria of (50-135).

VWAI-MW04-0512 (L1093-05B), recovery is below criteria for Terphenyl-d14 at 30% with criteria of (50-135).

VWAI-MW07-0512 (L1093-06B), recovery is below criteria for Terphenyl-d14 at 34% with criteria of (50-135).

VWAI-MW07P-0512 (L1093-07B), recovery is below criteria for Terphenyl-d14 at 43% with criteria of (50-135).
D. Spikes:

1. Laboratory Control Spikes (LCS):
   
   Percent recoveries for lab control samples were within the QC limits.

2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):
   
   Matrix spikes were performed on samples: VWAI-MW05-0512 (L1093-01BMS) and VWAI-MW05-0512 (L1093-01BMSD).
   
   Percent recoveries were within the QC limits with the following exceptions:
   
   VWAI-MW05-0512 (L1093-01BMS) Percent Recovery is outside QC Limits, recovery is below criteria for Bis(2-ethylhexyl)phthalate at 37% with criteria of (40-125).
   
   VWAI-MW05-0512 (L1093-01BMSD) Percent Recovery is outside QC Limits, recovery is below criteria for Bis(2-ethylhexyl)phthalate at 30% with criteria of (40-125).
   
   Replicate RPDs were within the advisory QC limits.

E. Internal Standards:

Internal standard peak areas were within the QC limits.

F. Dilutions:

No sample in this SDG required analysis at dilution.

G. Samples:

No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.
Signed: [Signature]

Date: 6/14/2012
I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:

All samples were prepared within the method-specified holding times.

B. Sample Analysis:

All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 6010C

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW3005A
V. INSTRUMENTATION

The following instrumentation was used to perform analysis:

Instrument Code: OPTIMA3
Instrument Type: ICP
Description: Optima ICP-OES
Manufacturer: Perkin-Elmer
Model: 4300 DV

VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Spikes:

1. Laboratory Control Spikes (LCS):
   Percent recoveries for laboratory control samples were within the QC limits.

2. Matrix spike (MS):
   A matrix spike was not performed on any sample in this SDG.

D. Post Digestion Spike (PDS):
   A post-digestion spike was not performed on any sample in this SDG.

E. Duplicate sample:
   A duplicate analysis was not performed on any sample in this SDG.

F. Serial Dilution (SD):
   A serial dilution was not performed on any sample in this SDG.
G. Samples:

No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: 

Date: 06/14/12
### CHAIN OF CUSTODY RECORD

**Special Handling:**
- TAT: Indicate Date Needed:
  - All TATs subject to laboratory approval. Min. 24-hour notification needed for rushed.
  - Samples disposed of after 30 days unless otherwise instructed.

**Project No.:** 392485, PR, FK

**Site Name:** Vieques, PR

**Location:** Vieques, PR

**State:** PR

**Sample(s):** D. Whitney, M. Diaz, P. Marquez

---

**List preservative code:**
- 6 W/4 W/9

**QA/QC Reporting Level**
- Level I
- Level II
- Level III
- Level IV
- Others

**State specific reporting standards:**
- None

---

**Containers:**
- 01
- 02
- 03

**Analyses:**
- VOCs
- SVOCs
- Metals
- Other
- Other

---

**Conditions Upon Receipt:**
- Rushed
- Ambient

---

**Condition upon Receipt:**
- Yes
- No

---

**Relinquished by:**
- ((Signature))

**Received by:**
- ((Signature))

**Date:** 05/01/12

**Time:** 13:00

**Quality Control:**
- ((Signature))

**Date:** 05/03/11

**Time:** 8:55

---

**Notes:**
- Trip blank

---

**Project Mgr.:** S. Brand

**PO No.:** __________

**RQN:** __________

---

**Report To:** Stephen Brand

**Invoice To:** Stephen Brand

**Address:**
- Spectrum Analytical Inc.
- 175 Metro Center Boulevard
- Warwick, RI 02886-1755
- Phone: 401-732-3400
- Fax: 401-732-3499
- www.spectrum-analytical.com

---

**Sample(s):**
- DW = Drinking Water
- GW = Groundwater
- WW = Wastewater
- O = Oil
- SW = Surface Water
- SO = Soil
- SL = Sludge
- A = Air
- X1 = __________
- X2 = __________
- X3 = __________

**Preservative Code:**
- G = Grab
- C = Composite

---

**Lab Id.:**
- 01
- 02
- 03

**Sample Id.:**
- 05/01/12
- 05/01/12
- 05/01/12

**Date:**
- 05/01/12
- 05/01/12
- 05/01/12

**Time:**
- 10:55
- 10:55
- 10:55

**Type:**
- G
- G
- G

**Matrix:**
- GW
- GW
- GW

**Vials:**
- 01
- 02
- 03

**Aerobic Glass:**
- 2
- 2
- 2

**Plastic:**
- 1
- 1
- 1

**QA/QC:**
- None

---

**EDD Format:**
- None

---

**Condition upon receipt:**
- Ambient

---

**Notes:**
- None

---

**175 Metro Center Boulevard • Warwick, RI 02886-1755 • 401-732-3400 • Fax 401-732-3499 • www.spectrum-analytical.com**
# Chain of Custody Record

**Report To:** Stephen Bread / 1180

**Invoice To:** Stephen Bread / 1180

**Project No.:** 382085 FE FK

**Site Name:** Vieques AGCT

**Location:** Vieques, PR  State: PR

**Sampler(s):** P. Murphy, D. Walsizer, M. Fiedler

<table>
<thead>
<tr>
<th>Lab Id.</th>
<th>Sample Id.</th>
<th>Date</th>
<th>Time</th>
<th>Type</th>
<th>Matrix</th>
<th># of VOA Vials</th>
<th># of Plastic</th>
<th># of Glass</th>
<th>Analyzers</th>
</tr>
</thead>
<tbody>
<tr>
<td>03</td>
<td>WVA-EO1-092D</td>
<td>5/23/12</td>
<td>07:05</td>
<td>G</td>
<td>G</td>
<td>6</td>
<td>2</td>
<td>2</td>
<td>NVOV</td>
</tr>
<tr>
<td>01</td>
<td>WVA-TBE1-052D</td>
<td>6/13/12</td>
<td>07:45</td>
<td>G</td>
<td>TBE</td>
<td>6</td>
<td>1</td>
<td>1</td>
<td>NVOV</td>
</tr>
<tr>
<td>05</td>
<td>WVA-AMO2-052D</td>
<td>6/13/12</td>
<td>07:25</td>
<td>G</td>
<td>AMO</td>
<td>6</td>
<td>2</td>
<td>2</td>
<td>NVOV</td>
</tr>
<tr>
<td>06</td>
<td>WVA-AMO2-052D</td>
<td>6/13/12</td>
<td>18:30</td>
<td>G</td>
<td>AMO</td>
<td>6</td>
<td>4</td>
<td>2</td>
<td>NVOV</td>
</tr>
<tr>
<td>07</td>
<td>WVA-AMO2-052D</td>
<td>6/13/12</td>
<td>10:35</td>
<td>G</td>
<td>AMO</td>
<td>6</td>
<td>2</td>
<td>2</td>
<td>NVOV</td>
</tr>
</tbody>
</table>

**Conditions upon receipt:** Fed Ex

**Special Handling:**
- E-mail to
- EDD Format
- Relinquished by:
- Received by:
- Date: 05/24/12
- Time: 12:30

**QA/QC Reporting Level:**
- Level I
- Level II
- Level III
- Level IV
- Other

**State Specific Reporting Standards:** Equipment blank, TRIP blank.
## Sample Condition Form 6

**Spectrum Analytical, Inc. Featuring Manibal Technology -- Rhode Island Division**

**Work Order:** L1093  |  **Client Name:** CH2M Hill, Inc.

**Project Name/Event:** CT0-0083 Viegges MOD E and 1

**Remarks:** (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.

<table>
<thead>
<tr>
<th>Custody Seal(s)</th>
<th>Present/Absent</th>
<th>Intact/Broken</th>
<th>Lab Sample ID</th>
<th>Preservation (pH)</th>
<th>Soil Headspace or Air Bubble &amp; or equal to 1/4&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td></td>
<td></td>
<td>L1093-01</td>
<td>&lt;2</td>
<td>HCl JV 5/23/12</td>
</tr>
<tr>
<td>2.</td>
<td></td>
<td></td>
<td>L1093-02</td>
<td>&lt;2</td>
<td>Acetic Acid</td>
</tr>
</tbody>
</table>

**Custody Seal Nos:**

**Traffic Reports/Chain of Custody Records (TRA/COR) or Packing Lists:**

**Airbill:**

**Airbill No.:** FedEx 8708 2606 2940

**Sample Tags:**

**Sample Tag Numbers:**

**Sample Condition:**

**Cooler Temperature Indicator Bottle:**

**Cooler Temperature:** 4.0 °C

**Use Information on TRA/COR and sample tags agree?**

**Date Received at Laboratory:** 05/23/2012

**Time Received:** 08:55

**Sample Transfer:**

**Fraction (1) TVOA/VOA**

**Fraction (2) SVOA/FEAT/FAD**

**Area 1**

**Area 2**

**1° Temp Temp ID:** MT-1

**Coolant/Condition:** Ice

**Preservative Name/OF No.:**

**VOA Matrix Key:**

- US = Unpreserved Soil
- A = Air
- UA = Unpreserved Aqueous
- H = HCl
- M = MeOH
- E = Encore
- N = NaH2SO4
- F = Freeze

**See Sample Condition Notification/Corrective Action Form**

**Yes/No**

**Rad OK**

**Yes/No**

**Sample Condition Form 6**
<table>
<thead>
<tr>
<th>Remarks: (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Custody Seal(s)</td>
</tr>
<tr>
<td>2. Custody Seal Pos.</td>
</tr>
<tr>
<td>3. Traffic Reports/ Chain of Custody Records (TR/CCDR) or Packing Lists</td>
</tr>
<tr>
<td>4. Airbill</td>
</tr>
<tr>
<td>5. Airbill No.</td>
</tr>
<tr>
<td>6. Sample Tags</td>
</tr>
<tr>
<td>Sample Tag Numbers</td>
</tr>
<tr>
<td>7. Sample Condition</td>
</tr>
<tr>
<td>8. Cooler Temperature Indicator Bottle</td>
</tr>
<tr>
<td>9. Cooler Temperature</td>
</tr>
<tr>
<td>10. Does information on TR/CCDR and sample tags agree?</td>
</tr>
<tr>
<td>11. Date Received at Laboratory</td>
</tr>
<tr>
<td>Sample Transfer</td>
</tr>
<tr>
<td>Fraction (1) TVOA/VOA</td>
</tr>
<tr>
<td>Area #</td>
</tr>
<tr>
<td>By</td>
</tr>
<tr>
<td>On</td>
</tr>
<tr>
<td>IR Temp Gun ID: HT-1</td>
</tr>
<tr>
<td>COOLANT CONDITION: ICE</td>
</tr>
<tr>
<td>Preservation Name/No.</td>
</tr>
<tr>
<td>VOA Matrix Key:</td>
</tr>
<tr>
<td>US = Unpreserved Soil</td>
</tr>
<tr>
<td>UA = Unpreserved Aqueous</td>
</tr>
<tr>
<td>M = MeOH</td>
</tr>
<tr>
<td>N = NaHSO4</td>
</tr>
<tr>
<td>See Sample Condition Notification/Corrective Action Form Yes/No</td>
</tr>
<tr>
<td>Rad OK Yes/No</td>
</tr>
<tr>
<td>Sample Condition Form 7</td>
</tr>
</tbody>
</table>
I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: LI093  LAB: Spectrum

SITE NAME: Vieques AOC I C70-083

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent? YES NO N/A

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, and/or cover letter signed release present? YES NO N/A

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, and/or Chain of Custodies from the field samplers present for all samples sign release present? YES NO N/A

ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)? YES NO N/A

1.3 Sample Conditions/Problems
1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

 ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

 ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

 ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects "R".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

 The maximum holding time for aqueous samples is 14 days.

 The maximum holding time for soils nonaqueous samples is 14 days.

 NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with HCL acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. For non-aqueous samples for volatile components that are frozen (less than 7°C) or are properly cooled (4°C ± 2°C) and perserved with NaHSO₄, the maximum holding time is 14 days from sample collection. If
uncertain about preservation, contact the laboratory /sampling team to determine whether or not samples were preserved.

**ACTION:** Qualify sample results according to Table 1:

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Preserved</th>
<th>Criteria</th>
<th>Action</th>
<th>Detected Associated Compounds</th>
<th>Non-Detected Associated Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aqueous</td>
<td>No</td>
<td>≤7 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>No</td>
<td>&gt;7 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>&gt;14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td>Non Aqueous</td>
<td>No</td>
<td>≤14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yes/No</td>
<td>&gt;14 days</td>
<td>J</td>
<td>R</td>
<td></td>
</tr>
</tbody>
</table>

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Water

b. Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:

a. Water

b. Soil

**ACTION:** If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data
Assessments and contact the laboratory/project officer/appropriate official for an explanation/resubmittal, make any necessary corrections and document effect in the Data Assessment.

3.3 Were the surrogate recovery limits followed per Table 2. If Table 2 criteria were not followed, the laboratory may use in-house performance criteria (per SW-846, Method 8000C, section 9.7). Other compounds may be used as surrogates, depending upon the analysis requirements.

<table>
<thead>
<tr>
<th>DMC</th>
<th>Recovery Limits (%)</th>
<th>Recovery Limits Soil/Sediment</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Bromofluorobenzene</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dibromofluoromethane</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Toluene-d₈</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dichloroethane-d₄</td>
<td>80-120</td>
<td>70-130</td>
</tr>
</tbody>
</table>

Note: Use above table if laboratory did not provide in-house recovery criteria.

Note: Other compounds may be used as surrogates depending upon the analysis requirements.

3.4 Were outliers marked correctly with an asterisk?

ACTION: Circle all outliers with a red pencil.

3.5 Were one or more volatile surrogate recoveries out of specification for any sample or method blank. Table 2.

If yes, were samples reanalyzed?

Were method blanks reanalyzed?
ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects, but qualify positive results as estimated "J".

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. If one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose the blank problem to be an isolated occurrence.

3.6 Are there any transcription/calculation errors between raw data and reported data? [ ] [ ]

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Sample (Form III/Equivalent)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG. [ ] [ ]
Use: Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

A. Water
B. Soil
C. Med Soil

Note: LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation/resubmittals. Make note in the data assessment.

4.2 Were the Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

A. Water
B. Soil
C. Med Soil

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

4.3 Have in house LCS recovery limits been developed (Method 8000C, Sect 9.7).

4.4 If in house limits are not developed, are LCS acceptance recovery limits between 70 - 130% (Method 8000C Sect 9.5)?

4.5 Were one or more of the volatile LCS recoveries outside the in house laboratory recovery criteria for spiked analytes? If in house limits are not present use 70 - 130% recovery limits.
Table 3. LCS Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Detected Spiked Compounds</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td>UJ</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td>No Qualifications</td>
</tr>
</tbody>
</table>

5.0 Matrix Spikes (Form III or equivalent)

5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix?

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, Method 8260B, Sect 8.4.2).

5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III?

ACTION: If any data are missing take action as specified in section 3.2 above.

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples...
of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month [page 8000C, section 9.5.]

a. Water
b. Waste
c. Soil/Solid

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The concentration of the LCS should be determined as described SW-Method 8000C Section 9.5.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

5.4 Have in house MS recovery limits been developed (Method 8000C, Sect 9.7) for each matrix.

5.5 Were one or more of the volatile MS/MSD recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000C, Sect. 9.5.4.

ACTION: Circle all outliers with a red pencil.

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.
NOTE: No qualification of data is necessary on MS and MSD data alone. However, using informed professional judgement, the data reviewer may use MS and MSD results in conjunction with other QC criteria to determine the need for some qualification.

Note: The data reviewer should first try to determine to what extent the results of the MS and MSD affect the associated data. This determination should be made with regard to the MS and MSD sample itself, as well as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affect all associated samples, and the reviewer must use professional judgement to qualify the data from all associated samples.

Note: The reviewer must use professional judgement to determine the need for qualification of non-spiked compounds.

ACTION: Follow criteria in Table 4 when professional judgement deems qualification of sample.

Table 4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Detected Spiked Compounds</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td>UJ</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td>No Qualifications</td>
</tr>
</tbody>
</table>
6.0 Blank (CLP Form IV Equivalent)

6.1 Is the Method Blank Summary form present? 

6.2 Frequency of Analysis: Has a method blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch? 

6.3 Has a method blank been analyzed for each GC/MS system used? 

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data. 

6.4 Chromatography: review the blank raw data – chromatograms, quant reports or data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable for volatile organic compounds? 

7.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below. 

7.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary. 

- 15 VOA -
7.2 Do any field/rinse blanks have positive volatile organic compound results?

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in Table 5 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.
### Table 5. Volatile Organic Analysis Blank Contamination Criteria

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td></td>
<td>No qualification</td>
</tr>
<tr>
<td>&lt; CRQL*</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
<td></td>
</tr>
<tr>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
<td></td>
</tr>
<tr>
<td>&gt; CRQL*</td>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report the concentration for the sample with a U, or qualify the data as unusable R</td>
<td></td>
</tr>
<tr>
<td>≥ CRQL and ≥ blank contamination</td>
<td>Use professional judgement</td>
<td></td>
<td></td>
</tr>
<tr>
<td>= CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
<td></td>
</tr>
<tr>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gross contamination</td>
<td>Detects</td>
<td>Qualify results as unusable R</td>
<td></td>
</tr>
</tbody>
</table>

* 2x the CRQL for methylene chloride, 2-butanone, and acetone

** Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 ug/L.

**NOTE:** If gross blank contamination exists (e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected volatile organic target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
7.3 Are there field/rinse/equipment blanks associated with every sample?  
   __ __  

   ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.  
   __ __  

   NOTE: For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

   ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?  
   __ __  

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?  
   __ __  

9.3 Has an instrument performance check solution (BFB)
been analyzed for every twelve hours of sample analysis per instrument? (see Table 4, SW-846, page 8260B-36)

**ACTION:** List date, time, instrument ID, and sample analyses for which no associated GC/MS GC/MS tuning data are available.

**ACTION:** If the laboratory/project officer cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

**ACTION:** If mass assignment is in error, flag all associated sample data as unusable, "R".

9.4 Have the ion abundances been normalized to m/z 95?

9.5 Have the ion abundance criteria been met for each instrument used?

**ACTION:** List all data which do not meet ion abundance criteria (attach a separate sheet).

**ACTION:** If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.)

9.7 Have the appropriate number of significant figures (two) been reported?

**ACTION:** If large errors exist, take action as specified in section 3.2.

9.8 Are the spectra of the mass calibration compounds acceptable.

**ACTION:** Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.
10.0 Target Analytes (CLP Form I Equivalent)

10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:
   a. Samples and/or fractions as appropriate
   b. Matrix spikes and matrix spike duplicates
   c. Blanks
   d. Laboratory Control Samples

10.2 Are the reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?
   a. Samples and/or fractions as appropriate
   b. Matrix spikes and matrix spike duplicates (Mass spectra not required)
   c. Blanks
   d. Laboratory Control Samples

ACTION: If any data are missing, take action specified in 3.2 above.

10.3 Is chromatographic performance acceptable with respect to:
   Baseline stability?
### Resolution?

<table>
<thead>
<tr>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tbody>
</table>

### Peak shape?

<table>
<thead>
<tr>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
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<td></td>
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</tbody>
</table>

### Full-scale graph (attenuation)?

<table>
<thead>
<tr>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</table>

**Other:**

________

**ACTION:** Use professional judgement to determine the acceptability of the data.

### 10.4 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample?

<table>
<thead>
<tr>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tbody>
</table>

**ACTION:**

- If any mass spectra are missing, take action specified in 3.2 above.
- If the lab does not generate their own standard spectra, make a note in the Data Assessment.
- If spectra are missing, contact the lab for missing spectra.

### 10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

<table>
<thead>
<tr>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tbody>
</table>

### 10.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

<table>
<thead>
<tr>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
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</tbody>
</table>

**ACTION:**

- Use professional judgement to determine acceptability of data.
- If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit.

---

056
positively identified, the data must comply with the
criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility,
professional judgement should be used to determine
if instrument cross-contamination has affected any
positive compound identification.

11.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

11.1 If Tentatively Identified Compound were required for this
project, are all Tentatively Identified Compound reporting forms
present; and do listed TICs include scan number or retention
time, estimated concentration and a qualifier? [ ] [ ] [ ]

NOTE: Add "N" qualifier to all TICs which have CAS
number, if missing.

NOTE: Have the project officer/appropriate official check the
project plan to determine if lab was required to identify
non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

11.2 Are the mass spectra for the tentatively identified compounds
and associated "best match" spectra included in the sample
package for each of the following:

a. Samples and/or fractions as appropriate [ ] [ ] [ ]

b. Blanks [ ] [ ] [ ]

ACTION: If any TIC data are missing, take action specified
in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a
CAS#.

NOTE: If TICs are present in the associated blanks take
action as specified in section 3.2 above.
11.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)?  

ACTION:  1. Flag with "R" any target compound listed as a TIC.  
           2. Make sure all rejected compounds are properly reported if they are target compounds.

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?  

11.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?  

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO₂ (M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits  

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found?  

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be
reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL’s adjusted to reflect sample dilutions and, for soils, sample moisture? YES [ ] NO [ ] N/A [ ]

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC accedence dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration? YES [ ] NO [ ] N/A [ ]

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above.

14.0 GC/MS Initial Calibration (CLP Form VI Equivalent)
14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction?  

**ACTION:** If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

**ACTION:** If the percent relative standard deviation (% RSD) is > 20%, (8000C-39) qualify positive results for that analyte "J". When % RSD > 90%, qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.2 Are all average RRFs > 0.050?  

**NOTE:** (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

- Chloromethane: 0.10
- 1,1-Dichloroethane: 0.10
- Bromoform: 0.10
- Chlorobenzene: 0.30
- 1,1,2,2-Tetrachloroethane: 0.30

**ACTION:** Circle all outliers with red pencil.

**ACTION:** For any target analyte with average RRF < 0.05, or for the requirements for the 5 compounds in 14.2 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration.

**NOTE:** (Method Requirement) For the following CCC compounds, the %RSD values must be ≤ 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment.
USEPA Region II
SW846 Method 8260B VOA
Date: August 2008
SOP: HW-24, Rev. 2

YES NO N/A

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

**ACTION:** Circle all outliers with a red pencil.

**ACTION:** If the % RSD is > 20.0%, or > 30% for the 6 compounds in 14.3 above, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

**NOTE:** The above data qualification action applies regardless of method requirements.

**NOTE:** Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF?

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.)

**ACTION:** Circle errors with a red pencil.

**ACTION:** If errors are large, take action as specified in section 3.2 above.

15.0 GC/MS Calibration Verification (CLP Form VII Equivalent)
15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest? [ ] [ ] [ ]

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument? [ ] [ ] [ N/A ]

**ACTION:** List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

**ACTION:** If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

15.3 Was the % D determined from the calibration verification determined using RRF or CF? [ ] [ ] [ ]

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.

15.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2). [ ] [ ] [ ]

**NOTE:** (Method Requirement) For the following CCC compounds, the %D values must be ≤ 20.0%. If %D values reported are > 20.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride
ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

15.5 Do any volatile compounds have a RRF < 0.05? YES NO N/A

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

<table>
<thead>
<tr>
<th>Compound</th>
<th>RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloromethane</td>
<td>0.10</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>0.10</td>
</tr>
<tr>
<td>Bromoform</td>
<td>0.10</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>0.30</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>0.30</td>
</tr>
</tbody>
</table>

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, or < the requirements for the 5 compounds is section 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard (IS) areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to + 100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)? YES NO N/A
ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area Lower Limit</th>
<th>Area Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
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<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when the associated IS are counts area > + 100%.

3. If the IS area is below the lower limit (< - 50%), qualify all associated non-detects (U-values) "J".

4. If extremely low area counts are reported (< - 25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.
17.0 Field Duplicates

17.1 Were any field duplicates submitted for volatile analysis?

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.

VWAI-MW07-0512
VWAI-MW07P-0512 >no qual, see attached sheet
**DataQual**

**Initial Calibration Date:** 5/23/2012

**RRF and %RSD Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Area of Compound</th>
<th>Area of Internal STD</th>
<th>Conc. of Internal STD</th>
<th>Conc. of Compound</th>
<th>Calculated RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-dichloroethane</td>
<td>0.2960</td>
<td>743143</td>
<td>626681</td>
<td>50</td>
<td>200</td>
<td>0.296</td>
</tr>
</tbody>
</table>

**RRF of STD 1** 0.9470  
**RRF of STD 2** 0.9830  
**RRF of STD 3** 0.8970  
**RRF of STD 4** 0.8660  
**RRF of STD 5** 0.7840  
**RRF of STD 6** 0.9050  
**Calculated % RSD** 7.7

**Continuing Calibration File ID:** 5/25/2012

**RRF and %D Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Area of Compound</th>
<th>Area of Internal STD</th>
<th>Conc. of Internal STD</th>
<th>Conc. of Compound</th>
<th>Calculated RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-dichloroethylene</td>
<td>0.262</td>
<td>165930</td>
<td>632646</td>
<td>50</td>
<td>50</td>
<td>0.262</td>
</tr>
</tbody>
</table>

**Average RRF** 0.301  
**Calibration Check RRF** 0.319  
**Calculated % D** -6.0
FIELD DUPLICATE SAMPLE SUMMARY

Sample ID: VWAI-MW07-0512
Duplicate Sample ID: VWAI-MW07P-0512

Water: RPD > 20%
Soil: RPD > 30%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>benzene</td>
<td>2.9</td>
<td>2.8</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>

COMMENTS: No qualifications required

* one of the results below the LOD
The concentration of this analyte exceeds the calibration range of the instrument.

Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

**I. PACKAGE COMPLETENESS AND DELIVERABLES**

**CASE NUMBER:** L1093

**LAB:** Spectrum

**SITE NAME:** Vieques AOC I CTO-083

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format?

ACTION: If not, note the effect on review of the data in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present?

2.2 Are case number and SDG number(s) contained in the narrative or cover letter?
II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples?

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all non-detects data are qualified as unusable (R), and detects are flagged "J".

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any semivolatile technical holding times, determined from date of collection to date of extraction, been exceeded?

Continuous extraction of water samples for semivolatile analysis must be started within 7 days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within
40 days of the date of extraction.

Table of Holding Time Violations

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Matrix</th>
<th>Date Sampled</th>
<th>Date Lab Received</th>
<th>Date Extracted</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tbody>
</table>

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).
3.0 Surrogate Recovery (Form II/ Equivalent)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water
   - (Marked No)

b. Low/Med Soil
   - (Marked No)

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water
   - (Marked Yes)

b. Low/Med Soil
   - (Marked Yes)

ACTION: If CLP deliverables are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk?

- (Marked No)

ACTION: Circle all outliers in red.

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank (Reviewer should use lab in house recovery limits. Use surrogate recovery limits from USEPA National Functional Guidelines January 2005 page 130, if in house limits are not available. See Method 80008-43 or 80000C-24).

- (Marked Yes)

Note: Examine lab in house limits for reasonableness.

If yes, were samples re-analyzed?
- (Marked No)
LAB NAME: SPECTRUM ANALYTICAL, INC.  
LAB CODE: MITKEM  
CASE NO.: L1093  
MOD. REF NO.:  
SDG NO.: SL1093

<table>
<thead>
<tr>
<th>EPA SAMPLE NO.</th>
<th>SDMC1 (NBZ) #</th>
<th>SDMC2 (FBP) #</th>
<th>SDMC3 (TPH) #</th>
<th>TOT OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td>01 MB-66318</td>
<td>75</td>
<td>72</td>
<td>77</td>
<td>0</td>
</tr>
<tr>
<td>02 LCS-66318</td>
<td>72</td>
<td>73</td>
<td>73</td>
<td>0</td>
</tr>
<tr>
<td>03 MB-66345</td>
<td>80</td>
<td>76</td>
<td>78</td>
<td>0</td>
</tr>
<tr>
<td>04 LCS-66345</td>
<td>77</td>
<td>78</td>
<td>80</td>
<td>0</td>
</tr>
<tr>
<td>05 LCSD-66345</td>
<td>79</td>
<td>75</td>
<td>85</td>
<td>0</td>
</tr>
<tr>
<td>06 VWAI-EB01-052312</td>
<td>58</td>
<td>59</td>
<td>46 *</td>
<td>1</td>
</tr>
<tr>
<td>07 VWAI-MW04-0512</td>
<td>58</td>
<td>59</td>
<td>30 *</td>
<td>1</td>
</tr>
<tr>
<td>08 VWAI-MW07-0512</td>
<td>60</td>
<td>63</td>
<td>34 *</td>
<td>1</td>
</tr>
<tr>
<td>09 VWAI-MW07P-0512</td>
<td>56</td>
<td>62</td>
<td>43 *</td>
<td>1</td>
</tr>
<tr>
<td>10 VWAI-MW05-0512</td>
<td>73</td>
<td>65</td>
<td>22 *</td>
<td>1</td>
</tr>
<tr>
<td>11 VWAI-MW05-0512MS</td>
<td>78</td>
<td>69</td>
<td>18 *</td>
<td>1</td>
</tr>
<tr>
<td>12 VWAI-MW05-0512MSD</td>
<td>79</td>
<td>72</td>
<td>17 *</td>
<td>1</td>
</tr>
</tbody>
</table>

SDMC1 (NBZ) = Nitrobenzene-d5  
SDMC2 (FBP) = 2-Fluorobiphenyl  
SDMC3 (TPH) = Terphenyl-d14

QC LIMITS

- SDMC1 (NBZ) = Nitrobenzene-d5: (40-110)
- SDMC2 (FBP) = 2-Fluorobiphenyl: (50-110)
- SDMC3 (TPH) = Terphenyl-d14: (50-135)

* Column to be used to flag recovery values
* Values outside of contract required QC limits
D DMC diluted out
Were method blanks re-analyzed?

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e., either base-neutral or acid compounds):

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".
2. Non-detects for that fraction should be qualified as unusable (R).

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II?

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document.
effect in data assessments.

4.0 Matrix Spikes (Form III/Equivalent)

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate/or duplicate unspiked Sample recoveries been listed on the Recovery Form (Form III)?

YES NO N/A

NOTE: Method 3500B/page 4 states the spiking compounds:

Base/neutrals
1,2,4-Trichlorobenzene
Acenaphthene
2,4-Dinitrotoluene
Pyrene
N-Nitroso-di-n-propylamine
1,4-Dichlorobenzene

Acids
Pentachlorophenol
Phenol
2-Chlorophenol
4-Chloro-3-methylphenol
4-Nitrophenol

Note: Some projects may require the spiking of specific compounds of interest.

Note: See Method 8270D-sec 8.4.2 for deciding on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate. If samples are expected to contain target analytes, then laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratory should use a matrix spike and matrix spike duplicate pair.

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water

b. Low Solid

c. Med Solid
ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. It may be necessary to contact the lab to obtain the required data.

NOTE: If the data has not been reported on CLP equivalent form, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

4.3 Were matrix spikes performed at concentration equal to 100ug/L for acid compounds, and 200ug/l for base compounds (Method 3500B-4), or those specified in project plan.

4.4 How many semivolatile spike recoveries are outside Laboratory in house MS/MSD recovery limits (use recovery limits values in Method 8270D-43&44 Table 6 if in house values not available).

Water

2 out of 6

Solids

____ out of ____
### 3C - FORM III SV-1
WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

**Lab Name:** SPECTRUM ANALYTICAL, INC.  
**Lab Code:** MITKEM  
**Case No.:** L1093  
**Contract:**  
**Mod. Ref No.:**  
**SDG No.:** SL1093

Matrix Spike - EPA Sample No.: VWAI-MW05-0512

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>SPIKE ADDED CONCENTRATION (ug/L)</th>
<th>SAMPLE CONCENTRATION (ug/L)</th>
<th>MS CONCENTRATION (ug/L)</th>
<th>MS %REC</th>
<th>LIMITS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Naphthalene</td>
<td>50.0000</td>
<td>1.2732</td>
<td>35.7388</td>
<td>69</td>
<td>40-100</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>50.0000</td>
<td>11.0377</td>
<td>67.3900</td>
<td>71</td>
<td>45-105</td>
</tr>
<tr>
<td>Bis[2-ethylhexyl]phthalate</td>
<td>50.0000</td>
<td>0.0000</td>
<td>18.4985</td>
<td>37 %REC</td>
<td>40-125</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>SPIKE ADDED CONCENTRATION (ug/L)</th>
<th>MSD CONCENTRATION (ug/L)</th>
<th>MSD %REC</th>
<th>% RPD</th>
<th>QC LIMITS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Naphthalene</td>
<td>50.0000</td>
<td>38.1297</td>
<td>74</td>
<td>7</td>
<td>0-40 40-100</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>50.0000</td>
<td>48.7442</td>
<td>75</td>
<td>6</td>
<td>0-40 45-105</td>
</tr>
<tr>
<td>Bis[2-ethylhexyl]phthalate</td>
<td>50.0000</td>
<td>14.7979</td>
<td>30 %REC</td>
<td>22</td>
<td>0-40 40-125</td>
</tr>
</tbody>
</table>

**COMMENTS:**

* Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: 0 out of 3 outside limits
Spike Recovery: 2 out of 6 outside limits
4.5 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

<table>
<thead>
<tr>
<th>Water</th>
<th>Solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 out of 3</td>
<td>___ out of ___</td>
</tr>
</tbody>
</table>

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a Laboratory Control Sample (LCS) analyzed with each analytical batch?

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 Blanks (Form IV/Equivalent)

5.1 Is the Method Blank Summary (Form IV) present? [ ]

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch? [ ]

5.3 Has a method blank been analyzed either after
the calibration standard or at any other time
during the analytical shift for each GC/MS system
used?

ACTION: If any method blank data are missing, call
lab for explanation/resubmittal. If not
available, use professional judgement to
determine if the associated sample data
should be qualified.

5.4 Chromatography: review the blank raw data -
chromatograms (RICs), quant reports or data system
printouts and spectra.

Is the chromatographic performance (baseline
stability) for each instrument acceptable for
the semivolatiles?

ACTION: Use professional judgement to determine the
effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled
water blanks" are validated like any other
sample and are not used to qualify the data.
Do not confuse them with the other QC blanks
discussed below.

6.1 Do any method/instrument/reagent blanks have
positive results for target analytes and/or TICs?
When applied as described below, the contaminant
concentration in these blanks are multiplied by
the sample dilution factor and corrected for
percent moisture where necessary.

6.2 Do any field/rinse/ blanks have positive results
for target analytes and/or TICs (if required,
see section 10 below)?
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).
### Blank Action for Semivolatile Analyses

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>&lt; CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>= CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>&gt; CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and &lt; blank contamination</td>
<td></td>
<td>Report concentration of sample with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and ≥ blank contamination</td>
<td></td>
<td>No qualification required</td>
</tr>
</tbody>
</table>

**NOTE:** Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

**NOTE:** If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required.

6.3 Are there field/rinse/equipment blanks associated with every sample?

**ACTION:** For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

6.4 Was a instrument blank analyzed after each sample/dilution which contained a target compound
that exceeded the initial calibration range.

6.5 Does the instrument blank have positive results for target analytes and/or TICs?

Note: Use professional judgement to determine if carryover occurred and qualify analytes accordingly.

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270D? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 \text{ m} \times 0.25 \text{ mm ID} (or 0.32 \text{ mm ID}), silicone-coated, fused silica, capillary column.

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (Form V/Equivalent)

8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)?

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20\% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5
8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift?  

8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?  

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.  

<table>
<thead>
<tr>
<th>DATE</th>
<th>TIME</th>
<th>INSTRUMENT</th>
<th>SAMPLE NUMBERS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.  

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).  

8.4 Have the ion abundances been normalized to m/z 198?  

8.5 Have the ion abundance criteria been met for each instrument used?  

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).
ACTION: If ion abundance criteria are not met, take action specified in section 3.2

8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)  [ ] [ ] [ ]

8.7 Have the appropriate number of significant figures (two) been reported?

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

8.8 Are the spectra of the mass calibration compound acceptable?

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

9.0 Target Analytes

9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate  [ ] [ ] [ ]
b. Matrix spikes and matrix spike duplicates  [ ] [ ] [ ]
c. Blanks  [ ] [ ] [ ]

9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270D-14)?  [ ] [ ] [ ]
ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

a. Samples and/or fractions as appropriate

b. Matrix spikes and matrix spike duplicates (Mass spectra not required)

c. Blanks

ACTION: If any data are missing, take action specified in 3.2 above.

9.4 Are the response factors shown in the Quant Report?

9.5 Is chromatographic performance acceptable with respect to:

Baseline stability?

Resolution?

Peak shape?

Full-scale graph (attenuation)?

Other: __________________________

ACTION: Use professional judgement to determine the acceptability of the data.

9.6 Are the lab-generated standard mass spectra of identified semivolatile compounds present for
each sample?

**ACTION:** If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

**ACTION:** Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.7, 9.8, and 9.9.

9.8 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

9.9 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?

**ACTION:** When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.
10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form Is, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "IN" qualifier?

NOTE: Review sampling reports to determine if the lab was required to identify non target analytes (refer to section 7.6.2, page 82700-21).

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate

b. Blanks

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "IN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)?

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the
sample mass spectrum?

10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R."

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and, for soils, sample moisture?
ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration?  

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI/Equivalent)

13.1 Is the Initial Calibration Form (Form VI/ Equivalent) present and complete for the semivolatile fraction?  

ACTION: If any calibration forms or standard row data are missing, take action specified in 3.2 above.

13.2 Are all base neutral or acid RRFs > 0.050?
Check the average RRFs of the four System Performance Check Compounds (SPCCs):
N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. These compounds must have average RRFs greater than or equal to 0.05 before running samples and should not show any peak tailing.

**ACTION:** Circle all outliers in red.

**ACTION:** For any target analyte with average RRF < 0.05

1. "R" all non-detects;
2. "J" all positive results.

13.3 Are response factors for base neutral or acid target analytes stable over the concentration range of the calibration (% Relative standard deviation [%RSD] < 20.0%)?

**NOTE:** The % RSD for each individual Calibration Check Compound (CCC, Method 8270D-40 see Table 4) must be less than 30% before analysis can begin. If greater 30%, the lab must clean and recalibrate the instrument.

**CALIBRATION CHECK COMPOUNDS**

<table>
<thead>
<tr>
<th>Base/Neutral Fraction</th>
<th>Acid Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acenaphthene</td>
<td>4-Chloro-3-methylphenol</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>2,4-Dichlorophenol</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>2-Nitrophenol</td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>Phenol</td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>2,4,6-Trichlorophenol</td>
</tr>
</tbody>
</table>
Benzo(a)pyrene

ACTION: If the %RSD for any CCC >30% and no corrective action taken, then "J" qualify all positive hits and "UJ" qualify all non-detects.

ACTION: Circle all outliers in red.

ACTION: If the % RSD is ≥ 20.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detect results for that analyte "R," unusable. Alternatively, the lab should calculate first or second order regression fit of the calibration curve and select the fit which introduces the least amount of error.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

13.4 Did the laboratory calculate the calibration curve by the least squares regression fit?

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.)

ACTION: Circle Errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.5 Do the target compounds for this SDG include Pesticides?
13.6 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%?

ACTION: If DDT percent breakdown exceeds 20%:

   i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

   ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "IN".

14.0 GC/MS Calibration Verification (Form VII/Equivalent)

14.1 Are the Calibration Verification Forms (Form VII) present and complete for all compounds of interest?

14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis,
call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

14.3 Do any of the SPCCs have an RRF < 0.05?

If YES, make a note in data assessment if the lab did not take corrective action specified in section 7.4.4, page 82700-18.

14.4 Do any of the CCCs have a %D between the initial and continuing RRF which exceeds 20.0%?

ACTION: If yes, make a note in data assessment.

14.5 Do any semivolatile compounds have a % Difference (%D) between the initial and continuing RRF which exceeds 20.0%?

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is above 90%, qualify all non-detects for that analyte as "R", unusable.

14.6 Do any semivolatile compounds have a RRF < 0.05?

ACTION: Circle all outliers in red.

ACTION: If RRF < 0.05, qualify as unusable ("R") associated non-detects and "J" associated positive values.

14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more).
ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area</th>
<th>LowerLimit</th>
<th>Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
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</tr>
</tbody>
</table>

(Note: Check Table 5, 8270D-41 for associated analytes.

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.)
iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Laboratory Control Samples (LCS)

16.1 Were any LCS samples run in order to verify analytes which failed criteria for spike recovery?

16.2 Did the lab spike LCS sample spiked with the same analytes and the same concentrations as the matrix spike?

16.3 Were the mean and standard deviation of all analytes within the QC acceptance ranges as shown in Table 6, 8270D-43?

ACTION: If the recovery of any analyte falls out of the designated range, the analytical results for that compound is suspect and should be qualified "J" in the unspiked samples.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for semivolatile analysis?
ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

VWAI-MW07-0512  No qualifications

VWAI-MW07P-0512  plE attached sheet
DataQual

Initial Calibration Date: 6/1/2012
RRF and %RSD Calculations:

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Area of Compound</th>
<th>Area of Internal STD</th>
<th>Conc. of Internal STD</th>
<th>Conc. of Compound</th>
<th>Calculated RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>naphthalene</td>
<td>1.006</td>
<td>385375</td>
<td>191614</td>
<td>40</td>
<td>80</td>
<td>1.006</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>RRF of STD 1</th>
<th>RRF of STD 2</th>
<th>RRF of STD 3</th>
<th>RRF of STD 4</th>
<th>RRF of STD 5</th>
<th>RRF of STD 6</th>
<th>RRF of STD 7</th>
<th>Calculated % RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-methyl naphthalene</td>
<td>5.0</td>
<td>0.711</td>
<td>0.824</td>
<td>0.771</td>
<td>0.734</td>
<td>0.734</td>
<td>0.725</td>
<td>0.759</td>
<td>5.1</td>
</tr>
</tbody>
</table>

RRF and %D Calculations:

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Area of Compound</th>
<th>Area of Internal STD</th>
<th>Conc. of Internal STD</th>
<th>Conc. of Compound</th>
<th>Calculated RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>bis(2-ethylhexyl)phthalate</td>
<td>0.599</td>
<td>181980</td>
<td>486010</td>
<td>40</td>
<td>25</td>
<td>0.599</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Average RRF</th>
<th>Calibration Check RRF</th>
<th>Calculated % D</th>
</tr>
</thead>
<tbody>
<tr>
<td>naphthalene</td>
<td>0.7</td>
<td>1.056</td>
<td>1.063</td>
<td>-0.7</td>
</tr>
</tbody>
</table>
FIELD DUPLICATE SAMPLE SUMMARY

Sample ID: VWAI-MW07-0512
Duplicate Sample ID: VWAI-MW07P-0512

Water: RPD > 75%
Soil: RPD > 100%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>naphthalene</td>
<td>3.3</td>
<td>3.2</td>
<td>3</td>
</tr>
<tr>
<td>2-methylnaphthalene</td>
<td>3.4</td>
<td>3.3</td>
<td>3</td>
</tr>
</tbody>
</table>

* one values below LOD
  only values above LOD listed

COMMENTS: No qualifications required
<table>
<thead>
<tr>
<th>A.1.1 Contract Compliance Screening Report</th>
<th>Present?</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ACTION:</td>
<td>If no, contact RSCC/PO.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.2 Record of Communication (from RSCC)</th>
<th>Present?</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ACTION:</td>
<td>If no, request from the RSCC.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.3 Sampling Trip Report</th>
<th>Present and complete?</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ACTION:</td>
<td>If no, contact RSCC/PO.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.4 Chain of Custody/Sample Traffic Report</th>
<th>Present?</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Legible?</td>
<td>YES</td>
<td>NO</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>Signature of sample custodian present?</td>
<td>YES</td>
<td>NO</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>ACTION:</td>
<td>If no, contact RSCC/WAM/PO.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>A.1.5 Cover Page</th>
<th>Present?</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?</td>
<td>YES</td>
<td>NO</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Do the sample identification numbers on the Cover Page agree with sample identification numbers on: (a) Traffic Report Sheet?</td>
<td>YES</td>
<td>NO</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>(b) Form I's?</td>
<td>![Yes/No/N/A]</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>---</td>
<td>---</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?</td>
<td>[ ] [ ] [ ]</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

### A.1.6 SDG Narrative, DC-1 & DC-2 Form

<table>
<thead>
<tr>
<th></th>
<th>![Yes/No/N/A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Is the SDG Narrative present?</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td>Is Sample Log-In Sheet (Form DC-1) present and complete?</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td>Is Complete SDG Inventory Sheet (Form DC-2) present and complete?</td>
<td>[ ] [ ] [ ]</td>
</tr>
</tbody>
</table>

**ACTION:**
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

### A.1.7 Form I to XV

#### A.1.7.1 Are all the Form I through Form XV labeled with:

<table>
<thead>
<tr>
<th></th>
<th>![Yes/No/N/A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laboratory Name?</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td>Laboratory Code?</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td>RAS/Non-RAS Case No.?</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td>SDG No.?</td>
<td>[ ] [ ] [ ]</td>
</tr>
</tbody>
</table>
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Contract No.?  [✓]  [-]  [N/A]

**ACTION:**
If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

A.1.7.2
After comparing values on Forms I-IX against the raw data, do any computation/transcription errors exceed 10% of the reported values on the Forms for:

(a) all analytes analyzed by ICP-AES?  [✓]
(b) all analytes analyzed by ICP-MS?  [-]
(c) Mercury?  [✓]
(d) Cyanide?  [✓]

**ACTION:**
If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 **Raw Data**
Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 **Digestion/Distillation Log**

Digestion Log for ICP-AES (Form XII) present?  [✓]

Digestion Log for ICP-MS (Form XII) present?  [✓]

Digestion Log for mercury (Form XII) present?  [✓]

Distillation Log for cyanide (Form XII) present?  [✓]

Are pH values for metals and
cyanide reported for each aqueous sample? 

Are percent solids calculations present for soils/sediments? 

Are preparation dates present on the sample preparation logs/bench sheets? 

**NOTE:**
Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.

A.1.8.2 Is the analytical instrument real-time printouts present for:
- ICP-AES? 
- ICP-MS? 
- Mercury? 
- Cyanide? 

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:
- Legible? 
- Properly labeled? 

Are all field samples, QC samples and field QC samples present on:
- Digestion/Distillation log? 
- Instrument Printouts? 

**ACTION:**
If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.
A.1.9 **Technical Holding Times:** (Aqueous and soil samples)

(Examine sample Traffic Reports and digestion/distillation logs to determine the holding time from the sample collection date to the sample preparation date.)

A.1.9.1 Cyanide distillation (14 days) exceeded? [ ] [✓]

Mercury analysis (28 days) exceeded? [ ] [✓]

Other Metals analysis (180 days) exceeded? [ ] [✓]

**ACTION:**
If yes, reject (R) and red-line non-detects and flag as estimated (J) results ≥ MDL even if sample(s) was preserved properly.

**NOTE:**
In addition to qualifying the data, a list of all samples and analytes which exceeded the holding times must be prepared. Report for each sample the number of days that were exceeded. (Subtract the sample collection date from the sample preparation date). Attach this list to the data review narrative.

A.1.9.2 Is pH of aqueous samples for:

Metals Analysis ≤ 2? [✓] [ ]

Cyanide Analysis ≥ 12? [ ] [✓]

**ACTION:**
If no for any of the above, flag non-detects as "R" and detects as "J".

A.1.9.3 Is the cooler temperature ≤ 10°C? [✓]

**ACTION:**
If cooler temperature is >10°C, flag non-detects as "UJ" and detects as "J".

A.1.10 **Final Data Correctness - Form I**

A.1.10.1 Are Form I's for all samples
present and complete?

**ACTION:**
If no, prepare Telephone Record Log and contact CLP PO/TOPO for submittal from the laboratory.

A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

Is the calculation error less than 10% of the correct result? [✓] [ ] [ ]

Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)? [X] [ ] [ ]

Are results on Form I's reported by correct significant figures? [✓] [ ] [ ]

Are soil sample results on Form I's corrected for percent solids? [✓] [ ] [ ]

Are all "less than MDL" values reported by the CRQ's and coded with "U"? [✓] [ ] [ ]

Are values less than the CRQ's but greater than or equal to the MDL's flagged with "J"? [✓] [ ] [ ]

Are appropriate contractual quality control and Method qualifiers used? [✓] [ ] [ ]

**ACTION:**
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3 Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data? [✓] [ ] [ ]

Was a brief physical description: [ ] [ ] [ ]
of the samples before and after digestion given on the Form I's? 

Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I? 

**ACTION:**
If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

### A.1.11 Initial Calibration

**A.1.11.1** Is a record of at least 2 point (A blank and a standard) calibration present for ICP-AES analysis? 

**A.1.11.2** Is a record of at least 2 point (a blank and a standard) calibration present for ICP-MS analysis? 

**A.1.11.3** Is a record of at least 5 point calibration (a blank & 4 standards) present for Hg analysis? 

**A.1.11.4** Is a record of at least 4 point calibration (a blank & 4 standards) present for cyanide? 

**ACTION:**
If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).

Is one initial calibration standard at the CRQL level for cyanide and mercury? 

**ACTION:**
If no, write in the Contract Problem/Non-Compliance Section of the Data Review Narrative.

**A.1.11.2** Is the curve correlation coefficient ≥ 0.995 for:
Mercury Analysis? [ ] [ ] [✓]
Cyanide Analysis? [ ] [✓] [ ]
ICP-AES (more than 2 point Calib.)? [✓] [ ] [ ]
ICP-MS (more than 2 point calib.)? [ ] [ ] [✓]

**ACTION:**
If no, qualify the associated sample results ≥ MDL as estimated "J" and non-detects as "UJ".

**NOTE:**
The correlation coefficient shall be calculated by the data validator using standard concentrations and the corresponding instrument response (e.g. absorbance, peak area, peak height, etc.).

---

**A.1.12 Initial and Continuing Calibration Verification - Form IIIA**

**A.1.12.1** Present and complete for every metal and cyanide? [✓] [ ] [ ]
Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte? [✓] [ ] [ ]

**ACTION:**
If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.

**A.1.12.2** Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent? [ ] [ ] [✓]

**ACTION:**
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

**A.1.12.3** Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples? [ ] [ ] [✓]
A.1.12 Action: If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative and qualify results ≥ MDL as estimated (J).

A.1.12.2 Circle on each Form IIA all percent recoveries that are outside the contract windows.

Are ICV/CCVs within control limits for:
- Metals - 90-110%R?
- Hg - 80-120%R?
- Cyanide - 85-115%R?

A.1.13 CRQL Standard Analysis - Form IIB
A.1.13.1 For each ICP-AES run, was a CRI
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(CRQL or MDL when MDL > CRQL)
standard analyzed?
(Note: CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)

For each ICP-MS run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis?

For each mercury run, was a CRQL standard analyzed?

For each cyanide run, was a CRQL standard analyzed?

ACTION:
If no for any of the above, write this deficiency in the Contract Problems/Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL) as J and non-detects UJ.

The affected ranges are:
ICP-AES Analysis - *True Value ± CRQL
ICP-MS Analysis - *True Value ± CRQL
Mercury Analysis - *True Value ± CRQL
Cyanide Analysis - *True Value ± CRQL

* True value of the CRQL Standard

A.1.13.2 Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis?

ACTION:
If no, write in the Contract Problem/Non-Compliance Section of the "Data Review Narrative".

A.1.13.3 Circle on each Form IIB all percent recoveries that are outside the acceptance windows.

[ ] [ ] [ ]

CRI Std was not run but a Std at LOQ was analyzed for Mn.
Is the CRQL standard within control limits for:

- Metals (ICP-AES/ICP-MS) - 70 - 130%? 
  YES   NO   N/A

- Mercury - 70 - 130%? 
  YES   NO   N/A

- Cyanide - 70 - 130%? 
  YES   NO   N/A

**ACTION:**
If no, flag detects <2xCRLQ as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag(J) only detects <2xCRLQ if the recovery is between 131% and <180%. If the recovery is less than 750%, reject(R) and red-line non-detects and detects < 2xCRLQ, and flag (J) detects between 2xCRLQ and ICV/CCV. Reject and red-line only detects <2xCRLQ and flag (J) detects ≥ 2xCRLQ but < ICV/CCV if the recovery is > 180%.

**NOTE:**
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard.
2. Flag (J) or reject (R) only the final sample results on Form I's when Sample raw data are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

**A.1.14 Initial and Continuing Calibration Blanks - Form III**

**A.1.14.1** Present and complete for all the instruments used for the metals and cyanide analyses?

- YES   NO   N/A

Was an initial Calibration Blank analyzed after ICV?

- YES   NO   N/A

Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?

- YES   NO   N/A

Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by
using MDLs from direct analysis (Preparation Method "NP1")? [___] [___] [✓]
(Check Form III against the raw data)

**ACTION:**
if no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".

A.1.14.2 Circle with red pencil on each Form III all Calib. Blank values that are:

- \( \geq \text{MDL} \) but \( \leq \text{CRQL} \)
- \( > \text{CRQL} \)

A.1.14.2.1 When MDL \( < \text{CRQL} \), is any Calib. Blank value \( \geq \text{MDL} \) but \( \leq \text{CRQL} \)? [___] [✓] [___]

**ACTION:**
if yes, change sample results \( \geq \text{MDL} \) but \( \leq \text{CRQL} \) to the CRQL with a "U".
Do not qualify non-detects.

A.1.14.2.2 When MDL \( < \text{CRQL} \), is any Calib. Blank value \( > \text{CRQL} \)? [___] [✓] [___]

**ACTION:**
if yes, reject (R) and red line the associated sample results \( > \text{CRQL} \) but \( < \text{ICB/CCB Blank Result} \). Flag as "J" detects \( > \text{ICB/CCB Blank value} \) but \( < 10 \times \text{ICB/CCB value} \). Change the sample results \( \geq \text{MDL} \) but \( \leq \text{CRQL} \) to CRQL with a "U".

A.1.14.2.3 Is any Calibration Blank value below the negative CRQL? [___] [✓] [___]

**ACTION:**
if yes, flag (J) as estimated all associated sample results \( \geq \text{CRQL} \) but \( < 10 \times \text{CRQL} \).

**NOTE:**
1. For ICB that does not meet the technical QC Criteria, apply the action to all samples.
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A.1.15 Preparation Blank - FORM III
NOTE: The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1 Was one Preparation Blank prepared with and analyzed for:

- Each Sample Delivery Group (SDG)? [ ] [ ] [ ]
- Each batch of the SDG samples digested/distilled? [ ] [ ] [ ]
- Each matrix type? [ ] [ ] [ ]
- All instruments used for metals and cyanide analyses? [ ] [ ] [ ]

ACTION:
If no for any of the above, flag as estimated (J) all the associated positive data <10xMDL for which the Preparation Blank was not analyzed.

NOTE:
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated (J), but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

$\geq$ MDL but $\leq$ CRQL, and $>$ CRQL

A.1.15.2.1 When MDL $<$ CRQL, is any preparation blank value $\geq$ MDL but $\leq$ CRQL? [ ] [ ]

ACTION:
If yes, change sample result $\geq$ MDL
but $\leq$ CRQL to CRQL with a "U".

A.1.15.2.2 When the MDL $\leq$ CRQL, is any Preparation Blank value greater than its CRQL?  

If yes, is the Prep. Blank value greater than the value of the associated Field Blank collected and analyzed with the SDG samples?  

If yes, is the lowest concentration of that analyte in the associated samples less than 10 times the Preparation Blank value?  

**ACTION:**  
If yes, reject (R) and red-line all associated sample results greater than the CRQL but less than the Prep. Blank value. Flag as "U" detects $>\text{Prep. Blank value}$ but $<10\times\text{Prep. Blank}$.  
If the sample result $\geq$ MDL but $\leq$ CRQL, replace it with CRQL-$U$.  
If the Prep. Blank value is less than the same analyte value in the Field Blank, do not qualify the sample results due to the Prep. Blank criteria.

**NOTE:**  
Convert soil sample result to mg/Kg on wet weight basis to compare with the soil Prep. Blank result on Form III.

A.1.15.2.3 Is the Prep. Blank concentration below the negative CRQL?  

**ACTION:**  
If yes, flag (J) all associated sample results less than $10\times\text{CRQL}$. Qualify non-detects as estimated (UJ).

A.1.15.2.4 When the MDL is greater than the CRQL, is the preparation blank concentration on Form III greater than two times the MDL?  

**ACTION:**
If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.

A.1.16 **ICP-AES/ICP-MS Interference Check Sample (ICS) - Form IV**

**NOTE:** Not required for CN, Hg, Al, Ca, Fe and Mg.

A.1.16.1 Present and complete? [ ] [ ] [ ]

Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples? [ ] [ ] [ ]

Was ICS analyzed at the beginning of the ICP-MS analytical run? [ ] [ ] [ ]

**ACTION:**
If no, flag as estimated (J) all sample results.

A.1.16.2 **ICP-AES Method**

A.1.16.2.1 **ICSA Solution:**
For ICP-AES, are the ICSA "Found" analyte values within the control limits + of CRQL of the true/established mean value? [ ] [ ] [ ]

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (µg/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV? [ ] [ ] [ ]

**ACTION:**
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:
Flag (J) as estimated only sample results ≥ MDL.
A.1.16.2.3 ICSAB Solution

For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value?

If no, for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (μg/L or MG/KG) greater than or equal to its respective concentration in the ICSAB Solution on Form IV?

**ACTION:**
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79%, qualify sample results ≥ MDL as “J” and non-detects as “UJ”. Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.

A.1.16.3 ICP-MS Method

A.1.16.3.1 ICSA Solution:

For ICP-MS, are the ICSA “Found” analyte values within the control limits of ±CRQL of the true/established mean value?

**ACTION:**
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated only sample results ≥ MDL if the ICSA “Found” value is greater than (True value+CRQL). Do not qualify non-detects.
If the ICSA “Found” value is less than (True value-CRQL), flag the associated sample detects as “J” and non-detects as “UJ”. 

---

-29-
A.1.16.3.3 ICSAB Solution
For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater?

**ACTION:**
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but < 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only detects (≥ MDL).

A.1.17 Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A

**Note:** Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only)

A.1.17.1 Was Matrix Spike analysis performed:

For each matrix type?

For each SDG?

On one of the SDG samples?

For each concentration range (i.e., low, med., high)?

For each analytical Method (ICP-AES, ICP-MS, Hg, CN) used?

Was a spiked sample prepared and analyzed with the SDG samples?

**ACTION:**
If no for any of the above, flag as estimated (J) all the positive data for which a spiked sample was not analyzed.

**NOTE:**
If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.
A.1.17.2 Was a field blank or PE sample used for the spiked sample analysis?

**ACTION:**
If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.

A.1.17.3 Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.

Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?

**NOTE:**
Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.

Are results outside the control limits (75-125%) flagged with Lab Qualifier "N" on Form I's and Form VA?

**ACTION:**
If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.

A.1.17.4 Aqueous

Are any spike recoveries:

(a) less than 30%?

(b) between 30-74%?

(c) between 126-150%?

(d) greater than 150%?

**ACTION:**
If the matrix spike recovery is less than 30%, reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as “J” and non-detects...
### A.1.17.5 Soil/Sediment

Are any spike recoveries:

(a) less than 10%?
(b) between 10-74%?
(c) between 126-200%?
(d) greater than 200%?

**ACTION:**

- If yes for any of the above, proceed as follows:
  - If the matrix spike recovery is less than 10%, reject (R) and red-line all associated data (detects & non-detects);
  - If between 10-74%, qualify all associated data ≥ MDL as "J" and non-detects as "UJ";
  - If between 126-200%, flag (J) all associated data ≥ MDL as "J"; if greater than 200%, reject (R) and red-line all associated data ≥ MDL.

(Note: Replace "N" with "J" or "R" as appropriate.)

### A.1.18 Lab Duplicates - Form VI

#### A.1.18.1 Was the lab duplicate analysis performed:

- For each SDG?
- On one of the SDG samples?
- For each matrix type?
- For each concentration range (low or med.)?
- For each analytical Method (ICP-AES/ICP-MS,Hg,CN) Used?
- Was a lab duplicate prepared and analyzed with the SDG samples?
ACTION:
If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.

NOTE:
If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

A.1.18.2 Was a Field Blank or PE sample used for the Lab Duplicate analysis?

ACTION:
If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.

A.1.18.3 Circle on each Form VI all values that are:
   
   RPD > 20%, or
   
   Absolute Difference > CRQL

   Are all values within control limits (RPD ≤ 20% or absolute difference ≤ ±CRQL)?

   If no, are all results outside the control limits flagged with an "*" (Lab Qualifier) on Form VI and on all Form I's?

ACTION:
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

NOTE:
The laboratory is not required to report on Form VI the RPD when both values are non-detects.

A.1.18.4 Aqueous

A.1.18.4.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),
is any RPD > 20% but < 100%?  

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

is any RPD ≥ 100%?  

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**

If the RPD is > 20% but < 100%, flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

(NOTE: Replace “*” with “J” or “R” as appropriate.)

A.1.18.4.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate values:

> + CRQL?

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

> + 2xCRQL?

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**

If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5xCRQL as “J” and non-detects as “UJ”. If the absolute difference is > 2xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but < 5xCRQL.

(NOTE: Replace “*” with “J”, “UJ” or “R” as appropriate.)

1. If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

is any RPD ≥ 120%?

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**

If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample...
data ≥ CRQL. If the RPD is ≥ 120%, reject (R) and red-line the associated sample data ≥ CRQL.

A.1.18.5.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

\[ V > \pm 2 \times CRQL? \]
\[ V' > \pm 4 \times CRQL \]

**ACTION:**

If the absolute difference is > 2 x CRQL, flag all the associated sample results ≥ MDL but < 5xCRQL as “J” and non-detects as “UJ”. If the absolute difference is > 4xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but <5xCRQL.

**NOTE:**

1. Replace “*” with “J”, “UJ” or “R” as appropriate.
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

---

**A.1.19 Field Duplicates**

**Aqueous Field Duplicates**

**A.1.19.1** Was an aqueous Field Duplicate pair collected and analyzed? (Check Sampling Trip Report)

[ ] [ ] [ ]

**ACTION:**

If yes, prepare a Form (Appendix A.4) for each aqueous Field Duplicate pair. Report the sample and Field Duplicate results on Appendix A.4 from their respective Form I’s. Calculate and report RPD on Appendix A.4 when sample and its Field Duplicate values are both > 5xCRQL. Calculate and report the absolute difference on Appendix A.4 when at least one value (sample or duplicate) is <5xCRQL. Evaluate the aqueous Field Duplicate analysis in accordance with the
QC criteria stated in Sections A.1.19.2 and A.1.19.3.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

\[ \text{RPD} \geq 20\% \quad \text{or} \quad \text{Difference} > \pm \text{CRQL} \]

When sample and duplicate values are both \( \geq 5 \times \text{CRQL} \) (substitute MDL for CRQL when MDL > CRQL),

- is any RPD \( \geq 20\% \)? [ ] [ ] [ ]
- is any RPD \( \geq 100\% \)? [ ] [ ] [ ]

ACTION:
If the RPD is \( >20\% \) but \( <100\% \), flag (J) only the associated sample and its Field Duplicate results \( \geq \text{CRQL} \). If the RPD is \( >100\% \), reject (R) and red-line only the associated sample and its Field Duplicate result \( > \text{CRQL} \).

A.1.19.3 When the sample and/or duplicate value(s) \( <5 \times \text{CRQL} \) (substitute MDL for CRQL when MDL > CRQL),

- is the absolute difference between sample and duplicate:
  \[ \geq \pm \text{CRQL} \] [ ] [ ] [ ]
  \[ > \pm 2 \times \text{CRQL} \] [ ] [ ] [ ]

ACTION:
If the absolute difference is \( > \text{CRQL} \), flag detects \( \geq \text{MDL} \) but \( <5 \times \text{CRQL} \) as "J" and non-detects as "UJ". If the difference is \( >2 \times \text{CRQL} \), reject (R) and red-line non-detects.
and results ≥ MDL but < 5xCRQL of the sample and its Field Duplicate.

**Soil/Sediment Field Duplicates**

A.1.19.4 Was a soil field duplicate pair collected and analyzed? (Check Sampling Trip Report)

**ACTION:**

If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than 5xCRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is < 5xCRQL. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

**NOTE:**

1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and apply the criteria to qualify the results.

A.1.19.5 Circle on each Appendix A.4 all values that have:

RPD ≥ 35%, or Difference > ± 2xCRQL When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%? [___] [___] __

is any RPD ≥ 120%? [___] [___] __

**ACTION:**

If the RPD is ≥ 35% but < 120%,
flag only the associated sample and its Field Duplicate results ≥ CRQL as "J". If the RPD is ≥ 120%, reject (R) and red-line only the sample and its Field Duplicate results ≥ CRQL.

A.1.19.6 When the sample and/or duplicate value(s) <5xCRQL (substitute MOL for CRQL when MOL > CRQL), is the absolute difference between sample and Field Duplicate:

<table>
<thead>
<tr>
<th>≥ 2 x CRQL?</th>
<th>YES</th>
<th>NO</th>
</tr>
</thead>
<tbody>
<tr>
<td>≥ 4 x CRQL?</td>
<td>YES</td>
<td>NO</td>
</tr>
</tbody>
</table>

**ACTION:**
If the absolute difference is > 2xCRQL, flag Sample and its Field Duplicate results ≥ MDL but <5xCRQL as "J" and non-detects as "UJ".
If the difference is >4xCRQL, reject (R) and red-line non-detects and detects ≥ MDL but <5xCRQL of the sample and its Field Duplicate.

A.1.20 **Laboratory Control Sample (LCS) - Form VII**

A.1.20.1 Was one LCS prepared and analyzed for:

- Each SDG? [✓] [NO] [N/A]
- Each matrix type? [✓] [NO] [N/A]
- Each batch samples digested/distilled? [✓] [NO] [N/A]
- For each Method (ICP-AES, ICP-MS, Hg, CN) used? [✓] [NO] [N/A]
- Was an LCS prepared and analyzed with the samples? [✓] [NO] [N/A]

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact CLP PO or TOPO for submittal of the LCS results. Flag (J) as estimated all the data for which an LCS was not analyzed.

**NOTE:**
If only one LCS was analyzed for
### A.1.20.2 Aqueous LCS

Circle on each Form VII the LCS percent recoveries outside control limits 80-120%.

**NOTE:**
1. Use digested ICV as LCS for aqueous mercury
2. Use distilled ICV as LCS for aqueous cyanide

<table>
<thead>
<tr>
<th>LCS Recovery</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Less than 50%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Between 50% and 79%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Between 121% and 150%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Greater than 150%</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**
- If the LCS recovery is less than 50%, reject (R) and red-line all associated sample data (detects & non-detects); for a recovery between 50-79%, flag detects as "J" all non-detects as "UJ". If the LCS recovery is between 121-150%, flag only detects as "J". If the recovery is greater than 150%, reject (R) and red-line all detects.

### A.1.20.3 Solid LCS

If an analyte's MDL is equal to or greater than the true value of LCS, disregard the "Action" below for that analyte even though the LCS is out of control limits.

<table>
<thead>
<tr>
<th>LCS Found Value</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Greater than the Upper Control Limit reported on Form VII?</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**
If yes, flag (J) all the associated detects ≥ MDL as estimated (J).

Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII?

**ACTION:**
If yes, flag detects as "J" and non-detects as "UJ".

### A.1.21 ICP-AES/ICP-MS Serial Dilution - Form VIII

**NOTE:** Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 x MDL.

#### A.1.21.1 Was a Serial Dilution analysis performed:
- For each SDG?
- On one of the SDG samples?
- For each matrix type?
- For each concentration range (low or med.)?

**ACTION:**
If no for any of the above, flag as estimated (J) detects ≥ MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.

#### A.1.21.2 Was a Field Blank or PE sample used for the Serial Dilution Analysis?

**ACTION:**
If yes, flag as estimated (J) detects ≥ MDL of all the SDG samples

#### A.1.21.3 Circle on Form VIII the Percent Differences (%D) between sample results and its dilution results that are outside the control limits ± 10%
when initial concentrations \( \geq 50 \times \text{MDLs} \).

Are results outside the control limits flagged with an "E" (Lab Qualifier) on Form VIII and all Form I's?  

\( \text{ACTION:} \)

If no, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.21.4 Are any \( \%D \) values:

\( > 10\%? \)

\( \geq 100\%? \)

\( \text{ACTION:} \)

If the Percent Difference (\( \%D \)) is greater than 10\%, flag (J) as estimated all associated samples whose raw data \( \geq \) MDL; if the \( \%D \) is \( > 100\% \), reject (R) and red-line all associated samples with raw data \( \geq \) MDL.

\( \text{(NOTE: Replace "E" with "J" or "R" as appropriate.)} \)

A.1.22 Total/Dissolved or Inorganic/Total Analytes

A.1.22.1 Were any analyses performed for dissolved as well as total analytes on the same sample(s)?

Were any analyses performed for inorganic as well as total analytes on the same sample(s)?

\( \text{ACTION:} \)

If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic) and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:

(1) The dissolved (or inorganic) concentration is greater than total concentration, and

(2) greater than or equal to 5xMDL.

A.1.22.2 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20\%?
A.1.22.3 Is any dissolved(or inorganic) concentration greater than its total concentration by more than 50%? 

**ACTION:**
If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23 Field Blank - Form I

**NOTE:** Designate "Field Blank" as such on Form I

A.1.23.1 Was a Field/Rinsate Bank collected and analyzed with the SDG samples? 

If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL(or 2xMDL when MDL>CRQL)? 

If yes, circle the Field Blank value on Form I that is greater than the CRQL, (or 2 x MDL when MDL > CRQL). 

Is any Field Blank value greater than CRQL also greater than the Preparation Blank value? 

If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria? 

**ACTION:**
If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results) greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value, If the sample result > MDL but ≤ CRQL, replace it with CRQL-U.

If the Field Blank value is less than the
Prep. Blank value, do not qualify the sample results due to the Field Blank criteria.

**NOTE:**
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

**A.1.24 Verification of Instrumental Parameters - Form IX, XA, XB, XI**

**A.1.24.1** Is verification report present for:

- Method Detection Limits (Form IX - Annually)?
- ICP-AES Interelement Correction Factors (Form XA & XB - Quarterly)?
- ICP-AES & ICP-MS Linear Ranges (Form XI - Quarterly)?

**ACTION:**
If no, contact CLP PO/TOPO for submittal from the laboratory.

**A.1.24.2 Method Detection Limits - Form IX**

**A.1.24.2.1** Are MDLs present on Form IX for:

- All the analytes?
- All the instruments used?
- Digested and undigested samples and Calib. Blanks?
- ICP-AES and ICP-MS when both instruments are used for the same analyte?

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than 4 CRQL.
A.1.24.2.2 Is MDL greater than the CRQL for any analyte?

If yes, is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?

**ACTION:**
If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.

A.1.24.3 Linear Ranges - Form XI

A.1.24.3.1 Was any sample result higher than the high linear range for ICP-AES or ICP-MS?

Was any sample result higher than the highest calibration standard for mercury or cyanide?

If yes for any of the above, was the sample diluted to obtain the result reported on Form I?

**ACTION:**
If no, flag (J) as estimated the affected detects (≥ MDL) reported on Form I.

A.1.25 ICP-MS Tune Analysis - Form XIV

A.1.25.1 Was the ICP-MS instrument tuned prior to calibration?

**ACTION:**
If no, reject (R) and red-line all sample data for which tuning was not performed.

A.1.25.2 Was the tuning solution analyzed or scanned at least five times consecutively?

Were all the required isotopes spanning the analytical range present in the tuning solution?

Was the mass resolution within
0.1 amu for each isotope in the tuning solution? [ ] [ ] [✓]

Was %RSD less than 5% for each isotope of each analyte in the tuning solution? [ ] [ ] [✓]

**ACTION:**
If no for any of the above, qualify all results ≥ MDL associated with that Tune as estimated "J", and all non-detects associated with that Tune as "UJ".

A.1.26.1 Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)? [ ] [ ] [✓]

Were all the target analyte masses bracketed by the masses of the five internal standards? [ ] [x] [ ]

**ACTION:**
If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.

A.1.26.2 Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank? [ ] [ ] [✓]

If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed? [ ] [ ] [✓]

Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)? [ ] [ ] [✓]

**ACTION:**
If no for any of the above, flag detects as "J" and non-detects "UJ" of all the analytes with atomic masses between the atomic masses of the internal standards.
than the affected internal standard, and the
atomic mass of the internal standard heavier
than the affected internal standard.

A.1.27  **Percent Solids of Sediments**

A.1.27.1 Are percent solids in sediment(s):

< 50%?  

[ ] [ ]

**ACTION:**
If yes, qualify as estimated (J) all detects and
non-detects of a sample that has percent solids
less than 50% (i.e., moisture content greater than 50%).

**NOTE:**
Flag(J) only the sample results
that were not previously flagged
due to other QC criteria.

---

**Inorganic Data Review Narrative**

<table>
<thead>
<tr>
<th>Case#</th>
<th>Site:</th>
<th>Matrix: Soil</th>
</tr>
</thead>
<tbody>
<tr>
<td>SDG#</td>
<td>Lab:</td>
<td>Water</td>
</tr>
<tr>
<td>Sampling Team:</td>
<td>Reviewer:</td>
<td>Other</td>
</tr>
</tbody>
</table>

**A.2.1 Data Validation Flags:**
The following flags may have been applied in red by the data validator and must
be considered by the data user.

- **J** - This flag indicates the result qualified as **estimated**

- **R and Red-Line** - A red-line drawn through a sample result indicates **unusable** value.
The red-lined data are known to contain significant errors based on
documented information and must not be used by the data user.

- **U** - This data validation qualifier is applied to sample results
  ≥ MDL when associated blank is contaminated

**Fully Usable Data** - The results that do not carry "J" or "red-line" are fully **usable**.

**A.2.2 Laboratory Qualifiers:**
The CLP laboratory applies a contractual qualifier on all
**SAMPLE CALCULATION**

**EPA SAMPLE ID:** WWAI-MW05-0512  
**COMPOUND:** Manganese  
**CONCENTRATION:** 1230 ug/L  
**%Solids:** NA

Raw Data result: 1.2334 mg/L

\[
1.2334 \text{ mg/L (1000ug/1mg)} = 1233.4 \text{ ug/L}
\]

**FIELD DUPLICATE SAMPLE SUMMARY**

Note: All reported results are noted in the table below because the client requested that the MDL be used as reporting limit instead of the RL for this project. RPDs or absolute differences were calculated based on Region II guidelines: if results are >5X RL RPD is calculated, if results are <5X RL the absolute difference is calculated. Flags are applied to field duplicate pair only as follows: For RPD values - RPD ≥ 35% but <120% results are J, RPD >120%, results are R. For absolute difference values - >+/- 2X RL results are J, >+/- 4X RL results are R.

<table>
<thead>
<tr>
<th>Sample ID: none</th>
<th>Duplicate Sample ID:</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sample ID:</strong> none</td>
<td><strong>Sample Conc.</strong> 0.000</td>
</tr>
<tr>
<td>Comments: No qualifications required.</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample ID: none</th>
<th>Duplicate Sample ID:</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sample ID:</strong> none</td>
<td><strong>Sample Conc.</strong> 0.000</td>
</tr>
<tr>
<td>Comments: No qualifications required.</td>
<td></td>
</tr>
</tbody>
</table>
January 25, 2013
SDG# SL2472, Spectrum Analytical, Inc.
Vieques Island, Puerto Rico, CTO-083

Dear Ms. Dean,

The following Data Validation report is provided as requested for the parameters noted in the table below for SDG # SL2472. The data validation was performed in accordance with the SW-846 methods utilized by the laboratory, the Region II Standard Operating Procedures for the Validation of Organic Data Acquired Using SW-846 Methods (8260B-Rev 2, August 2008- SOP #HW-24 and 8270D-Rev 4, August 2008-SOP #HW-22), and professional judgment. Region II has not developed a validation checklist SOP for the methods used to assess the metals in this SDG (SW-846 method 6010B). The Region II Standard Operating Procedure for the Evaluation of Metals Data for the CLP was used as applicable for the metals data. Region II flagging conventions were used. All areas of concern are discussed in the body of the report and a summary of data qualifications is provided.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Lab ID</th>
<th>Matrix</th>
<th>VOA</th>
<th>SVOA</th>
<th>Fe, Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW04-1112</td>
<td>L2472-01</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VWAI-EB01-112812</td>
<td>L2472-02</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VWAI-MW05-1112</td>
<td>L2472-03</td>
<td>water</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VWAI-TB01-112812</td>
<td>L2472-04</td>
<td>water</td>
<td>X</td>
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<td></td>
</tr>
<tr>
<td>VWAI-MW07-1112</td>
<td>L2472-05</td>
<td>water</td>
<td>X</td>
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<td></td>
</tr>
<tr>
<td>VWAI-MW07P-1112</td>
<td>L2472-06</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
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<tr>
<td>VWAI-MW04-112912</td>
<td>L2472-07</td>
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<td></td>
</tr>
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<td>VWAI-MW04-112912</td>
<td>L2472-08</td>
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<td></td>
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<tr>
<td>VWAI-MW04-1112</td>
<td>L2472-01MS</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
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<tr>
<td>VWAI-MW04-1112</td>
<td>L2472-01MS</td>
<td>water</td>
<td>X</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The following quality control samples were provided with this SDG: samples VWAI-TB01-112812 and VWAI-TB01-112912-trip blanks; sample VWAI-EB01-112812 and VWAI-EB01-112912-equipment blanks; and sample VWAI-MW07P-1112-field duplicate of sample VWAI-MW07-1112.

The samples were evaluated based on the following criteria:

- Data Completeness
- Sample Condition
- Technical Holding Times
- GC/MS Tuning
- GC Performance
- ICP MS Tuning
- Initial/Continuing Calibrations
- ICSA/ICSAB Standards
- RL Standards
- Blanks
- Internal Standards
- Surrogate Recoveries
- Laboratory Control Samples
- Matrix Spike Recoveries
- Matrix Duplicate RPDs
- Serial Dilutions
- Field Duplicates
- Identification/Quantitation
- Reporting Limits
- Tentatively Identified Compounds

* - indicates that qualifications were not required based on this criteria

Overall Evaluation of Data/Potential Usability Issues

A summary of qualifications applied to the sample results are noted below for the fractions validated. Specific details regarding qualification of the data are addressed in the Specific Evaluation section of this narrative. If an issue is not addressed there were no actions required based on unmet quality criteria. When more than one qualifier is associated with a compound/analyte the validator has chosen the qualifier that best indicates possible bias in the results and flagged the data accordingly. However, information regarding all quality control issues is provided in the body of the report and on the qualification summary page. Please note that when a compound or analyte is flagged due to blank contamination the BL qualifier code takes precedence over all other qualifier codes except a code that explains rejected data.

VOA

The field duplicate pair exhibited non-compliant field duplicate reproducibility which resulted in qualifications to the data.

SVVOA

The field duplicate pair exhibited non-compliant field duplicate reproducibility which resulted in qualifications to the data.
Select Filtered Metals

The laboratory did not perform a matrix spike or a matrix duplicate in this SDG. These QC samples are required by Region II. Qualifications were required.

Specific Evaluation of Data

Data Completeness

The SDG was received complete and intact. Resubmissions were not required.

Technical Holding Times

According to chain of custody records, sampling was performed on 11/28-29/12 and samples were received at the laboratory 11/29-30/12. All sample preparation and analysis was performed within Region II and/or method holding time requirements.

Matrix Spike/Matrix Duplicate

Select Filtered Metals

The laboratory did not perform a matrix spike/matrix duplicate on a field sample from this SDG. Region II required that all positive and non-detect results be qualified as estimated JU/UJ because of this. Therefore, the reported results for iron and manganese were qualified as estimated JU/UJ with a qualifier code of OT.

Field Duplicates

VOA

Sample VWAI-MW07-1112 and duplicate sample VWAI-MW07P-1112 exhibited non-compliant field duplicate reproducibility for benzene with 200% RPD; therefore the results for this compound were qualified as estimated (J/UJ), qualifier code: FD.

SVOA

Sample VWAI-MW07-1112 and duplicate sample VWAI-MW07P-1112 exhibited non-compliant field duplicate reproducibility for 2-methylnaphthalene with 200% RPD; therefore the results for this compound were qualified as estimated (J/UJ), qualifier code: FD.
A summary of qualifications required is provided on the following page. Please do not hesitate to contact DataQual ES with any questions regarding this validation report.

Sincerely,

Jacqueline Cleveland
Vice President
# Summary of Data Qualifications

## VOA

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
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</thead>
<tbody>
<tr>
<td>VWAI-MW07-1112, VWAI-MW07P-1112</td>
<td>benzene</td>
<td>+/-</td>
<td>J/UJ</td>
<td>FD</td>
</tr>
</tbody>
</table>

## SVOA

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Compound</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>VWAI-MW07-1112, VWAI-MW07P-1112</td>
<td>2-methylnaphthalene</td>
<td>+/-</td>
<td>J/UJ</td>
<td>FD</td>
</tr>
</tbody>
</table>

## Select Filtered Metals

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Analyte</th>
<th>Results</th>
<th>Q flag</th>
<th>Q Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>all samples</td>
<td>iron, manganese</td>
<td>+/-</td>
<td>J/UJ</td>
<td>OT</td>
</tr>
</tbody>
</table>
Glossary of Qualification Flags and Abbreviations

**Qualification Flags (Q-Flags)**

<table>
<thead>
<tr>
<th>Flag</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>not detected above the reported sample quantitation limit</td>
</tr>
<tr>
<td>J</td>
<td>estimated value</td>
</tr>
<tr>
<td>UJ</td>
<td>reported quantitation limit is qualified as estimated</td>
</tr>
<tr>
<td>N</td>
<td>analyte has been tentatively identified</td>
</tr>
<tr>
<td>JN</td>
<td>analyte has been tentatively identified, estimated value</td>
</tr>
<tr>
<td>R</td>
<td>result is rejected; the presence or absence of the analyte cannot be verified</td>
</tr>
</tbody>
</table>

**Method/Preparation/Field QC Blank Qualification Flags (Q-Flags)**

**Organic Methods**

- **NA**
  
  The sample result for the blank contaminant is greater than the LOD (2X sample LOD for common laboratory contaminants) when the blank value is less than the LOD. The sample result for the blank contaminant is not qualified with any blank qualifiers.

- **LOD**
  
  The sample result for the blank contaminant is less than the LOD (2X sample LOD for common laboratory contaminants) but greater than the MDL when the blank value is less than the LOD. The sample result for the blank contaminant is changed to the LOD and qualified as non-detect U.

**Inorganic Methods**

**ICB/CCB/PB Action:**

- **No Action**
  
  The sample result is greater than the LOD and greater than ten times (10X) the blank value.

- **U**
  
  The sample result is greater than or equal to the MDL but less than or equal to the LOD, result is reported as non-detect at the LOD, when the ICB/CCB/PB result is less or greater than the LOD.

- **R**
  
  Sample result is greater than the LOD and less than the ICB/CCB/PB value when the ICB/CCB/PB value is greater than the LOD.

- **J**
  
  Sample result is greater than the ICB/CCB/PB value but less than 10X the ICB/CCB/PB value when ICB/CCB/PB value is greater than the LOD.

- **J/UJ**
  
  Sample result is less than 10X LOD when blank result is below the negative LOD.
Glossary of Qualification Flags and Abbreviations, continued

**Field QC Blank action:**

*Note – Use field blanks to qualify data only if field blank results are greater than prep blank results.*

*Do not use rinsate blank associated with soils to qualify water samples and vice versa.*

**No Action** - The sample result is greater than the LOD and greater than ten times (10X) the blank value.

**U** - The sample result is greater than or equal to the MDL but less than or equal to the LOD, result is reported as non-detect at the LOD, when the FB result is less or greater than the LOD.

**R** - Sample result is greater than the LOD and less than the FB value when the FB value is greater than the LOD.

**J** - Sample result is greater than the FB value but less than 10X the FB value when FB value is greater than the LOD.

**General Abbreviations**

- **RL** reporting limit
- **MDL** method detection limit
- **IDL** instrument detection limit
- **LOD** Level of Detection
- **LOQ** Level of Quantitation
- **+** positive result
- **-** non-detect result
### QUALIFIER CODE REFERENCE

<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>TN</td>
<td>Tune</td>
</tr>
<tr>
<td>BSL</td>
<td>Blank Spike/LCS - High Recovery</td>
</tr>
<tr>
<td>BSH</td>
<td>Blank Spike/LCS - Low Recovery</td>
</tr>
<tr>
<td>BD</td>
<td>Blank Spike/Blank Spike Duplicate (LCS/LCSD) Precision</td>
</tr>
<tr>
<td>BRL</td>
<td>Below Reporting Limit</td>
</tr>
<tr>
<td>ISL</td>
<td>Internal Standard - Low Recovery</td>
</tr>
<tr>
<td>ISH</td>
<td>Internal Standard - High Recovery</td>
</tr>
<tr>
<td>MSL</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - Low Recovery</td>
</tr>
<tr>
<td>MSH</td>
<td>Matrix Spike and/or Matrix Spike Duplicate - High Recovery</td>
</tr>
<tr>
<td>MI</td>
<td>Matrix interference obscuring the raw data</td>
</tr>
<tr>
<td>MDP</td>
<td>Matrix Spike/Matrix Spike Duplicate Precision</td>
</tr>
<tr>
<td>2S</td>
<td>Second Source - Bad reproducibility between tandem detectors</td>
</tr>
<tr>
<td>SSL</td>
<td>Spiked Surrogate - Low Recovery</td>
</tr>
<tr>
<td>SSH</td>
<td>Spiked Surrogate - High Recovery</td>
</tr>
<tr>
<td>SD</td>
<td>Serial Dilution Reproducibility</td>
</tr>
<tr>
<td>ICL</td>
<td>Initial Calibration - Low Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICH</td>
<td>Initial Calibration - High Relative Response Factors (RRF)</td>
</tr>
<tr>
<td>ICB</td>
<td>Initial Calibration - Bad Linearity or Curve Function</td>
</tr>
<tr>
<td>CCL</td>
<td>Continuing Calibration - Low Recovery or %Difference</td>
</tr>
<tr>
<td>CCH</td>
<td>Continuing Calibration - High Recovery or %Difference</td>
</tr>
<tr>
<td>LD</td>
<td>Lab Duplicate Reproducibility</td>
</tr>
<tr>
<td>HT</td>
<td>Holding Time</td>
</tr>
<tr>
<td>PD</td>
<td>Pesticide Degradation</td>
</tr>
<tr>
<td>2C</td>
<td>Second Column - Poor Dual Column Reproducibility</td>
</tr>
<tr>
<td>LR</td>
<td>Concentration Exceeds Linear Range</td>
</tr>
<tr>
<td>MBL, EBL, FBL or TBL</td>
<td>Blank Contamination</td>
</tr>
<tr>
<td>RE</td>
<td>Redundant Result - due to Re-analysis or Re-extraction</td>
</tr>
<tr>
<td>DL</td>
<td>Redundant Result - due to Dilution</td>
</tr>
<tr>
<td>FD</td>
<td>Field Duplicate</td>
</tr>
<tr>
<td>OT</td>
<td>Other - explained in data validation report</td>
</tr>
<tr>
<td>%SOL</td>
<td>High moisture content</td>
</tr>
</tbody>
</table>

CH2M HILL
Vieques Island, Puerto Rico, CTO-083 AOC-I
SDG# SL2472
Lab Name: SPECTRUM ANALYTICAL, INC.
Lab Code: MITKEM
Case No.: L2472
Mod. Ref No.: ____________
SDG No.: SL2472

Matrix: (SOIL/SED/WATER) WATER
Sample wt/vol: 5.00 (g/mL) ML
Level: (TRACE/LOW/MED) LOW
% Moisture: not dec.
Lab Sample ID: L2472-01A
Lab File ID: V500540.D

GC Column: DB-624 ID: 0.25 (mm) Dilution Factor: 1.0
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)
Purge Volume: 5.0 (mL)

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50</td>
<td>U</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>2.2</td>
<td>J</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0</td>
<td>U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>

Date Received: 11/29/2012
Date Analyzed: 11/30/2012
<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50 u</td>
<td></td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>0.50 u</td>
<td></td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0 u</td>
<td></td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
1A - FORM I VOA-1
VOLATILE ORGANICS ANALYSIS DATA SHEET

Lab Name: SPECTRUM ANALYTICAL, INC.  Contract:  
Lab Code: MITKEM  Case No.: L2472  Mod. Ref No.:  
Matrix: (SOIL/SED/WATER)  WATER  Lab Sample ID: L2472-03A  
Sample wt/vol:  5.00 (g/mL) ML  Lab File ID: V500544.D  
Level: (TRACE/LOW/MED)  LOW  Date Received: 11/29/2012  
% Moisture: not dec.  Date Analyzed: 11/30/2012  
GC Column: DB-624  ID: 0.25 (mm)  Dilution Factor: 1.0  
Soil Extract Volume:  (uL)  Soil Aliquot Volume:  
Purge Volume:  5.0  (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
</tr>
</thead>
<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50 U</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>0.50 U</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0 U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
<td></td>
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</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L2472  
Lab Sample ID: L2472-04A  
Lab File ID: V500542.D  
Matrix: WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Dilution Factor: 1.0  
Soil Extract Volume: (uL)  
Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION: UG/L</th>
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<th>DL</th>
<th>LOD</th>
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<td></td>
<td>0.41</td>
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<td>Benzene</td>
<td>0.50 U</td>
<td></td>
<td>0.33</td>
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<td>5.0</td>
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<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0 U</td>
<td></td>
<td>0.61</td>
<td>1.0</td>
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<td>CAS NO.</td>
<td>COMPOUND</td>
<td>CONCENTRATION: US/G/L</td>
<td>Q</td>
<td>DL</td>
<td>LOD</td>
<td>LOQ</td>
</tr>
<tr>
<td>--------</td>
<td>----------------------</td>
<td>-----------------------</td>
<td>----</td>
<td>-----</td>
<td>------</td>
<td>-----</td>
</tr>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50</td>
<td>U</td>
<td>0.41</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>0.82</td>
<td>✓</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
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<td>1.0</td>
<td>U</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
<tr>
<td>CAS NO.</td>
<td>COMPOUND</td>
<td>CONCENTRATION:</td>
<td>Q</td>
<td>DL</td>
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<td>LOQ</td>
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<tr>
<td>107-06-2</td>
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<td>0</td>
<td>0.41</td>
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<td>5.0</td>
</tr>
<tr>
<td>71-43-2</td>
<td>Benzene</td>
<td>0.50</td>
<td>*</td>
<td>0.33</td>
<td>0.50</td>
<td>5.0</td>
</tr>
<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
<td>1.0</td>
<td>0</td>
<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L2472  
Mod. Ref No.:  
SDG No.: SL2472  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 5.00 (g/mL) ML  
Level: (TRACE/LOW/MED) LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Soil Extract Volume: (uL) Soil Aliquot Volume: (uL)  
Purge Volume: 5.0 (mL)  

<table>
<thead>
<tr>
<th>CAS NO.</th>
<th>COMPOUND</th>
<th>CONCENTRATION:</th>
<th>Q</th>
<th>DL</th>
<th>LOD</th>
<th>LOQ</th>
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<tbody>
<tr>
<td>107-06-2</td>
<td>1,2-Dichloroethane</td>
<td>0.50 U</td>
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<tr>
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<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L2472  
Mod. Ref No.:  
Matrix: (SOIL/SED/WATER) WATER  
Lab Sample ID: L2472-08A  
Sample wt/vol: 5.00 (g/mL) ML  
Lab File ID: V500543.D  
Level: (TRACE/LOW/MED) LOW  
Date Received: 11/30/2012  
% Moisture: not dec.  
Date Analyzed: 11/30/2012  
GC Column: DB-624 ID: 0.25 (mm)  
Dilution Factor: 1.0  
Soil Extract Volume:__ (uL) Soil Aliquot Volume:__ (uL)  
Purge Volume:__ (mL)  

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<td>78-87-5</td>
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<td>0.61</td>
<td>1.0</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L2472  
Matrix: WATER  
Sample wt/vol: 5.00 (g/mL)  
Level: LOW  
% Moisture: not dec.  
GC Column: DB-624  
ID: 0.25 (mm)  
Soil Extract Volume:  
Purge Volume: 5.0  

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<th>CAS NO.</th>
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<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L2472  
Matrix: WATER  
Sample wt/vol: 5.00 (g/mL)  
Level: LOW  
% Moisture: not dec.  
GC Column: DB-624 ID: 0.25 (mm)  
Soil Extract Volume:  
Purge Volume: 5.0 (mL)  

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<tr>
<td>78-87-5</td>
<td>1,2-Dichloropropane</td>
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<td>0.61</td>
<td>1.0</td>
<td>5.0</td>
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</table>
**Lab Name:** SPECTRUM ANALYTICAL, INC.  
**Lab Code:** MITKEM  
**Case No.:** L2472  
**Mod. Ref No.:**  
**SDG No.:** SL2472  
**Matrix:** (SOIL/SED/WATER) WATER  
**Sample wt/vol:** 1000 (g/mL) ML  
**Lab Sample ID:** L2472-01C  
**Lab File ID:** S6B1996.D  
**Level:** (LOW/MED) LOW  
**Extraction:** (Type) SEPF  
**% Moisture:**  
**Decanted: (Y/N)**  
**Concentrated Extract Volume:** 1000 (μL)  
**Injection Volume:** 1.0 (μL)  
**GPC Factor:** 1.00  
**Date Received:** 11/29/2012  
**Date Extracted:** 11/29/2012  
**Date Analyzed:** 12/18/2012  
**GPC Cleanup:** (Y/N) N  
**pH:**  
**Dilution Factor:** 1.0

<table>
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<tr>
<th>CAS NO.</th>
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<td>J</td>
<td>0.96</td>
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<tr>
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<td>2-Methylnaphthalene</td>
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<td>J</td>
<td>0.94</td>
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<td>117-81-7</td>
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<td>J</td>
<td>1.3</td>
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<td>5.0</td>
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</tbody>
</table>
**Lab Name:** SPECTRUM ANALYTICAL, INC.  
**Lab Code:** MITKEM  
**Case No.:** L2472  
**Contract:**  
**Mod. Ref No.:**  
**SDG No.:** SL2472  

**Matrix:** (SOIL/SED/WATER) WATER  
**Sample wt/vol:** 1000 (g/mL) ML  
**Lab Sample ID:** L2472-02B  
**Lab File ID:** S6B1999.D  

**Level:** (LOW/MED) LOW  
**Extraction:** (Type) SEPF  
**% Moisture:** Decanted: (Y/N) N  
**Date Received:** 11/29/2012  
**Concentrated Extract Volume:** 1000 (uL)  
**Date Extracted:** 11/29/2012  
**Injection Volume:** 1.0 (uL)  
**GPC Factor:** 1.00  
**Date Analyzed:** 12/18/2012  
**GPC Cleanup:** (Y/N) N  
**pH:**  
**Dilution Factor:** 1.0

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<td>91-20-3</td>
<td>Naphthalene</td>
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<td>0</td>
<td>0.96</td>
<td>2.0</td>
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<td>0.94</td>
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**SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET**

- **Lab Name:** SPECTRUM ANALYTICAL, INC.
- **Lab Code:** MITKEM
- **Matrix:** WATER
- **Lab Sample ID:** L2472-03C
- **Lab File ID:** S6B2000.D
- **Concentration:**

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<th>LOD</th>
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<td>1.3</td>
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- **Date Received:** 11/29/2012
- **Date Extracted:** 11/29/2012
- **Date Analyzed:** 12/18/2012
- **pH:**
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<td>2.0</td>
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<td>0.94</td>
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<td>5.0</td>
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Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Case No.: L2472  
Matrix: WATER  
Sample wt/vol: 1000 (g/mL) ML  
Level: LOW  
% Moisture:  
Decanted: (Y/N)  
Concentrated Extract Volume: 1000 (uL)  
Injection Volume: 1.0 (uL)  
GPC Cleanup: (Y/N) N  
GPC Factor: 1.00  
Date Analyzed: 12/18/2012  
Date Extracted: 11/30/2012  
Date Received: 11/30/2012  
Lab Sample ID: L2472-078  
Lab File ID: S6B2003.D  
Contract:  
Mod. Ref No.:  
SDG No.: SL2472
Lab Name: SPECTRUM ANALYTICAL, INC.  
Lab Code: MITKEM  
Matrix: (SOIL/SED/WATER) WATER  
Sample wt/vol: 1000 (g/mL) ML  
Level: (LOW/MED) LOW  
% Moisture:  
Concentrated Extract Volume: 1000 (μL)  
Injection Volume: 1.0 (μL)  
GPC Cleanup: (Y/N) N  
CAS NO. COMPOUND | CONCENTRATION: (UG/L) | Q | DL | LOD | LOQ  
--- | --- | --- | --- | --- | ---  
91-20-3 Naphthalene | 41 | 0.96 | 2.0 | 2.0 |  
91-57-6 2-Methylnaphthalene | 42 | 0.94 | 2.0 | 2.0 |  
117-81-7 Bis(2-ethylhexyl)phthalate | 41 | 1.3 | 2.0 | 5.0 |
Lab Name: SPECTRUM ANALYTICAL, INC.  Lab Code: MITKEM  Case No.: L2472  Contract:  Mod. Ref No.:  SDG No.: SL2472  

Matrix: (SOIL/SED/WATER) WATER  Lab Sample ID: L2472-01CMSD  
Sample wt/vol: 1000 (g/mL) ML  Lab File ID: S6B2032.D  
Level: (LOW/MED) LOW  Extraction: (Type) SEP   
% Moisture:  Decanted: (Y/N) Date Received: 11/29/2012  
Concentrated Extract Volume: 1000 (uL) Date Extracted: 11/29/2012  
Injection Volume: 1.0 (uL) GPC Factor: 1.00 Date Analyzed: 12/19/2012  
GPC Cleanup: (Y/N) N  pH: Dilution Factor: 1.0

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<th>CAS NO.</th>
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CAS: Chemical Abstracts Service
QC: Quality Control
LOD: Limit of Detection
LOQ: Limit of Quantification
Lab Name: Spectrum Analytical, Inc.  
Lab Code: MITKEM  
Matrix (soil/water): WATER  
Level (low/med): MED  
Solids: 0.0  
Concentration Units (ug/L or mg/kg dry weight): ug/L

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<th>Concentration</th>
<th>C</th>
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Comments:
Lab Name: Spectrum Analytical, Inc.  
Lab Code: MITKEM  
Matrix (soil/water): WATER  
Level (low/med): MED  
% Solids: 0.0  
Concentration Units (ug/L or mg/kg dry weight): ug/L  

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Comments:
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<th>CAS No.</th>
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<td>10.0</td>
<td>15.0</td>
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Comments:
REPORT NARRATIVE
Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.
Client: CH2M-Hill, Inc.
Project: CTO-0083 Vieques AOC I
Laboratory Workorder / SDG #: L2472
SW846 8260C, VOC by GC-MS

I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:
   All samples were prepared within the method-specified holding times.

B. Sample Analysis:
   All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code:
SW846 8260C

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW5035

V. INSTRUMENTATION
The following instrumentation was used

Instrument Code: V5
Instrument Type: GCMS-VOA
Description: HP6890 / HP6890
Manufacturer: Hewlett-Packard
Model: 6890 / 6890

VI. ANALYSIS

A. Calibration:
   Calibrations met the method/SOP acceptance criteria.

B. Blanks:
   All method blanks were within the acceptance criteria.

C. Surrogates:
   Surrogate standard percent recoveries were within the QC limits.

D. Spikes:
   1. Laboratory Control Spikes (LCS):
      Percent recoveries for lab control samples were within the QC limits.
   2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):
      Matrix spikes were performed on samples: VWAI-MW04-1112 (L2472-01AMS) and VWAI-MW04-1112 (L2472-01AMSD).
      Percent recoveries were within the QC limits.
      Replicate RPDs were within the advisory QC limits.

E. Internal Standards:
   Internal standard peak areas were within the QC limits.

F. Dilutions:
   No sample in this SDG required analysis at dilution.
G. Samples:

No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: __________

Date: ___________ 12/23/2012 ___________
REPORT NARRATIVE

Spectrum Analytical, Inc. Featuring Hanibal Technology, RI Division.

Client: CH2M-Hill, Inc.

Project: CTO-0083 Vieques AOC I

Laboratory Workorder / SDG #: L2472
SW846 8270D, SVOA by GC-MS

I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:

All samples were prepared within the method-specified holding times.

B. Sample Analysis:

All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 8270D

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW3510

V. INSTRUMENTATION

The following instrumentation was used
Instrument Code: S6
Instrument Type: GCMS-Semi
Description: HP7890A
Manufacturer: Agilent
Model: 7890A/5973
GC Column used: 30 m X 0.25 mm ID [0.25 um thickness] Rxi-5sil MS capillary column.

VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Surrogates:

Surrogate standard percent recoveries were within the QC limits with the following exceptions. Please note that the acceptance criteria allow one surrogate recovery outside of the QC limits per fraction.

VWAI-MW04-1112 (L2472-01CMS), recovery is below criteria for Terphenyl-d14 at 50% with criteria of (50-135).

VWAI-MW05-1112 (L2472-03C), recovery is below criteria for Terphenyl-d14 at 37% with criteria of (50-135).

VWAI-MW07P-1112 (L2472-06B), recovery is below criteria for Terphenyl-d14 at 33% with criteria of (50-135).

D. Spikes:

1. Laboratory Control Spikes (LCS):

Percent recoveries for lab control samples were within the QC limits.

2. Matrix Spike / Matrix Spike Duplicate (MS/MSD):

Matrix spikes were performed on samples: VWAI-MW04-1112 (L2472-01CMS) and VWAI-MW04-1112 (L2472-01CMSD).
Percent recoveries were within the QC limits.

Replicate RPDs were within the advisory QC limits.

E. Internal Standards:

Internal standard peak areas were within the QC limits.

F. Dilutions:

No sample in this SDG required analysis at dilution.

G. Samples:

No other unusual occurrences were noted during sample analysis.

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Signed: ______

Date: _______ 12/27/2012 ______
I. SAMPLE RECEIPT

No exceptions or unusual conditions were encountered unless a Sample Condition Notification Form, or other record of communication is included with the Sample Receipt Documentation.

II. HOLDING TIMES

A. Sample Preparation:

All samples were prepared within the method-specified holding times.

B. Sample Analysis:

All samples were analyzed within the method-specified holding times.

III. METHODS

Samples were analyzed following procedures in laboratory test code: SW846 6010C.

IV. PREPARATION

Aqueous Samples were prepared following procedures in laboratory test code: SW3005A.

V. INSTRUMENTATION
The following instrumentation was used:

Instrument Code: OPTIMA2
Instrument Type: ICP
Description: Optima 3100 XL
Manufacturer: Perkin-Elmer
Model: 3100 XL

VI. ANALYSIS

A. Calibration:

Calibrations met the method/SOP acceptance criteria.

B. Blanks:

All method blanks were within the acceptance criteria.

C. Spikes:

1. Laboratory Control Spikes (LCS):

   Percent recoveries for laboratory control samples were within the QC limits.

2. Matrix spike (MS):

   A matrix spike was not performed on any sample in this SDG.

D. Post Digestion Spike (PDS):

   A post-digestion spike was not performed on any sample in this SDG.

E. Duplicate sample:

   A duplicate analysis was not performed on any sample in this SDG.

F. Serial Dilution (SD):

   Serial Dilution analyses were performed on sample: VWAI-MW07-1112 (L2472-05DSD).

   Percent differences were within the QC limits.

G. Samples:
No other unusual occurrences were noted during sample analysis.

I certify that this data package is in compliance with the terms and conditions agreed to by the client and Spectrum, both technically and for completeness, except for the conditions noted above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or designated person, as verified by the following signature.

Signed: [Signature]

Date: 12/27/12
Data Flag/Qualifiers:

U  Not Detected. This compound was analyzed-for but not detected. For most analyses the reporting limit (lowest standard concentration) is the value listed. For Department of Defense programs, this is the Limit of Detection (LOD).

J  This flag indicates an estimated value due to either
- the compound was detected below the reporting limit, or
- estimated concentration for Tentatively Identified Compound

B  This flag indicates the compound was also detected in the associated Method Blank. The B flag has an alternative meaning for Inorganics analyses reported using CLP ILM-type metals forms, indicating a “trace” concentration below the reporting limit and equal to or above the detection limit.

D  For Organics analysis, this flag indicates the compound concentration was obtained from a secondary dilution analysis.

E  This flag indicates the compound concentration exceeded the Calibration Range. The E flag has an alternative meaning for Inorganics analyses reported using CLP metals forms, indicating an estimated concentration due to the presence of interferences, as determined by the serial dilution analysis.

P  This flag is used for pesticides/PCB/herbicide compound when there is a greater than 40% difference for detected concentration between the two GC columns used for primary and confirmation analyses. This difference typically indicates an interference, causing one value to be unusually high. The lower of the two values is generally reported on the Form 1, and both values reported on the Form 10.

A  Used to flag semivolatile organic Tentatively Identified Compound library search results for compounds identified as aldol condensation byproducts.

N  Used to flag results for volatile and semivolatile Organics analysis Tentatively Identified Compounds where an analyte has passed the identification criteria, and is considered to be positively identified. For Inorganics analysis the N flag indicates the matrix spike recovery falls outside of the control limit.

*  For Inorganics analysis the * flag indicates Relative Percent Difference for duplicate analyses is outside of the control limit.
Sample ID Suffixes

DL  Diluted analysis. The sample was diluted and reanalyzed. The DL may be followed by a digit if more than one diluted reanalysis is provided. The DL suffix is not attached to an analysis initially performed at dilution, only to reanalyses performed at dilution.

RE  Reanalysis. Appended to the client sample ID to indicate a reextraction and reanalysis or a reanalysis of the original sample extract.

RA  Reanalysis. Appended to the laboratory sample ID indicates a reanalysis of the original sample extract.

RX  Reextraction. Appended to the laboratory sample ID indicates a reextraction of the sample.

MS  Matrix Spike.

MSD  Matrix Spike Duplicate

DUP  Duplicate analysis

SD  Serial Dilution

PS  Post-digestion or Post-distillation spike. For metals or inorganic analyses.
**CHAIN OF CUSTODY RECORD**

<table>
<thead>
<tr>
<th>Lab Id:</th>
<th>Sample Id:</th>
<th>Date:</th>
<th>Time:</th>
<th>Matrix</th>
<th>Type</th>
<th># of VOA Vials</th>
<th># of Amber Glass</th>
<th># of Plastic</th>
</tr>
</thead>
<tbody>
<tr>
<td>2472-01</td>
<td>VWE-M96-112</td>
<td>11/28/12</td>
<td>0905</td>
<td>GW</td>
<td></td>
<td>4</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>VWE-M98-605</td>
<td>0905</td>
<td>GW</td>
<td></td>
<td></td>
<td>2</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>VWE-M98-857</td>
<td>0905</td>
<td>AG</td>
<td></td>
<td></td>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>VWE-M98-812</td>
<td>11/25</td>
<td>GW</td>
<td></td>
<td></td>
<td>4</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>VWE-M98-112</td>
<td>11/28/12</td>
<td>T8</td>
<td></td>
<td></td>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

**Special Handling:**
- All TATs subject to laboratory approval.
- Min. 24-hour notification needed for rushes.
- Samples disposed of after 30 days unless otherwise instructed.
- TAT: Indicate Date Needed: PER CONTRACT

**Project No.:** 392-495 F.E.FK

**Site Name:** Vieques, PR

**Location:** Vieques

**State:** PR

**Site Name:** Vieques

**Project Mgr.:** Stephen Brand

**P.O. No.:** RQN:

**Notes:** ATB has HCl prep
**CHAIN OF CUSTODY RECORD**

**Special Handling:**
- TAT: Indicate Date Needed.
  - All TATs subject to laboratory approval.
  - Min. 24-hour notification needed for rushes.
- Samples disposed of after 30 days unless otherwise instructed.

**Report To:** Michael Zooboni
**Invoice To:** PER CONTRACT
**Project No.:** 312485 FL FK
**Site Name:** Vieques, PR AEC I
**Location:** Vieques
**State:** PR
**P.O. No.:** Recovered
**RQN:**
**Project Mgr.:** P. Murphy T. Horn
**Sampler(s):**

<table>
<thead>
<tr>
<th>No.</th>
<th>Code</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Na₂S₂O₃</td>
<td>2 = HCl</td>
</tr>
<tr>
<td>2</td>
<td>H₂SO₄</td>
<td>3 = HNO₃</td>
</tr>
<tr>
<td>3</td>
<td>NaOH</td>
<td>4 = Ascorbic Acid</td>
</tr>
<tr>
<td>4</td>
<td>H₃PO₄</td>
<td>5 = NaHSO₄</td>
</tr>
<tr>
<td>5</td>
<td>H₂S₂O₃</td>
<td>6 = CH₃OH</td>
</tr>
<tr>
<td>6</td>
<td>Na₂S₂O₃</td>
<td>7 = Na₂S₂O₃</td>
</tr>
</tbody>
</table>

**Containers:**
- DW = Drinking Water
- GW = Groundwater
- WW = Wastewater
- O = Oil
- SW = Surface Water
- SO = Soil
- Sludge
- A = Air
- X1
- X2
- X3

**Analyses:**
- QA/QC Reporting Level
  - Level I
  - Level II
  - Level III
  - Level IV
  - Other

**Preservative Code:**
- Code: 6 NA 4 NA 9

**Notes:**
- Site specific reporting standards:
- Other

**Lab Id:** LVAF-MW01-112
**Sample Id:** LVAF-MW01-112
**Date:** 11/21/12
**Time:** 03:05

**Condition upon receipt:**
- Ambient
- Ambient

**Location:**
- 175 Metro Center Boulevard
- Warwick, RI 02886-1755
- 401-732-3400 • Fax 401-732-3499 • www.spectrum-analytical.com
Received By:]

Reviewed By:]

Work Order: L2472  Client Name: CH2M Hill, Inc.

Project Name/Event: CTO-0083 Vieques AOC E and I

Remarks: (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.

1. Custody Seal(s)  Present/Absent
2. Custody Seal Nos.  N/A
3. Traffic Reports/Chain of Custody Records (TR/COCS) or Packing Lists
4. Airbill  Present/Absent
5. Airbill No.  FedEx 8765 4395 6027,
6. Sample Tags  Present/Absent
   Sample Tag Numbers  Listed/
   Not Listed on Chain-of-Custody
7. Sample Condition  Intact/Broken/
   Leaking
8. Cooler Temperature Indicator Bottle  Present/Absent
9. Cooler Temperature  4.3 °C
10. Does information on TR/COCS and sample tags agree?  Yes/No
11. Date Received at Laboratory  11/29/2012
12. Time Received  10:54

Sample Transfer
Fraction (1) TVOA/VOA  Fraction (2) SVOA/PST/ARS
Area #  Area #
By  By
On  On
IR Temp Gun ID: MT-1
Coolant Condition: ICE
Preservative Name/Lot No.

VOA Matrix:

US = Unpreserved Soil  A = Air
UA = Unpreserved Aqueous  H = HCl
M = MeOH  E = Encore
N = NaHSO4  F = Freeze

See Sample Condition Notification/Corrective Action Form

Rad OK  Yes/No

Sample Condition Form 6/3
<table>
<thead>
<tr>
<th>Remarks: (1/2) Please see associated sample/extract transfer logbook pages submitted with this data package.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Custody Seal(s)</td>
</tr>
<tr>
<td>2. Custody Seal Nos.</td>
</tr>
<tr>
<td>3. Traffic Reports/Chain of Custody Records (TR/COCs) or Packing Lists</td>
</tr>
<tr>
<td>4. Airbill</td>
</tr>
<tr>
<td>5. Airbill No.</td>
</tr>
<tr>
<td>6. Sample Tags</td>
</tr>
</tbody>
</table>
| Sample Tag Numbers | Listed/
| NOT Listed on Chain-of-Custody |
| 7. Sample Condition | Intact/Broken/Leaking |
| 8. Cooler Temperature Indicator Bottle | Present/Absent |
| 9. Cooler Temperature | 1.5 °C |
| 10. Does information on TR/COCs and sample tags agree? | Yes/No |
| 11. Date Received at Laboratory | 11/30/2012 |
| 12. Time Received | 10:25 |

<table>
<thead>
<tr>
<th>Preservative Name/Lot No:</th>
</tr>
</thead>
<tbody>
<tr>
<td>VOA Matrix Key:</td>
</tr>
<tr>
<td>US = Unpreserved Soil</td>
</tr>
<tr>
<td>UA = Unpreserved Aqueous</td>
</tr>
<tr>
<td>M = MeOH</td>
</tr>
<tr>
<td>N = NaHSO4</td>
</tr>
<tr>
<td>A = Air</td>
</tr>
<tr>
<td>H = HCl</td>
</tr>
<tr>
<td>E = Encore</td>
</tr>
<tr>
<td>F = Freeze</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>See Sample Condition Notification/Corrective Action Form</th>
<th>Yes</th>
<th>No</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Lab Sample ID</th>
<th>Preservation (pH)</th>
<th>VOA Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>L2472-05</td>
<td>&lt;2</td>
<td>ASCORBIC</td>
</tr>
<tr>
<td>L2472-06</td>
<td>ASCORBIC</td>
<td></td>
</tr>
<tr>
<td>L2472-07</td>
<td>ASCORBIC</td>
<td></td>
</tr>
<tr>
<td>L2472-08</td>
<td>H</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample Transfer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fraction (1) TVOA/VOA</td>
</tr>
<tr>
<td>Area #</td>
</tr>
<tr>
<td>By</td>
</tr>
<tr>
<td>On</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>IR Temp Gun ID: MT-87</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coolant Condition: ICE</td>
</tr>
<tr>
<td>Preservative Name/Lot No:</td>
</tr>
</tbody>
</table>
I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:  LA473-   LAB: Spectrum Analytical

SITE NAME: Vieques ADCJ

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent?

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, and/or cover letter signed release present?

2.2 Are case number and SDG number(s) contained in the narrative or cover letter?

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, and/or Chain of Custodies from the field samplers present for all samples sign release present?

ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)?

1.3 Sample Conditions/Problems
1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects non"UJ".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

The maximum holding time for aqueous samples is 14 days.

The maximum holding time for soils non aqueous samples is 14 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with HCL acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. For non-aqueous samples for volatile components that are frozen (less than 7°C) or are properly cooled (4°C ± 2°C) and perserved with NaHSO₄, the maximum holding time is 14 days from sample collection. If
uncertain about preservation, contact the laboratory /sampling team to determine whether or not samples were preserved.

ACTION: Qualify sample results according to Table 1:

Table 1. Holding Time Actions for Trace Volatile Analysis

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Preserved</th>
<th>Criteria</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Detected Associated</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Compounds</td>
</tr>
<tr>
<td>Aqueous</td>
<td>No</td>
<td>≤7 days</td>
<td>No qualifications</td>
</tr>
<tr>
<td></td>
<td>No</td>
<td>&gt; 7 days</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>&gt; 14 days</td>
<td>J</td>
</tr>
<tr>
<td>Non Aqueous</td>
<td>No</td>
<td>≤14 days</td>
<td>J</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
<td>≤14 days</td>
<td>No qualifications</td>
</tr>
</tbody>
</table>

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Water

b. Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:

a. Water

b. Soil

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data
Assessments and contact the laboratory/project officer/appropriate official for an explanation/resubmittal, make any necessary corrections and document effect in the Data Assessment.

3.3 Were the surrogate recovery limits followed per Table 2. If Table 2 criteria were not followed, the laboratory may use in-house performance criteria (per SW-846, Method 8000C, section 9.7). Other compounds may be used as surrogates, depending upon the analysis requirements.

Table 2. Surrogate Spike Recovery Limits for Water and Soil/Sediments

<table>
<thead>
<tr>
<th>DMC</th>
<th>Recovery Limits (%) Water</th>
<th>Recovery Limits Soil/Sediment</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-Bromofluorobenzene</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dibromofluoromethane</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Toluene-d$_8$</td>
<td>80-120</td>
<td>70-130</td>
</tr>
<tr>
<td>Dichloroethane-d$_4$</td>
<td>80-120</td>
<td>70-130</td>
</tr>
</tbody>
</table>

Note: Use above table if laboratory did not provide in-house recovery criteria.

Note: Other compounds may be used as surrogates depending upon the analysis requirements.

3.4 Were outliers marked correctly with an asterisk? [ ] [ ] [✓]

ACTION: Circle all outliers with a red pencil.

3.5 Were one or more volatile surrogate recoveries out of specification for any sample or method blank. Table 2. [ ] [✓]

If yes, were samples reanalyzed? [ ] [✓]

Were method blanks reanalyzed? [ ] [✓]
ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J")
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects, but qualify positive results as estimated "J".

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J")
2. Non-detects for that should be qualified as unusable ("R")

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. If one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose the blank problem to be an isolated occurrence.

3.6 Are there any transcription/calculation errors between raw data and reported data?

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Sample (Form III/Equivalent)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG.
USEPA Region II
SW846 Method 8260B VOA
Date: August 2008
SOP: HW-24, Rev. 2

YES NO N/A

Note: LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation/resubmittals. Make note in the data assessment.

4.2 Were the Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

A. Water

B. Soil

C. Med Soil

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

4.3 Have in house LCS recovery limits been developed (Method 8000C, Sect 9.7).

4.4 If in house limits are not developed, are LCS acceptance recovery limits between 70 - 130% (Method 8000c Sect 9.5)?

4.5 Were one or more of the volatile LCS recoveries outside the in house laboratory recovery criteria for spiked analytes? If in house limits are not present use 70 - 130% recovery limits.
# Table 3. LCS Actions for Volatile Analysis

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Action</th>
<th>Detected Spiked Compounds</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td></td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td></td>
<td>UJ</td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td></td>
<td>No Qualifications</td>
</tr>
</tbody>
</table>

## 5.0 Matrix Spikes (Form III or equivalent)

### 5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix?

**NOTE:** The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, Method 8260B, Sect 8.4.2).

### 5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III?

**ACTION:** If any data are missing take action as specified in section 3.2 above.

### 5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples)
of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month (page 8000C, section 9.5.)

a. Water
b. Waste
c. Soil/Solid

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The concentration of the LCS should be determined as described SW-Method 8000C Section 9.5.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

5.4 Have in house MS recovery limits been developed (Method 8000C, Sect 9.7) for each matrix.

5.5 Were one or more of the volatile MS/MSD recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000C, Sect. 9.5.4.

ACTION: Circle all outliers with a red pencil.

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.
NOTE: No qualification of data is necessary on MS and MSD data alone. However, using informed professional judgement, the data reviewer may use MS and MSD results in conjunction with other QC criteria to determine the need for some qualification.

Note: The data reviewer should first try to determine to what extent the results of the MS and MSD affect the associated data. This determination should be made with regard to the MS and MSD sample itself, as well as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affect all associated samples, and the reviewer must use professional judgement to qualify the data from all associated samples.

Note: The reviewer must use professional judgement to determine the need for qualification of non-spiked compounds.

ACTION: Follow criteria in Table 4 when professional judgement deems qualification of sample.

**Table 4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Volatile Analysis**

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Detected Spiked Compounds</th>
<th>Action</th>
<th>Non-Detected Spiked Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>%R &gt; Upper Acceptance Limit</td>
<td>J</td>
<td>J</td>
<td>No Qualifiers</td>
</tr>
<tr>
<td>%R &lt; Lower Acceptance Limit</td>
<td>J</td>
<td>UJ</td>
<td></td>
</tr>
<tr>
<td>Lower Acceptance Limit ≤ %R</td>
<td></td>
<td>No Qualifications</td>
<td></td>
</tr>
</tbody>
</table>
6.0 Blank (CLP Form IV Equivalent)

6.1 Is the Method Blank Summary form present? 

6.2 Frequency of Analysis: Has a method blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch?

6.3 Has a method blank been analyzed for each GC/MS system used?

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for volatile organic compounds?

7.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.
7.2 Do any field/rinse blanks have positive volatile organic compound results?  

**YES**  **NO**  **N/A**

**ACTION:** Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

**NOTE:** All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

**ACTION:** Follow the directions in Table 5 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

```
VWAI-TB01-112812  MOQ
VWAI-EB01-112812  MOQ
VWAI-TB01-112912  MOQ
VWAI-EB01-112912  MOQ
```
### Table 5. Volatile Organic Analysis Blank Contamination Criteria

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Detects</td>
<td>Not detected</td>
<td>No qualification</td>
</tr>
<tr>
<td></td>
<td>&lt; CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
</tr>
<tr>
<td></td>
<td></td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>&gt; CRQL*</td>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report the concentration for the sample with a U, or qualify the data as unusable R</td>
</tr>
<tr>
<td></td>
<td></td>
<td>≥ CRQL and ≥ blank contamination</td>
<td>Use professional judgement</td>
</tr>
<tr>
<td></td>
<td>= CRQL*</td>
<td>&lt; CRQL</td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td></td>
<td>≥ CRQL</td>
<td>Use professional judgement</td>
</tr>
<tr>
<td></td>
<td>Gross contamination</td>
<td>Detects</td>
<td>Qualify results as unusable R</td>
</tr>
</tbody>
</table>

* 2x the CRQL for methylene chloride, 2-butanone, and acetone
** Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 μg/L.

NOTE: If gross blank contamination exists (e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected volatile organic target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
USEPA Region II
SW846 Method 8260B VOA

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YES NO N/A

7.3 Are there field/rinse/equipment blanks associated with every sample?

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.

NOTE: For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?

9.3 Has an instrument performance check solution (BFB)
been analyzed for every twelve hours of sample analysis per instrument? (see Table 4, SW-846, page 8260B-36)

YES NO N/A

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS GC/MS tuning data are available.

ACTION: If the laboratory/project officer cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable, "R".

9.4 Have the ion abundances been normalized to m/z 95?

9.5 Have the ion abundance criteria been met for each instrument used?

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.)

9.7 Have the appropriate number of significant figures (two) been reported?

ACTION: If large errors exist, take action as specified in section 3.2.

9.8 Are the spectra of the mass calibration compounds acceptable.

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.
10.0 **Target Analytes (CLP Form I Equivalent)**

10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

- a. Samples and/or fractions as appropriate
- b. Matrix spikes and matrix spike duplicates
- c. Blanks
- d. Laboratory Control Samples

10.2 Are the reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

- a. Samples and/or fractions as appropriate
- b. Matrix spikes and matrix spike duplicates (Mass spectra not required)
- c. Blanks
- d. Laboratory Control Samples

**ACTION:** If any data are missing, take action specified in 3.2 above.

10.3 Is chromatographic performance acceptable with respect to:

Baseline stability?
Resolution?  
YES NO N/A

Peak shape?  
YES NO N/A

Full-scale graph (attenuation)?  
YES NO N/A

Other: ____________________________

**ACTION:** Use professional judgement to determine the acceptability of the data.

10.4 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample?  

**ACTION:** If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, contact the lab for missing spectra.

10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?  

10.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?  

10.7 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?  

**ACTION:** Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be
positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

11.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

11.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier?  

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

11.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate  

b. Blanks

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a CAS#.

NOTE: If TICs are present in the associated blanks take action as specified in section 3.2 above.
11.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)? [ ] [ ] √

**ACTION:**
1. Flag with "R" any target compound listed as a TIC.
2. Make sure all rejected compounds are properly reported if they are target compounds.

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? [ ] [ ] [ ]

11.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%? [ ] [ ] √

**ACTION:** Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO₂ (M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found? [ ] [ ] √

**NOTE:** Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be
reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL's adjusted to reflect sample dilutions and, for soils, sample moisture?  YES  

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC accedence dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration?  YES  

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above.

14.0 GC/MS Initial Calibration (CLP Form VI Equivalent)
USEPA Region II
SW846 Method 8260B VOA
SOP: HW-24, Rev. 2
Date: August 2008

14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction?

**YES NO N/A**

**YES NO N/A**

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

ACTION: If the percent relative standard deviation (% RSD) is > 20%, (8000C-39) qualify positive results for that analyte "J". When % RSD > 90%, qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.2 Are all average RRFs > 0.050?

**YES NO N/A**

ACTION: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

Chloromethane 0.10
1,1-Dichloroethane 0.10
Bromoform 0.10
Chlorobenzene 0.30
1,1,2,2-Tetrachloroethane 0.30

NOTE: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, or for the requirements for the 5 compounds in 14.2 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration?

**YES NO N/A**

NOTE: (Method Requirement) For the following CCC compounds, the %RSD values must be ≤ 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment.
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

**ACTION:** Circle all outliers with a red pencil.

**ACTION:** If the % RSD is > 20.0%, or > 30% for the 6 compounds in 14.3 above, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

**NOTE:** The above data qualification action applies regardless of method requirements.

**NOTE:** Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF? [ ] [X] [ ]

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.) [ ] [X] [ ]

**ACTION:** Circle errors with a red pencil.

**ACTION:** If errors are large, take action as specified in section 3.2 above.

15.0 GC/MS Calibration Verification (CLP Form VII Equivalent) -26 VOA.
15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest?  

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument? 

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

15.3 Was the % D determined from the calibration verification determined using RRF or CF? 

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.

15.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2).

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be ≤ 20.0%. If %D values reported are > 20.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride
USEPA Region II  
Date: August 2008  
SOP: HW-24, Rev. 2  
SWM846 Method 8260B VOA  
YES NO N/A

**ACTION:** Circle all outliers with a red pencil.

**ACTION:** Qualify both positive results and non-detects for the outlier compound(s) as estimated, “J”. When %D is above 90%, qualify all positive results for that analyte “J” and all non-detect results for that analyte “R”.

**NOTE:** The above data qualification action applies regardless of method requirements.

15.5 Do any volatile compounds have a RRF < 0.05?  

**NOTE:** (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

<table>
<thead>
<tr>
<th>Compound</th>
<th>RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloromethane</td>
<td>0.10</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>0.10</td>
</tr>
<tr>
<td>Bromoform</td>
<td>0.10</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>0.30</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>0.30</td>
</tr>
</tbody>
</table>

**ACTION:** Circle all outliers with a red pencil.

**ACTION:** If RRF < 0.05, or < the requirements for the 5 compounds is section 15.5 above, qualify all positive results for that analyte “J” and all non-detect results for that analyte “R”.

**NOTE:** The above data qualification action applies regardless of method requirements.

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard (IS) areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to +100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)?  

- 28 VOA -
ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

Sample ID | IS # | Area Lower Limit | Area Upper Limit
--- | --- | --- | ---

(Attach additional sheets if necessary.)

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when the associated IS are counts area > + 100%.

3. If the IS area is below the lower limit (< -50%), qualify all associated non-detects (U-values) "J".

4. If extremely low area counts are reported (< -25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

-29 VOA-
17.0 Field Duplicates

17.1 Were any field duplicates submitted for volatile analysis?

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.
Sample ID: VWAI-MW07-1112
Duplicate Sample ID: VWAI-MW07P-1112

Water: RPD>30%
Soil: RPD>30%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>benzene</td>
<td>0.82</td>
<td></td>
<td>200</td>
</tr>
</tbody>
</table>

COMMENTS: Qualify benzene as estimated.

* one of the results below the LOD
only results with both above the LOD are reported
### Initial Calibration Date:
11/26/2012

### RRF and %RSD Calculations:

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Area of Compound</th>
<th>Area of Internal STD</th>
<th>Conc. of Internal STD</th>
<th>Conc. of Compound</th>
<th>Calculated RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-dichloroethane</td>
<td>0.546</td>
<td>1025756</td>
<td>939357</td>
<td>50</td>
<td>100</td>
<td>0.546</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>RRF of STD 1</th>
<th>RRF of STD 2</th>
<th>RRF of STD 3</th>
<th>RRF of STD 4</th>
<th>RRF of STD 5</th>
<th>RRF of STD 6</th>
<th>Calculated % RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>benzene</td>
<td>7.7</td>
<td>0.9470</td>
<td>0.9830</td>
<td>0.8970</td>
<td>0.8660</td>
<td>0.7840</td>
<td>0.9050</td>
<td>7.7</td>
</tr>
</tbody>
</table>

### Continuing Calibration File ID:
11/30/2012

### RRF and %D Calculations:

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Area of Compound</th>
<th>Area of Internal STD</th>
<th>Conc. of Internal STD</th>
<th>Conc. of Compound</th>
<th>Calculated RRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-dichloropropane</td>
<td>0.460</td>
<td>386446</td>
<td>840654</td>
<td>50</td>
<td>50</td>
<td>0.460</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
<th>Average RRF</th>
<th>Calibration Check RRF</th>
<th>Calculated % D</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-dichloroethane</td>
<td>4.0</td>
<td>0.596</td>
<td>0.572</td>
<td>4.0</td>
</tr>
</tbody>
</table>
## SAMPLE CALCULATION

**Sample ID:** VWAI-MW04-1112  
**Standard ID:** 11/30/2012  
**Compound:** benzene  
**Concentration:** 2.2 ug/L

<table>
<thead>
<tr>
<th></th>
<th>Water (ug/L)</th>
<th>Soil (ug/Kg)</th>
<th>Soil ug/Kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>52376</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>795127</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conc. of Internal (ng)</td>
<td>250</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>RRF of Compound</td>
<td>1.481</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dilution Factor</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>Weight of Sample</td>
<td>NA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of Sample</td>
<td>5</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>% Moisture</td>
<td>NA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aliquot of sample</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Concentration</td>
<td>2.22</td>
<td>#DIV/0!</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>RT of Internal STD</th>
<th>RT of Compound</th>
<th>RRT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td>4.476</td>
<td>4.232</td>
<td>0.945</td>
</tr>
<tr>
<td>Standard</td>
<td>4.477</td>
<td>4.233</td>
<td>0.945</td>
</tr>
</tbody>
</table>
The concentration of this analyte exceeds the calibration range of the instrument.

Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: L2472
LAB: Spectrum Analytical
SITE NAME: Vieques

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format? [X]

ACTION: If not, note the effect on review of the data in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present? [X]

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? [X]
II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples? 

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all non-detects data are qualified as unusable (R), and detects are flagged "J".

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

<table>
<thead>
<tr>
<th>Date</th>
<th>Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>11/28-29/12</td>
<td></td>
</tr>
<tr>
<td>11/29-30/12</td>
<td></td>
</tr>
<tr>
<td>12/18-19/12</td>
<td></td>
</tr>
</tbody>
</table>

2.1 Have any semivolatile technical holding times, determined from date of collection to date of extraction, been exceeded?

Continuous extraction of water samples for semivolatile analysis must be started within 7 days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within
40 days of the date of extraction.

Table of Holding Time Violations

(See Traffic Report)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Matrix</th>
<th>Date Sampled</th>
<th>Date Received</th>
<th>Date Extracted</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).
3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water

b. Low/Med Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water

b. Low/Med Soil

ACTION: If CLP deliverables are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk?  

ACTION: Circle all outliers in red.

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank (Reviewer should use lab in house recovery limits. Use surrogate recovery limits from USEPA National Functional Guidlines January 2005 page 130, if in house limits are not available. See Method 8000B-43 or 80000C-24).

Note: Examine lab in house limits for reasonableness.

If yes, were samples re-analyzed?
<table>
<thead>
<tr>
<th>EPA SAMPLE NO.</th>
<th>SDMC1 (NBZ)</th>
<th>SDMC2 (FBP)</th>
<th>SDMC3 (TPH)</th>
<th>TOT OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td>01 MB-69471</td>
<td>96</td>
<td>94</td>
<td>105</td>
<td>0</td>
</tr>
<tr>
<td>02 LCS-69471</td>
<td>99</td>
<td>100</td>
<td>106</td>
<td>0</td>
</tr>
<tr>
<td>03 MB-69496</td>
<td>97</td>
<td>95</td>
<td>104</td>
<td>0</td>
</tr>
<tr>
<td>04 LCS-69496</td>
<td>86</td>
<td>87</td>
<td>91</td>
<td>0</td>
</tr>
<tr>
<td>05 LCSD-69496</td>
<td>85</td>
<td>86</td>
<td>90</td>
<td>0</td>
</tr>
<tr>
<td>06 VWAI-MW04-11</td>
<td>84</td>
<td>81</td>
<td>57</td>
<td>0</td>
</tr>
<tr>
<td>07 VWAI-EB01-11</td>
<td>84</td>
<td>83</td>
<td>78</td>
<td>0</td>
</tr>
<tr>
<td>08 VWAI-MW05-11</td>
<td>82</td>
<td>81</td>
<td>37*</td>
<td>1</td>
</tr>
<tr>
<td>09 VWAI-MW07P-1</td>
<td>87</td>
<td>84</td>
<td>33*</td>
<td>1</td>
</tr>
<tr>
<td>10 VWAI-EB01-11</td>
<td>84</td>
<td>83</td>
<td>83</td>
<td>0</td>
</tr>
<tr>
<td>11 VWAI-MW04-11</td>
<td>80</td>
<td>80</td>
<td>50*</td>
<td>1</td>
</tr>
<tr>
<td>12 VWAI-MW04-11</td>
<td>81</td>
<td>82</td>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>13 VWAI-MW07-11</td>
<td>90</td>
<td>88</td>
<td>65</td>
<td>0</td>
</tr>
</tbody>
</table>

**QC LIMITS**

- SDMC1 (NBZ) = Nitrobenzene-d5 (40-110)
- SDMC2 (FBP) = 2-Fluorobiphenyl (50-110)
- SDMC3 (TPH) = Terphenyl-d14 (50-135)

*Column to be used to flag recovery values

* Values outside of contract required QC limits

D DMC diluted out
Were method blanks re-analyzed?

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

1. Flag all positive results as estimated ("J").

2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.

3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".

2. Non-detects for that fraction should be qualified as unusable (R).

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II?

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document
effect in data assessments.

4.0 Matrix Spikes (Form III/Equivalent)

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate/or duplicate unspiked Sample recoveries been listed on the Recovery Form (Form III)?

NOTE: Method 3500B/page 4 states the spiking compounds:

<table>
<thead>
<tr>
<th>Base/neutrals</th>
<th>Acids</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>Acenaphthene</td>
<td>Phenol</td>
</tr>
<tr>
<td>2,4-Dinitrotoluene</td>
<td>2-Chlorophenol</td>
</tr>
<tr>
<td>Pyrene</td>
<td>4-Chloro-3-methylphenol</td>
</tr>
<tr>
<td>N-Nitroso-di-n-propylamine</td>
<td>4-Nitrophenol</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td></td>
</tr>
</tbody>
</table>

Note: Some projects may require the spiking of specific compounds of interest.

Note: See Method 8270D-sec 8.4.2 for deciding on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate. If samples are expected to contain target analytes, then laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratory should use a matrix spike and matrix spike duplicate pair.

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water

b. Low Solid

c. Med Solid
ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. It may be necessary to contact the lab to obtain the required data.

NOTE: If the data has not been reported on CLP equivalent form, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

4.3 Were matrix spikes performed at concentration equal to 100ug/L for acid compounds, and 200ug/l for base compounds (Method 3500B-4), or those specified in project plan. ___

4.4 How many semivolatile spike recoveries are outside Laboratory in house MS/MSD recovery limits (use recovery limits values in Method 8270D-43&44 Table 6 if in house values not available).

Water

☑ out of 3

Solids

☐ out of ___
4.5 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water

   0 out of 3

Solids

   ___ out of ____

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a Laboratory Control Sample (LCS) analyzed with each analytical batch?

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 Blanks (Form IV/Equivalent)

5.1 Is the Method Blank Summary (Form IV) present?

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch?

5.3 Has a method blank been analyzed either after
the calibration standard or at any other time during the analytical shift for each GC/MS system used?

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

5.4 Chromatography: review the blank raw data – chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for the semivolatiles?

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

6.2 Do any field/rinse/ blanks have positive results for target analytes and/or TICs (if required, see section 10 below)?
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

VWAI - EB01 - 112812
bio(2-ethylhexylphth) 1.55 (2.0)

VWAI - EB01 - 112912 no @

[Signature: no qual]
### Blank Action for Semivolatile Analyses

<table>
<thead>
<tr>
<th>Blank Type</th>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Action for Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detects</td>
<td>Not detected</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>&lt; CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td>≥ CRQL</td>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>= CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td>≥ CRQL</td>
<td>≥ CRQL</td>
<td></td>
<td>No qualification required</td>
</tr>
<tr>
<td>&gt; CRQL *</td>
<td>&lt; CRQL</td>
<td></td>
<td>Report CRQL value with a U</td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and &lt; blank contamination</td>
<td>Report concentration of sample with a U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>≥ CRQL and ≥ blank contamination</td>
<td>No qualification required</td>
<td></td>
</tr>
</tbody>
</table>

**NOTE:** Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

**NOTE:** If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required.

6.3 Are there field/rinse/equipment blanks associated with every sample? 

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

6.4 Was a instrument blank analyzed after each sample/dilution which contained a target compound?
that exceeded the initial calibration range.

6.5 Does the instrument blank have positive results for target analytes and/or TICs?

Note: Use professional judgement to determine if carryover occurred and qualify analytes accordingly.

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270D? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column.

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (Form V/Equivalent)

8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)?

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5
8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift?

8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?

**ACTION:** List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

<table>
<thead>
<tr>
<th>DATE</th>
<th>TIME</th>
<th>INSTRUMENT</th>
<th>SAMPLE NUMBERS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:** If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

**ACTION:** If mass assignment is in error, flag all associated sample data as unusable (R).

8.4 Have the ion abundances been normalized to m/z 198?

8.5 Have the ion abundance criteria been met for each instrument used?

**ACTION:** List all data which do not meet ion abundance criteria (attach a separate sheet).
ACTION: If ion abundance criteria are not met, take action specified in section 3.2

8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)

8.7 Have the appropriate number of significant figures (two) been reported?

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

8.8 Are the spectra of the mass calibration compound acceptable?

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

9.0 Target Analytes

9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:
   a. Samples and/or fractions as appropriate
   b. Matrix spikes and matrix spike duplicates
   c. Blanks

9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270D-14)?
ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

   a. Samples and/or fractions as appropriate

   b. Matrix spikes and matrix spike duplicates (Mass spectra not required)

   c. Blanks

ACTION: If any data are missing, take action specified in 3.2 above.

9.4 Are the response factors shown in the Quant Report?

9.5 Is chromatographic performance acceptable with respect to:

   Baseline stability?

   Resolution?

   Peak shape?

   Full-scale graph (attenuation)?

   Other: ____________________________

ACTION: Use professional judgement to determine the acceptability of the data.

9.6 Are the lab-generated standard mass spectra of identified semivolatile compounds present for
each sample?

**ACTION:** If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

**ACTION:** When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

9.8 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

9.9 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?

**ACTION:** Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.7, 9.8, and 9.9.
10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form Is, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?

NOTE: Review sampling reports to determine if the lab was required to identify non target analytes (refer to section 7.6.2, page 8270D-21).

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate

b. Blanks

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)?

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the...
sample mass spectrum?

10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R."

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and, for soils, sample moisture?
ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration?

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI/Equivalent)

13.1 Is the Initial Calibration Form (Form VI/Equivalent) present and complete for the semivolatile fraction?

ACTION: If any calibration forms or standard row data are missing, take action specified in 3.2 above.

13.2 Are all base neutral or acid RRFs > 0.050?
Check the average RRFs of the four System Performance Check Compounds (SPCCs):
N-nitroso-di-n-propylamine, hexachlorocyclopentadiene,
2,4-dinitrophenol, and 4-nitrophenol. These compounds must have average RRFs greater than or equal to 0.05 before running samples and should not show any peak tailing.

**ACTION:** Circle all outliers in red.

**ACTION:** For any target analyte with average RRF < 0.05
1. "R" all non-detects;
2. "J" all positive results.

13.3 Are response factors for base neutral or acid target analytes stable over the concentration range of the calibration (% Relative standard deviation [%RSD] < 20.0%)?

**NOTE:** The % RSD for each individual Calibration Check Compound (CCC, Method 8270D-40 see Table 4) must be less than 30% before analysis can begin. If greater 30%, the lab must clean and recalibrate the instrument.

### CALIBRATION CHECK COMPOUNDS

<table>
<thead>
<tr>
<th>Base/Neutral Fraction</th>
<th>Acid Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acenaphthene</td>
<td>4-Chloro-3-methylphenol</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>2,4-Dichlorophenol</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>2-Nitrophenol</td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>Phenol</td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>2,4,6-Trichlorophenol</td>
</tr>
</tbody>
</table>
Benzo(a)pyrene

**ACTION:** If the %RSD for any CCC >30% and no corrective action taken, then "J" qualify all positive hits and "UJ" qualify all non-detects.

**ACTION:** Circle all outliers in red.

**ACTION:** If the % RSD is ~20.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detect results for that analyte "R," unusable. Alternatively, the lab should calculate first or second order regression fit of the calibration curve and select the fit which introduces the least amount of error.

**NOTE:** Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

13.4 Did the laboratory calculate the calibration curve by the least squares regression fit? [ ]

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.) [ ]

**ACTION:** Circle Errors in red.

**ACTION:** If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.5 Do the target compounds for this SDG include Pesticides? [ ]
13.6 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%? [ ] [ ]

**ACTION:** If DDT percent breakdown exceeds 20%:

i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "IN".

14.0 GC/MS Calibration Verification (Form VII/Equivalent)

14.1 Are the Calibration Verification Forms (Form VII) present and complete for all compounds of interest? [ ] [ ]

14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument? [ ] [ ]

**ACTION:** List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

________________________________________

________________________________________

________________________________________

**ACTION:** If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis,
call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

14.3 Do any of the SPCCs have an RRF < 0.05?

If YES, make a note in data assessment if the lab did not take corrective action specified in section 7.4.4, page 8270D-18.

14.4 Do any of the CCCs have a %D between the initial and continuing RRF which exceeds 20.0%?

ACTION: If yes, make a note in data assessment.

14.5 Do any semivolatile compounds have a % Difference (% D) between the initial and continuing RRF which exceeds 20.0%?

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is above 90%, qualify all non-detects for that analyte as "R", unusable.

14.6 Do any semivolatile compounds have a RRF < 0.05?

ACTION: Circle all outliers in red.

ACTION: If RRF < 0.05, qualify as unusable ("R") associated non-detects and "J" associated positive values.

14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more).
ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?

ACTION: List each outlying internal standard below.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>IS #</th>
<th>Area</th>
<th>Lower Limit</th>
<th>Upper Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Attach additional sheets if necessary.)

Note: Check Table 5, 8270D-41 for associated analytes.

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.
iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard? ☑️

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Laboratory Control Samples (LCS)

16.1 Were any LCS samples run in order to verify analytes which failed criteria for spike recovery? ☑️

16.2 Did the lab spike LCS sample spiked with the same analytes and the same concentrations as the matrix spike? ☑️

16.3 Were the mean and standard deviation of all analytes within the QC acceptance ranges as shown in Table 6, 8270D-43? ☑️

ACTION: If the recovery of any analyte falls out of the designated range, the analytical results for that compound is suspect and should be qualified "J" in the unspiked samples.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for semivolatile analysis? ☑️
ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

VWAI-MW07-1112 > qual.
VWAI-MW07P-1112
All attached
**FIELD DUPLICATE SAMPLE SUMMARY**

Sample ID: VWAI-MW07-1112  
Duplicate Sample ID: VWAI-MW07P-1112

Water: RPD>30%  
Soil: RPD>30%

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Conc.</th>
<th>Dup. Sample Conc.</th>
<th>%RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-methylnaphthalene</td>
<td></td>
<td>1.1</td>
<td>200</td>
</tr>
</tbody>
</table>

* one or both values below LOD

COMMENTS: Qualify as estimated.
**DataQual**

**Initial Calibration Date:** 12/18/2012  
**RRF and %RSD Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>naphthalene</td>
<td>0.756</td>
</tr>
</tbody>
</table>

- **Area of Compound** : 348674  
- **Area of Internal STD** : 230477  
- **Conc. of Internal STD** : 40  
- **Conc. of Compound** : 80  
- **Calculated RRF** : 0.756

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-methylnaphthalene</td>
<td>12.5</td>
</tr>
</tbody>
</table>

- **RRF of STD 1** : 0.818  
- **RRF of STD 2** : 0.788  
- **RRF of STD 3** : 0.82  
- **RRF of STD 4** : 0.668  
- **RRF of STD 5** : 0.692  
- **RRF of STD 6** : 0.579  
- **RRF of STD 7** : 0.788  
- **Calculated % RSD** : 12.45

**Continuing Calibration File ID:** 12/19/2012  
**RRF and %D Calculations:**

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>bis(2-ethylhexyl)phthalate</td>
<td>0.725</td>
</tr>
</tbody>
</table>

- **Area of Compound** : 205333  
- **Area of Internal STD** : 452887  
- **Conc. of Internal STD** : 40  
- **Conc. of Compound** : 25  
- **Calculated RRF** : 0.725

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Lab Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>naphthalene</td>
<td>7.5</td>
</tr>
</tbody>
</table>

- **Average RRF** : 0.962  
- **Calibration Check RRF** : 1.034  
- **Calculated % D** : -7.5
**Sample Calculation**

Sample ID: VWAI-MW04-112  
Standard ID: 12/18/2012  
Compound: naphthalene  
Concentration: 1.6 J ug/L

<table>
<thead>
<tr>
<th></th>
<th>Water (ug/L)</th>
<th>Soil (ug/Kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of Compound</td>
<td>7031</td>
<td></td>
</tr>
<tr>
<td>Area of Internal STD</td>
<td>185698</td>
<td></td>
</tr>
<tr>
<td>Conc. of Internal (ng)</td>
<td>40</td>
<td>2</td>
</tr>
<tr>
<td>RRF of Compound</td>
<td>0.962</td>
<td></td>
</tr>
<tr>
<td>Final Volume</td>
<td>1000</td>
<td>1000</td>
</tr>
<tr>
<td>Dilution Factor</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>GPC Factor</td>
<td>NA</td>
<td>1</td>
</tr>
<tr>
<td>Injection Volume</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Weight of Sample</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Initial Volume of Sample</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td>% Moisture</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Concentration</td>
<td>1.57</td>
<td>#DIV/0!</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>RT of Internal STD</th>
<th>RT of Compound</th>
<th>RRT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td>5.71</td>
<td>5.727</td>
<td>1.003</td>
</tr>
<tr>
<td>Standard</td>
<td>5.716</td>
<td>5.733</td>
<td>1.003</td>
</tr>
</tbody>
</table>
A.1.1 Contract Compliance Screening Report

Present?

ACTION: If no, contact RSCC/PO.

A.1.2 Record of Communication (from RSCC)

Present?

ACTION: If no, request from the RSCC.

A.1.3 Sampling Trip Report

Present and complete?

ACTION: If no, contact RSCC/PO.

A.1.4 Chain of Custody/Sample Traffic Report

Present? Legible?

Signature of sample custodian present?

ACTION: If no, contact RSCC/WAM/PO.

A.1.5 Cover Page

Present?

Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?

Do the sample identification numbers on the Cover Page agree with sample identification numbers on:

(a) Traffic Report Sheet?
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(b) Form I's?

Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data case? [ ] [ ] [N/A]

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 SDG Narrative, DC-1 & DC-2 Form

Is the SDG Narrative present? [ ] [ ] [N/A]

Is Sample Log-In Sheet (Form DC-1) present and complete? [ ] [ ] [V]

Is Complete SDG Inventory Sheet (Form DC-2) present and complete? [ ] [ ] [V]

**ACTION:**
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

A.1.7 Form I to XV

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name? [ ] [ ] [V]

Laboratory Code? [ ] [ ] [V]

RAS/Non-RAS Case No.? [ ] [ ] [N/A]

SDG No.? [ ] [ ] [V]
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Contract No.?

ACTION:
If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

A.1.7.2
After comparing values on Forms I-IX against the raw data, do any computation/transcription errors exceed 10% of the reported values on the Forms for:

(a) all analytes analyzed by ICP-AES?
(b) all analytes analyzed by ICP-MS?
(c) Mercury?
(d) Cyanide?

ACTION:
If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 Raw Data
Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 Digestion/Distillation Log

Digestion Log for ICP-AES (Form XII) present?

Digestion Log for ICP-MS (Form XII) present?

Digestion Log for mercury (Form XII) present?

Distillation Log for cyanide (Form XII) present?

Are pH values for metals and
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<table>
<thead>
<tr>
<th>SOP: HW-2 Revision 13</th>
<th>Appendix A.1</th>
<th>Sept. 2006</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th><strong>Question</strong></th>
<th><strong>YES</strong></th>
<th><strong>NO</strong></th>
<th><strong>N/A</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyanide reported for each aqueous sample?</td>
<td></td>
<td>[ ]</td>
<td>[x]</td>
</tr>
<tr>
<td>Are percent solids calculations present for soils/sediments?</td>
<td></td>
<td>[ ]</td>
<td>[x]</td>
</tr>
<tr>
<td>Are preparation dates present on the sample preparation logs/bench sheets?</td>
<td>[✓]</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**NOTE:**
Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.

**A.1.8.2** Is the analytical instrument real-time printouts present for:

<table>
<thead>
<tr>
<th>Instrument</th>
<th><strong>YES</strong></th>
<th><strong>NO</strong></th>
<th><strong>N/A</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>ICP-AES?</td>
<td>[✓]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ICP-MS?</td>
<td>[ ]</td>
<td>[ ]</td>
<td>[x]</td>
</tr>
<tr>
<td>Mercury?</td>
<td>[ ]</td>
<td></td>
<td>[x]</td>
</tr>
<tr>
<td>Cyanide?</td>
<td>[ ]</td>
<td>[x]</td>
<td>[ ]</td>
</tr>
</tbody>
</table>

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:

<table>
<thead>
<tr>
<th>Requirement</th>
<th><strong>YES</strong></th>
<th><strong>NO</strong></th>
<th><strong>N/A</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Legible?</td>
<td>[✓]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Properly labeled?</td>
<td>[✓]</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Are all field samples, QC samples and field QC samples present on:

<table>
<thead>
<tr>
<th>Log/Printouts</th>
<th><strong>YES</strong></th>
<th><strong>NO</strong></th>
<th><strong>N/A</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Digestion/Distillation log?</td>
<td>[✓]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Instrument Printouts?</td>
<td>[✓]</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**ACTION:**
If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.
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A.1.9 **Technical Holding Times:** (Aqueous and soil samples)
(Examine sample Traffic Reports and digestion/distillation logs to
determine the holding time from the sample collection date to the sample
preparation date.)

A.1.9.1 Cyanide distillation (14 days) exceeded? [ ]
Mercury analysis (28 days) exceeded? [ ]
Other Metals analysis (180 days) exceeded? [ ]

**ACTION:**
If yes, reject (R) and red-line non-detects
and flag as estimated (J) results ≥ MDL even
if sample(s) was preserved properly.

**NOTE:**
In addition to qualifying the data,
a list of all samples and analytes
which exceeded the holding times must
be prepared. Report for each sample
the number of days that were exceeded.
(Subtract the sample collection date
from the sample preparation date).
Attach this list to the data review
narrative.

A.1.9.2 Is pH of aqueous samples for:
Metals Analysis ≤ 2? [ ]
Cyanide Analysis ≥ 12? [ ]

**ACTION:**
If no for any of the above, flag
non-detects as "R" and detects as "J".

A.1.9.3 Is the cooler temperature ≤ 10 °C? [ ]

**ACTION:**
If cooler temperature is >10 °C, flag
non-detects as "UJ" and detects as
"J".

A.1.10 **Final Data Correctness - Form I**

A.1.10.1 Are Form I's for all samples
present and complete?

**ACTION:**
If no, prepare Telephone Record Log and contact CLP PO/TOPO for submittal from the laboratory.

A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

Is the calculation error less than 10% of the correct result? [ ] [ ]

Are results on Form I's reported in correct units (\( \text{ug/L for aqueous and MG/KG for soils} \)?) [ ] [ ]

Are results on Form I's reported by correct significant figures? [ ] [ ]

Are soil sample results on Form I's corrected for percent solids? [ ] [ ]

Are all "less than MDL" values reported by the CRQLs and coded with "U"? [ ] [ ]

Are values less than the CRQLs but greater than or equal to the MDLs flagged with "J"? [ ] [ ]

Are appropriate contractual quality control and Method qualifiers used? [ ] [ ]

**ACTION:**
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3 Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data? [ ] [ ]

Was a brief physical description?
of the samples before and after digestion given on the Form I's? [ ] [ ] [ ]

Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I? [ ] [ ] [ ]

**ACTION:**
If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11 Initial Calibration

A.1.11.1 Is a record of at least 2 point (A blank and a standard) calibration present for ICP-AES analysis? [ ] [ ] [ ]

Is a record of at least 2 point (a blank and a standard) calibration present for ICP-MS analysis? [ ] [ ] [ ]

Is a record of at least 5 point calibration (a blank & 4 standards) present for Hg analysis? [ ] [ ] [ ]

Is a record of at least 4 point calibration (a blank & 4 standards) present for cyanide? [ ] [ ] [ ]

**ACTION:**
If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).

Is one initial calibration standard at the CRQL level for cyanide and mercury? [ ] [ ] [ ]

**ACTION:**
If no, write in the Contract Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11.2 Is the curve correlation coefficient ≥ 0.995 for:
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Mercury Analysis? YES NO N/A

Cyanide Analysis? YES NO N/A

ICP-AES (more than 2 point Calib.)? YES NO N/A

ICP-MS (more than 2 point calib.)? YES NO N/A

**ACTIONS:**
If no, qualify the associated sample results ≥ MDL as estimated "J" and non-detects as "UJ".

**NOTE:**
The correlation coefficient shall be calculated by the data validator using standard concentrations and the corresponding instrument response (e.g. absorbance, peak area, peak height, etc.).

A.1.12 Initial and Continuing Calibration Verification—Form IIA

A.1.12.1 Present and complete for every metal and cyanide? YES NO N/A

Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte? YES NO N/A

**ACTIONS:**
If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.

A.1.12.2 Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent? YES NO N/A

**ACTIONS:**
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.12.3 Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples? YES NO N/A
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**A.1.12.2** Circle on each Form IIA all percent recoveries that are outside the contract windows.

Are ICV/CCVs within control limits for:
- Metals - 90-110%R?
- Hg - 80-120%R?
- Cyanide - 85-115%R?

**ACTION:**
If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89% (65-79% for Hg; 70-84% for CN).
Qualified only positive results (≥ MDL) as “J” if the ICV/CCV %R is between 111-125% (121-135% for Hg; 116-130% for CN). Reject (R) and red-line only detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detects) if the recovery is less than 75% (65% for Hg; 70% for CN).

**NOTE:**
For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

**A.1.12.3** Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)?

**ACTION:**
If no, Qualify all cyanide results ≥ MDL as “J”.

**A.1.13** CRQL Standard Analysis - Form IIB

**A.1.13.1** For each ICP-AES run, was a CR!
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---

**A.1.13.2** Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis?

**ACTION:**
If no, write in the Contract Problems/Non-Compliance Section of the Data Review Narrative.

---

**A.1.13.3** Circle on each Form II B all percent recoveries that are outside the acceptance windows.

---

**Note:** CRQL is not required for Al, Ba, Ca, Fe, Mg, Na and K.
Is the CRQL standard within control limits for:

- Metals (ICP-AES/ICP-MS) - 70 - 130%?
- Mercury - 70 - 130%?
- Cyanide - 70 - 130%?

**ACTION:**
If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag (J) only detects <2xCRQL if the recovery is between 131% and ≤180%. If the recovery is less than 750%, reject (R) and red-line non-detects and detects <2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detects <2xCRQL and flag (J) detects ≥2xCRQL but < ICV/CCV if the recovery is > 180%.

**NOTE:**
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard
2. Flag (J) or reject (R) only the final sample results on Form I’s when Sample raw data are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

**A.1.14 Initial and Continuing Calibration Blanks - Form III**

**A.1.14.1** Present and complete for all the instruments used for the metals and cyanide analyses?

Was an initial Calibration Blank analyzed after ICV?

Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?

Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by

...
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using MDLs from direct analysis(Preparation Method "NP1")?
(Check Form III against the raw data)

**ACTION:**
If no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".

A.1.14.2 Circle with red pencil on each Form III all Calib. Blank values that are:

- \[ \geq \text{MDL but } \leq \text{CRQL} \]
- \[ > \text{CRQL} \]

A.1.14.2.1 When MDL < CRQL, is any Calib. Blank value \[ \geq \text{MDL but } \leq \text{CRQL} \]?

**ACTION:**
If yes, change sample results \[ \geq \text{MDL but } \leq \text{CRQL} \] to the CRQL with a "U".
Do not qualify non-detects.

A.1.14.2.2 When MDL < CRQL, is any Calib. Blank value \[ > \text{CRQL} \]?

**ACTION:**
If yes, reject (R) and red line the associated sample results \[ > \text{CRQL} \] but \( < \text{ICB/CCB Blank Result. Flag as "J" detects } \text{ICB/CCB blank value but} \leq 10\times \text{ICB/CCB value. Change the sample results } \geq \text{MDL but } \leq \text{the CRQL to CRQL with a "U".} \]

A.1.14.2.3 Is any Calibration Blank value below the negative CRQL?

**ACTION:**
If yes, flag (J) as estimated all associated sample results \[ \geq \text{CRQL but} \leq 10\times \text{CRQL} \]

**NOTE:**
1. For ICB that does not meet the technical QC Criteria, apply the action to all samples.
2. For CCBs that do not meet the technical QC criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

A.1.15 Preparation Blank - FORM III

NOTE: The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1 Was one Preparation Blank prepared with and analyzed for:

- Each Sample Delivery Group (SDG)?
- Each batch of the SDG samples digested/distilled?
- Each matrix type?
- All instruments used for metals and cyanide analyses?

**ACTION:**
If no for any of the above, flag as estimated (J) all the associated positive data <10xMDL for which the Preparation Blank was not analyzed.

**NOTE:**
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated (J), but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

- ≥ MDL but ≤ CRQL, and
- > CRQL

A.1.15.2.1 When MDL < CRQL, is any preparation blank value ≥ MDL but ≤ CRQL?

**ACTION:**
If yes, change sample result ≥ MDL
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but ≤ CRQL to CRQL with a “U”.

A.115.2.2 When the MDL ≤ CRQL, is any Preparation
Blank value greater than its CRQL?

If yes, is the Prep. Blank value
greater than the value of the associated
Field Blank collected and analyzed with
the SDG samples?

If yes, is the lowest concentration of
that analyte in the associated samples
less than 10 times the Preparation
Blank value?

ACTION:
If yes, reject (R) and red-line all associated
sample results greater than the CRQL but less
than the Prep. Blank value. Flag as “J”
If the sample result ≥ MDL but ≤ CRQL, replace
it with CRQL-U.

If the Prep. Blank value is less than the same
analyte value in the Field Blank, do not
qualify the sample results due to the
Prep. Blank criteria.

NOTE:
Convert soil sample result to mg/Kg on
wet weight basis to compare with the soil
Prep. Blank result on Form III.

A.115.2.3 Is the Prep. Blank concentration
below the negative CRQL?

ACTION:
If yes, flag (J) all associated
sample results less than 10xCRQL.
Qualify non-detects as estimated (UJ).

A.115.2.4 When the MDL is greater than the
CRQL, is the preparation blank
concentration on Form III greater
than two times the MDL?

ACTION:
If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.

A.1.16 ICP-AES/ICP-MS Interference Check Sample (ICS) - Form IV

**NOTE:** Not required for Cn, Hg, Al, Ca, Fe and Mg.

A.1.16.1 Present and complete?

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
</table>

Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples?

ACTION:
If no, flag as estimated (J) all sample results.

A.1.16.2 ICP-AES Method

A.1.16.2.1 ICSA Solution:
For ICP-AES, are the ICSA "Found" analyte values within the control limits \( \pm \) of CRQL of the true/established mean value?

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV?

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results \( \geq \) MDL.
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A.1.16.2.3 ICSAB Solution
For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value?

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79%, qualify sample results ≥ MDL as "J" and non-detects as "UJ". Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.

A.1.16.3 ICP-MS Method
A.1.16.3.1 ICSA Solution:
For ICP-MS, are the ICSA "Found" analyte values within the control limits of ±CRQL of the true/established mean value?

ACTION:
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated only sample results ≥ MDL if the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag the associated sample detects as "J" and non-detects as "UJ".
A.1.16.3.3 ICSAB Solution

For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater?

ACTION:
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only detects (≥ MDL).

A.1.17 Spiked Sample Recovery: Pre-Digestion/Pre-Distillation—Form V A

Note: Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only)

Was Matrix Spike analysis performed:

For each matrix type?

For each SDG?

On one of the SDG samples?

For each concentration range (i.e., low, med., high)?

For each analytical Method (ICP-AES, ICP-MS, Hg, CN) used?

Was a spiked sample prepared and analyzed with the SDG samples?

ACTION:
If no for any of the above, flag as estimated (J) all the positive data for which a spiked sample was not analyzed.

NOTE:
If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.
A.1.17.2 Was a field blank or PE sample used for the spiked sample analysis?

**ACTION:**
If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.

A.1.17.3 Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.

Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?

**NOTE:**
Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.

Are results outside the control limits (75-125%) flagged with Lab Qualifier "N" on Form I's and Form VA?

**ACTION:**
If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.

A.1.17.4 Aqueous

Are any spike recoveries:

(a) less than 30%?
(b) between 30-74%?
(c) between 126-150%?
(d) greater than 150%?

**ACTION:**
If the matrix spike recovery is less than 30%, reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as “J” and non-detects...
as "UJ". If between 126-150%, flag (J) all data ≥ MDL as "J". If greater than 150%, reject (R) and red-line all associated data ≥ MDL.

(NOTE: Replace "N" with "J" or "R" as appropriate.)

A.1.17.5 **Soil/Sediment**

Are any spike recoveries:

(a) less than 10%?

(b) between 10-74%?

(c) between 126-200%?

(d) greater than 200%?

**ACTION:**

If yes for any of the above, proceed as follows:

- If the matrix spike recovery is less than 10%, reject (R) and red-line all associated data (detects & non-detects);
- if between 10-74%, qualify all associated data ≥ MDL as "J" and non-detects as "UJ";
- if between 126-200%, flag (J) all associated data ≥ MDL as "J" If greater than 200%, reject (R) and red-line all associated data ≥ MDL.

(NOTE: Replace "N" with "J" or "R" as appropriate.)

A.1.18 **Lab Duplicates** - Form VI

A.1.18.1 Was the lab duplicate analysis performed:

- For each SOG?
- On one of the SOG samples?
- For each matrix type?
- For each concentration range (low or med.)?
- For each analytical Method (ICP-AES/ICP-MS,Hg,CN)Used?
- Was a lab duplicate prepared and analyzed with the SOG samples?
ACTION:
If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.

NOTE:
If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

A.1.18.2 Was a Field Blank or PE sample used for the Lab Duplicate analysis?

ACTION:
If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.

A.1.18.3 Circle on each Form VI all values that are:

- RPD > 20%, or
- Absolute Difference > CRQL

Are all values within control limits (RPD < 20% or absolute difference ≤ CRQL)? [__] [__] [__]

If no, are all results outside the control limits flagged with an "*" (Lab Qualifier) on Form VI and on all Form I's? [__] [__] [__]

ACTION:
If no, write in the Contract-Problems/Non-Compliance Section of the Data Review Narrative.

NOTE:
The laboratory is not required to report on Form VI the RPD when both values are non-detects.

A.1.18.4 Aqueous

A.1.18.4.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

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<table>
<thead>
<tr>
<th>SOP: HW-2</th>
<th>Revision 13</th>
<th>Appendix A.1</th>
<th>Sept. 2006</th>
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</thead>
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<td></td>
<td>YES</td>
<td>NO</td>
<td>N/A</td>
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</table>

<table>
<thead>
<tr>
<th>is any RPD &gt; 20% but &lt; 100%?</th>
<th>[___]</th>
<th>[___]</th>
<th>[✓]</th>
</tr>
</thead>
<tbody>
<tr>
<td>is any RPD ≥ 100%?</td>
<td>[___]</td>
<td>[___]</td>
<td>[✓]</td>
</tr>
</tbody>
</table>

**ACTION:**

If the RPD is > 20% but < 100%, flag (J) as estimated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

*(NOTE: Replace "*" with "J" or "R" as appropriate.)*

A.1.18.4.2 When the sample and/or duplicate value <5×CRQL (substitute MDL for CRQL when MDL > CRQL),

is the absolute difference between sample and duplicate values:

> ± CRQL?  
> ± 2×CRQL?

**ACTION:**

If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5×CRQL as "J" and non-detects as "UJ". If the absolute difference is > 2×CRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but < 5×CRQL.

*(NOTE: 1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.)*

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5×CRQL (substitute MDL for CRQL when MDL > CRQL),

<table>
<thead>
<tr>
<th>is any RPD ≥ 35% but &lt; 120%?</th>
<th>[___]</th>
<th>[___]</th>
<th>[✓]</th>
</tr>
</thead>
<tbody>
<tr>
<td>is any RPD ≥ 120%?</td>
<td>[___]</td>
<td>[___]</td>
<td>[✓]</td>
</tr>
</tbody>
</table>

**ACTION:**

If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample...
data ≥ CRQL. If the RPD is ≥ 120%, reject (R) and red-line the associated sample data ≥ CRQL.

A.1.18.5.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

> ± 2 x CRQL?
> ± 4 x CRQL

ACTION:
If the absolute difference is > 2 x CRQL, flag all the associated sample results ≥ MDL but <5xCRQL as “J” and non-detects as “UJ”.
If the absolute difference is >4xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but <5xCRQL.

NOTE:
1. Replace “**” with “J”, “UJ” or “R” as appropriate.
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.19 Field Duplicates

Aqueous Field Duplicates

A.1.19.1 Was an aqueous Field Duplicate pair collected and analyzed? (Check Sampling Trip Report)

ACTION:
If yes, prepare a Form (Appendix A.4) for each aqueous Field Duplicate pair. Report the sample and Field Duplicate results on Appendix A.4 from their respective Form I’s. Calculate and report RPD on Appendix A.4 when sample and its Field Duplicate values are both > 5xCRQL. Calculate and report the absolute difference on Appendix A.4 when at least one value (sample or duplicate) is <5xCRQL. Evaluate the aqueous Field Duplicate analysis in accordance with the
QC criteria stated in Sections A.1.19.2 and A.1.19.3.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

RPD $\geq$ 20% or

Difference $>$ $\pm$ CRQL

When sample and duplicate values are both $\geq$ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD $\geq$ 20%?  

is any RPD $\geq$ 100%?  

ACTION:
If the RPD is $>$ 20% but $<$ 100%, flag (J) only the associated sample and its Field Duplicate results $\geq$ CRQL. If the RPD is $\geq$ 100%, reject (R) and red-line only the associated sample and its Field Duplicate result $\geq$ CRQL.

A.1.19.3 When the sample and/or duplicate value(s) < 5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

$>$ $\pm$ CRQL?  

$>$ $\pm$ 2 x CRQL?  

ACTION:
If the absolute difference is $>$ CRQL, flag detects $\geq$ MDL but $<$ 5xCRQL as "J" and non-detects as "UJ". If the difference is $>$ 2xCRQL, reject (R) and red-line non-detects.
and results $\geq$ MDL but $< 5 \times \text{CRQL}$ of the sample and its Field Duplicate.

**Soil/Sediment Field Duplicates**

A.1.19.4 Was a soil field duplicate pair collected and analyzed? (Check Sampling Trip Report)

[A.1.19.5] Is a soil field duplicate pair collected and analyzed?

**ACTION:**
If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than $5 \times \text{CRQL}$. Calculate and report the absolute difference when at least one value (sample or duplicate) is $< 5 \times \text{CRQL}$. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

**NOTE:**
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is $> \text{CRQL}$ and the other value is non-detect, calculate the absolute difference between the value $> \text{CRQL}$ and the MDL, and apply the criteria to qualify the results.

A.1.19.5 Circle on each Appendix A.4 all values that have:

RPD $\geq 35\%$, or Difference $> \pm 2 \times \text{CRQL}$
When sample and duplicate values are both $\geq 5 \times \text{CRQL}$ (substitute MDL for CRQL when MDL > CRQL),

Is any RPD $\geq 35\%$ but $< 120\%$?

Is any RPD $\geq 120\%$?

**ACTION:**
If the RPD is $\geq 35\%$ but $< 120\%$,
flag only the associated sample and its Field Duplicate results ≥ CRQL as “J”. If the RPD is ≥ 120%, reject (R) and red-line only the sample and its Field Duplicate results ≥ CRQL.

A.1.19.6 When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and Field Duplicate:

> ± 2 x CRQL? __ [__] ___

> ± 4 x CRQL? __ [__] ___

**ACTION:**
If the absolute difference is > 2xCRQL, flag Sample and its Field Duplicate results ≥ MDL but <5xCRQL as “J” and non-detects as “UJ”. If the difference is >4xCRQL, reject (R) and red-line non-detects and detects ≥ MDL but <5xCRQL of the sample and its Field Duplicate.

A.1.20 **Laboratory Control Sample (LCS) – Form VII**

A.1.20.1 Was one LCS prepared and analyzed for:

Each SDG? [✓] ___ ___

Each matrix type? [✓] ___ ___

Each batch samples digested/distilled? [✓] ___ ___

For each Method (ICP-AES, ICP-MS, Hg, CN) used? [✓] ___ ___

Was an LCS prepared and analyzed with the samples? [✓] ___ ___

**ACTION:**
If no for any of the above, prepare Telephone Record Log and contact CLP PO or TOPO for submittal of the LCS results. Flag (J) as estimated all the data for which an LCS was not analyzed.

**NOTE:**
If only one LCS was analyzed for
more than 20 samples, then the first
20 samples analyzed are not flagged (J),
but all additional samples must be
qualified (J).

A.1.20.2 Aqueous LCS

Circle on each Form VII the LCS percent
recoveries outside control limits 80-120%.

NOTE: 1. Use digested as LCS for aqueous mercury
2. Use distilled as LCS for aqueous cyanide

Is any LCS recovery:

Less than 50%? [ ] [ ]
Between 50% and 79%? [ ] [ ]
Between 121% and 150%? [ ] [ ]
Greater than 150%? [ ] [ ]

ACTION:
If the LCS recovery is less than 50%,
reject (R) and red-line all associated
sample data (detects & non-detects); for
a recovery between 50-79%, flag detects
as "J", all non-detects as "UJ", if the LCS
recovery is between 121-150%, flag only
detects as "J", if the recovery is greater
than 150%, reject (R) and red-line all detects.

A.1.20.3 Solid LCS

If an analyte's MDL is equal to or
greater than the true value of LCS,
disregard the "Action" below for that
analyte even though the LCS is out of
control limits.

Is the LCS "Found" value greater
than the Upper Control Limit
reported on Form VII? [ ] [ ]

ACTION:
If yes, flag (J) all the associated detects ≥ MDL as estimated (J).

Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII?

**ACTION:**
If yes, flag detects as "J" and non-detects as "UJ".

A.1.21 **ICP-AES/ICP-MS Serial Dilution – Form VIII**

**NOTE:** Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 x MDL.

A.1.21.1 Was a Serial Dilution analysis performed:

- For each SDG? [ ] [ ] [ ]
- On one of the SDG samples? [ ] [ ] [ ]
- For each matrix type? [ ] [ ] [ ]
- For each concentration range (low or med.)? [ ] [ ] [ ]
- Was a Serial Dilution sample analyzed with the SDG samples? [ ] [ ] [ ]

**ACTION:**
If no for any of the above, flag as estimated (J) detects ≥ MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.

A.1.21.2 Was a Field Blank or PE sample used for the Serial Dilution Analysis?

[ ] [ ] [ ]

**ACTION:**
If yes, flag as estimated (J) detects ≥ MDL of all the SDG samples

A.1.21.3 Circle on Form VIII the Percent Differences (%) between sample results and its dilution results that are outside the control limits ± 10%
when initial concentrations ≥ 50 x MDLs.

Are results outside the control limits flagged with an "E" (Lab Qualifier) on Form VIII and all Form I's?

**ACTION:**
If no, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.21.4 Are any %D values:

> 10%?

≥ 100%?

**ACTION:**
If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose raw data ≥ MDL; if the %D is ≥ 100%, reject (R) and red-line all associated samples with raw data ≥ MDL.

*(NOTE: Replace "E" with "J" or "R" as appropriate.)*

### Total/Dissolved or Inorganic/Total Analytes

A.1.22.1 Were any analyses performed for dissolved as well as total analytes on the same sample(s)?

Were any analyses performed for inorganic as well as total analytes on the same sample(s)?

**ACTION:**
If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic) and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:

(1) The dissolved (or inorganic) concentration is greater than total concentration, and
(2) greater than or equal to 5xMDL.

A.1.22.2 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%?
A.1.22.3 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 50%?  

**ACTION:**

If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23 Field Blank - Form I  
**NOTE:** Designate "Field Blank" as such on Form I

A.1.23.1 Was a Field/Rinsate Bank collected and analyzed with the SDG samples?  

If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL (or 2xMDL when MDL>CRQL)?

If yes, circle the Field Blank value on Form I that is greater than the CRQL, (or 2 x MDL when MDL > CRQL).  

Is any Field Blank value greater than CRQL also greater than the Preparation Blank value?

If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria?

**ACTION:**

If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results) greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result > MDL but < CRQL, replace it with CRQL-U.

If the Field Blank value is less than the
Prep. Blank value, do not qualify the sample results due to the Field Blank criteria.

NOTE:
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinse Blank associated with soils to qualify water samples and vice versa.

A.1.24 Verification of Instrumental Parameters - Form IX, XA, XB, XI

A.1.24.1 Is verification report present for:

Method Detection Limits (Form IX - Annually)?

ICP-AES Interelement Correction Factors (Form XA & XB - Quarterly)?

ICP-AES & ICP-MS Linear Ranges (Form XI - Quarterly)?

ACTION:
If no, contact CLP PO/TOPO for submittal from the laboratory.

A.1.24.2 Method Detection Limits - Form IX

A.1.24.2.1 Are MDLs present on Form IX for:

All the analytes?

All the instruments used?

Digested and undigested samples and Calib. Blanks?

ICP-AES and ICP-MS when both instruments are used for the same analyte?

ACTION:
If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than 4\% CRQL.
A.1.24.2.2 Is MDL greater than the CRQL for any analyte?

If yes, is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?

**ACTION:**
If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.

A.1.24.3 Linear Ranges - Form XI

A.1.24.3.1 Was any sample result higher than the high linear range for ICP-AES or ICP-MS?

Was any sample result higher than the highest calibration standard for mercury or cyanide?

If yes for any of the above, was the sample diluted to obtain the result reported on Form I?

**ACTION:**
If no, flag (J) as estimated the affected detects (≥ MDL) reported on Form I.

A.1.25 ICP-MS Tune Analysis - Form XIV

A.1.25.1 Was the ICP-MS instrument tuned prior to calibration?

**ACTION:**
If no, reject (R) and red-line all sample data for which tuning was not performed.

A.1.25.2 Was the tuning solution analyzed or scanned at least five times consecutively?

Were all the required isotopes spanning the analytical range present in the tuning solution?

Was the mass resolution within
0.1 amu for each isotope in the tuning solution? [YES] [NO] [N/A]

Was %RSD less than 5% for each isotope of each analyte in the tuning solution? [YES] [NO] [N/A]

**ACTION:**
If no for any of the above, qualify all results > MDL associated with that Tune as estimated “J”, and all non-detects associated with that Tune as “UJ”.

### A.1.26 ICP-MS Internal Standards - Form XV

#### A.1.26.1 Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)? [YES] [NO] [N/A]

Were all the target analyte masses bracketed by the masses of the five internal standards? [YES] [NO] [N/A]

**ACTION:**
If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.

#### A.1.26.2 Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank? [YES] [NO] [N/A]

If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed? [YES] [NO] [N/A]

Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)? [YES] [NO] [N/A]

**ACTION:**
If no for any of the above, flag detects as “J” and non-detects “UJ” of all the analytes with atomic masses between the atomic mass of the internal standard lighter
Standard Operating Procedure
USEPA Region 2
Evaluation of Metals Data for the Contract Laboratory Program
Data Assessment and Contract Compliance Review

OP: HW-2 Revision 13  Appendix A.2  Sept. 2006

than the affected internal standard, and the
atomic mass of the internal standard heavier
than the affected internal standard.

A.1.27  Percent Solids of Sediments

A.1.27.1 Are percent solids in sediment(s):
< 50%?  

ACTION:
If yes, qualify as estimated (J) all detects and
non-detects of a sample that has percent solids
less than 50% (i.e., moisture content greater than 50%).

NOTE:
Flag(J) only the sample results
that were not previously flagged
due to other QC criteria.

Inorganic Data Review Narrative

Case#  Site:  Matrix: Soil
SDG#  Lab:  Water
Sampling Team:  Reviewer:  Other

A.2.1 Data Validation Flags:
The following flags may have been applied in red by the data validator and must
be considered by the data user.

J - This flag indicates the result qualified as estimated

R and Red-Line - A red-line drawn through a sample result indicates unusable value.
The red-lined data are known to contain significant errors based on
documented information and must not be used by the data user.

U - This data validation qualifier is applied to sample results
≥ MDL when associated blank is contaminated

Fully Usable Data - The results that do not carry "J" or "red-line" are fully usable.

A.2.2 Laboratory Qualifiers:
The CLP laboratory applies a contractual qualifier on all
**SAMPLE CALCULATION**

EPA SAMPLE ID: VWAI-MW04-1112  
COMPOUND: Manganese  
CONCENTRATION: 1140 ug/L  
% Solids - NA  

Raw Data result: 1.1432 mg/L

1.1432 mg/L (1000 ug/l mg) = 1143.2 ug/L

**FIELD DUPLICATE SAMPLE SUMMARY**

Note: All reported results are noted in the table below because the client requested that the MDL be used as reporting limit instead of the RL for this project. RPDs or absolute differences were calculated based on Region II guidelines: if results are > 5X RL RPD is calculated, if results are <5X RL the absolute difference is calculated. Flags are applied to field duplicate pair only as follows: For RPD values - RPD ≥ 35% but < 120% results are J, RPD > 120%, results are R. For absolute difference values - > +/- 2X RL results are J, > +/- 4X RL results are R.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Sample Conc.</th>
<th>Duplicate Conc.</th>
<th>RPD or absolute difference</th>
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Comments: No qualifications required.

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Comments: No qualifications required.

Review:

Date: 1/25/13
Final Responses to EPA Comments on the
Draft In-Situ Remediation Pilot Study Report, Area of Concern I (AOC I)
Former Atlantic Fleet Weapons Training Area- Vieques
Naval Ammunition Support Detachment
Vieques, Puerto Rico
March 2013

EPA General Comments

1. While Section 4 (Conclusions and Path Forward) provides information to substantiate that concentrations decreased at AOC I, the Pilot Study does not provide a sufficient discussion to substantiate that the concentration decreases were specifically related to the application of in-situ chemical oxidation (ISCO) and enhanced in-situ bioremediation (EISB) and not natural processes. In addition, the Pilot Study does not discuss how the application of ISCO and EISB met performance based criteria and data quality objectives (DQOs). Revise Section 4 to provide a discussion to substantiate that the application of ISCO and EISB met performance based criteria and DQOs.

Navy Response:

The following paragraphs have been added to the end of Section 3.2:

“As stated in Section 1.1, the objectives of the Pilot Study implemented at AOC I were to: (1) determine if the groundwater Pilot Study technologies could reduce COC concentrations to acceptable levels and (2) determine if the Pilot Study technologies could reduce the groundwater cleanup timeframe (relative to that predicted by natural attenuation alone). The associated project quality objective (PQO), as documented in Worksheet 11 of the Pilot Study SAP (CH2M HILL, 2010a), was to collect data sufficient for determining whether unacceptable risk associated with potential potable groundwater use at the site was mitigated (i.e., all COC concentrations below Pilot Study PRGs) and, therefore, no further action was warranted.

As noted previously, the concentrations of all groundwater COCs in all wells (except benzene and naphthalene in well MW07) had declined to below Pilot Study PRGs before the Pilot Study baseline sampling (i.e., between 2004 and 2010). For MW07, Table 6 summarizes the percent reduction of benzene and naphthalene in monitoring well MW-07 prior to and during the Pilot Study implementation. The table also includes 2-methylnaphthalene because it helps demonstrate the potential affect on COC concentration decline by natural processes and the Pilot Study technologies. As shown in the table, the concentrations of these three COCs declined between 74 percent and 79 percent over the 5 ½ years prior to the Pilot Study (i.e., under the influence of natural attenuation processes alone). During the 2 ½-year Pilot Study, the same COCs declined by about 95 percent.

In addition to the above, natural attenuation modeling (see Attachment C of the Pilot Study SAP [CH2M HILL, 2010a]) indicated it would take approximately 7 years for benzene and 14 years for naphthalene to decline from levels measured at AOC I in 2008 to the Pilot Study PRGs under the influence of natural attenuation processes alone. As shown in Figures 12 and 14, the Pilot Study PRGs for both of these two COCs were achieved in about 4 years (i.e., 2008 to 2012).

The information above indicates the decreases in COC concentrations were attributable to both natural processes and Pilot Study technologies, with the Pilot Study technologies likely accelerating the decline to below the PRGs. Regardless of the relative contribution of natural processes and Pilot Study technologies, the monitoring conducted before and during the Pilot Study indicated all COCs at the site declined to below the PRGs without rebound.”
The sub-bullets of the third bullet in Section 4 were revised as follows:

- “...(from 14 µg/L to 0.82 µg/L during the Pilot Study). Benzene concentrations declined naturally by 76 percent prior to the Pilot Study and by 94 percent following the ISCO injection and EISB application; overall concentrations declined by 99 percent. Benzene fell below its PRG of 5 µg/L between November 2011 and May 2012 and no rebound was observed.”
- “...(from 21 µg/L to non-detect during the Pilot Study). Naphthalene concentrations declined naturally by 74 percent prior to the Pilot Study and by 95 percent following the ISCO injection and EISB application; overall concentrations declined by 99 percent. Naphthalene fell below its PRG of 6.1 µg/L between November 2011 and May 2012 and no rebound was observed.”

2. Section 2.5 (Enhanced In-Situ Bioremediation) indicates that oxygen releasing compound (ORC) socks were placed in monitoring wells MW-02, MW-03, MW-04, MW-05, and MW-07 and were removed in August 2011 according to the schedule in the Final In-Situ Remediation Pilot Studies (AOC E and AOC I Sites) Sampling and Analysis Plan, Vieques, Puerto Rico, dated June 2008 (SAP); however, Section 2.5 does not discuss whether the ORC socks met the performance criteria expectations established in the SAP before being removed. Clarify whether the ORC socks met the performance criteria established in the SAP prior to being removed.

**Navy Response:**

Please see the response to Comment #1.

3. A discussion of how the geology and potential preferential pathways at the site may have impacted the pilot study is not included in the Pilot Study. Based on Figure 5 (Geologic Cross Section A-A’) and Figure 6 (AOC I Conceptual Site Model), the monitoring wells which were used for the pilot study injections (e.g., MW-02, MW-03, MW-04, and MW-07) were screened in highly fractured bedrock which may have created preferential pathways within the bedrock.

**Navy Response:**

The following was added at the end of the third paragraph of Section 2.2:

“Although fractures in the bedrock at AOC I may have provided preferential pathways for contaminant migration, the ISCO injections would have followed those same pathways since the injections were intentionally performed at very low pressures to avoid creating additional preferential flow pathways. Monitoring during injection was performed and showed no mounding in surrounding wells.”

4. Include a discussion of how the geology and potential preferential pathways at the site were evaluated and may have impacted the implementation of the pilot study injections.

**Navy Response:**

Please see the response to Comment #3.

5. The Pilot Study does not describe any measurements of the oxidant demand. For example, the Pilot Study does not discuss whether the oxidant demand at AOC I was solely due to the hydrocarbon release or if there is a background oxidant demand that affected anaerobic conditions in the saturated zone. Depending on the amount of nonaqueous phase liquid (NAPL) present and the extent of hydrocarbon weathering (loss of soluble and volatile constituents), the oxidative treatment may have been affected if constituents subsequently dissolved into anaerobic groundwater. Include a discussion of oxidant demand during the pilot study injections.
**Navy Response:**

With respect to the parameters measured during the Pilot Study, they were those concurred upon by the Navy, USEPA, and PREQB via the SAP process. Regarding oxidant demand, the following paragraph has been added as the first paragraph of Section 2.2:

“During the Pilot Study design, the oxidant (persulfate) demand was estimated based on: a) the historical groundwater geochemical data and water quality parameters (showing the anaerobic nature of the subsurface and likelihood of reduced iron and manganese exerting a demand on persulfate), b) the stoichiometric demand based on the historical COC concentrations, and c) professional judgment from numerous persulfate applications. Due to the very low COC concentrations and lack of NAPL at AOC I, the stoichiometric demand, as is common, was negligible.”

6. Monitoring wells in the vicinity of the injection wells (i.e., MW-01, MW-06, MW-08, and MW-09) were not sampled during and after the pilot study injections in 2010, 2011 or 2012. Specifically, downgradient well MW-06 was evaluated in 2004, 2006, and 2008; downgradient wells MW-08 and MW-09 were evaluated in 2006 and 2008; and, upgradient well MW-01 was evaluated in 2004, 2006, and 2008. Clarify how contaminant migration, water geochemistry, and rebound were assessed when the other onsite wells were not evaluated during and after the pilot study injections.

**Navy Response:**

The following sentence has been added at the end of the first paragraph under Section 2:

“The Vieques Technical Subcommittee, comprising representatives of the Navy, USEPA, and EQB, concurred on the wells to include in the Pilot Study based on historical data and Pilot Study objectives. Wells MW-01, MW-06, MW-08, and MW-09 were excluded from contaminant analysis during the Pilot Study because they were either upgradient of (MW-01) or far downgradient from (MW-06, MW-08, and MW-09) the area of contamination. These wells had been installed during the RI for the purposes of nature and extent determination but were not relevant to the Pilot Study. Due to the small size of the groundwater plume and slow groundwater velocity rates (3 to 16 ft/yr), MW-02, MW-03, MW-04, MW-05, and MW-07 were determined by the Technical Subcommittee as the appropriate wells to be used for monitoring contaminant concentrations during the Pilot Study.”

Note that Section 2.7 states that to ensure contaminant rebound did not occur, the Technical Subcommittee agreed to perform two additional sampling events for a subset of the AOC I monitoring wells (i.e., MW-04, MW-05, and MW-07) and that the agreement was reached in the February 22, 2012 Technical Subcommittee meeting. Please also note the correspondence from USEPA in Appendix C stating which wells should be monitored for the two additional rounds used for potential rebound monitoring.

7. Section 2.4 (First Post-injection Performance Monitoring Event) indicates that, “At the concentrations observed at this site and given the water geochemistry, it does not appear to make a difference for VOC [volatile organic compounds] groundwater results how or if the samples are preserved;” however, the Pilot Study does not include information or a discussion to substantiate that the samples were not impacted by the persulfate or the ascorbic acid. Provide information and a discussion to substantiate that the samples were not impacted by the persulfate or the ascorbic acid.

**Navy Response:**

It is unclear what the commenter means by providing information to substantiate the samples were not impacted by ascorbic acid. The purpose of adding ascorbic acid is to sequester any residual persulfate that could oxidize contaminants in the sample between the time it is collected and
analysis in the laboratory. Therefore, ascorbic acid does not impact the sample; it potentially protects the sample from additional oxidation.

To provide additional clarity, Table 5 has been updated to include the preservative method associated with each sample and the second paragraph of Section 2.4 has been revised as follows:

“... (i.e., in accordance with the SAP). Table 4 shows the persulfate concentrations measured in wells at the time of sample collection. Table 5 shows the results of the three analyses (with identification of the preservative method for each) for each well. Of note is that the volatile organic compounds (VOCs) concentrations for each well were essentially the same among the samples preserved with hydrochloric acid, ascorbic acid, and unpreserved. For example, benzene concentrations in samples from well MW-07, which had a measured persulfate concentration between 14 and 21 mg/L, were 9.5 µg/L (unpreserved), 9.5 µg/L (ascorbic acid), and 9.4 µg/L (HCl). Therefore, at the concentrations observed ...”

8. A preliminary remediation goal (PRG) of 1.4 micrograms per liter (µg/L) was originally selected to represent a conservative screening value for naphthalene; however, a value of 6.1 µg/L was utilized. While Section 1.1 (Pilot Study Objectives and Goals) indicates that this value was determined to be more appropriate to use as a PRG, information is not provided and/or referenced in the Pilot Study to document that this value was approved for use. While this change does not significantly affect the outcome of the pilot study, some reporting limits, as shown in Table 5 (Analytical Results for COCs, Dissolved Iron and Manganese), would be above the lower PRG value. Revise the Pilot Study to include and/or reference information to document that the use of the higher value for naphthalene was approved.

**Navy Response:**

The following text has been added after the table of PRGs in Section 1.1:

“The 2011 Edition of the Drinking Water Standards and Health Advisories (issued by the USEPA Office of Water) indicates that the cancer classification of naphthalene is “I – inadequate information to assess carcinogenic potential.” The Lifetime Health Advisory (HA) Level of 100 µg/L for naphthalene is defined as the concentration of naphthalene in drinking water that is not expected to cause any adverse noncancerous effects for a lifetime of exposure. In the updated 2012 Edition of the Drinking Water Standards and Health Advisories, the HA Level of 100 µg/L for naphthalene is unchanged.

The Record of Decision (ROD) entries contained in the USEPA CERCLIS Public Access Database were searched for naphthalene cleanup goals in EPA Region 2. For the nine Superfund Sites where quantitative cleanup goals were available for naphthalene, goals ranged from 10 to 300 µg/L. A PRG of 10 µg/L was selected for three sites in New York, as stipulated in the NYSDEC Groundwater Standards, based on a non-carcinogenic endpoint HI of 1 with an uncertainty factor (UF) of 10 for “Group C” carcinogens to provide sufficient protection from possible carcinogenic effects. Additionally, naphthalene does not have a groundwater standard (SG) in the Puerto Rico Water Quality Standards (PRWQS).

The May 2013 USEPA Regional Screening Level (RSL) Table provides carcinogenic inhalation toxicity values for naphthalene, with a tap water RSL of 0.14 µg/L corresponding to a 1x10-6 excess lifetime cancer risk (ELCR) (or 14 µg/L corresponding to 1x10-4 ELCR). USEPA’s target range for ELCR is 1x10-4 to 1x10-6. The 2013 RSL table also identifies a tap water RSL of 6.1 µg/L for non-carcinogenic endpoints, based on an HI of 1 (for cumulative exposures via ingestion/dermal/inhalation).

Based on the above information, the HI-based PRG of 6.1 µg/L, especially considering it is within the USEPA’s acceptable ELCR range, is used as the PRG for naphthalene.”
Specific Comments

1. **Section 3, Groundwater Monitoring Results:** Based on Table 5 (Analytical Results for COCs, Dissolved Iron and Manganese), iron and manganese levels fluctuated throughout the pilot study; however, these fluctuations are not discussed in Section 3. Revise Section 3 to include a discussion of the varying levels in iron and manganese throughout the pilot study and the long-term effect it may have on AOC I.

   **Navy Response:**
   
   The following was added as the last paragraph of Section 3.1:
   
   “Dissolved iron and manganese were analyzed to confirm the presence of an oxidative environment post-injection, which would tend to decrease dissolved iron and manganese. As shown in Table 5, this is what was observed; iron and manganese concentrations declined at the injection wells (MW-02, MW-03, MW-04, and MW-07) following the ISCO injection, indicative of the desired oxidative conditions. Several wells also showed increases of these metals toward the end of the study, indicating a return to normal geochemical conditions.”

2. **Table 4, Persulfate Concentration:** The table indicates that persulfate in some wells was not measured; however, the Pilot Study does not discuss why persulfate was not measured. In addition, the Pilot Study does not discuss the decision criteria used for measuring or not measuring persulfate concentrations in the onsite wells. Revise Section 2.3 (Persulfate Monitoring) to document deviations from the proposed persulfate measurements. In addition, ensure all deviations are noted in the Pilot Study.

   **Navy Response:**
   
   The following was added as the last sentence of Section 2.3 and as a footnote in Table 4:
   
   “Persulfate monitoring was conducted in accordance with the SAP (CH2M HILL, 2010a).”
Final Responses to PREQB's Comments on the Draft In-Situ Remediation Pilot Study Report, Area of Concern I (AOC I) Former Atlantic Fleet Weapons Training Area-Vieques Former Naval Ammunition Support Detachment Vieques, Puerto Rico
March 28, 2013

PREQB has reviewed the report and provides the following minor editorial comments. Note that the substantive comments were discussed during the May 2013 ERP meeting and the Navy indicated that modifications as needed would be made in the draft final version of the report.

I. General Comments

1. Please note that it is reported that in November 2011 samples were submitted for GRO, DRO and ORO analyses, but the results were not tabulated. Since GRO, DRO and ORO are not chemicals of concern, please clarify why these analyses was performed or consider removing this information from the report.

   Navy Response:
   GRO, DRO, and ORO have been removed from all locations in the report.

II. Page-Specific Comments

1. Page 6, Section 2.5: Please correct the date the ORC socks were removed to July 2011, as per Table 1.

   Navy Response:
   Date has been changed from August 2011 to July 2011.

2. Page 7, Section 3.2: This section references Figure 16; however, there are only 15 figures. Please clarify.

   Navy Response:
   The first sentence of Section 3.2 has been edited to refer to Figure 7. The first sentence of the second paragraph has been revised to refer to Figures 7, 12, and 13. The first sentence of the third paragraph has been revised to refer to Figures 7, 14, and 15.

3. Page 7, Section 3.1:
   a. Please correct the text to state that the DO reading of 6.59 mg/L at MW-02 was from November 2010 (not November 2011).

   Navy Response:
   The date has been changed in Section 3.1 to November 2010.

   b. Please clarify that the DO readings of 11.15 and 5.44 mg/L in MW-07 are from 2011 and 2012, respectively.
**Navy Response:**

The sentence has been edited to “... 11.15 mg/L and 5.44 mg/L in 2011 and 2012, respectively, in MW-07 may be the result of localized oxidizing conditions ....”

4. **Appendix D:** For the March 2010 data validation report, please clarify why bis(2-ethylhexyl)phthalate was not qualified as a nondetect in sample MW-05 due to equipment blank contamination, as per the Region II guidelines.

**Navy Response:**

Field samples are associated with their equipment rinseate blanks by the date collected. VWAI-MW05-0310 (collected 3/18/10 12:20) contained bis(2-Ethylhexyl)phthalate at 1.4 J µg/L. The associated equipment blank, VWAI-EB01-031810 (collected 3/18/10 13:00), was nondetect for this compound.