

EIASOP-VOADIOX3
1,4-Dioxane
in water by GC/MS
Revision 3
11/30/04
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STANDARD OPERATING PROCEDURE FOR
MEASUREMENT OF PURGEABLE 1,4-DIOXANE IN WATER BY GC/MS

The Office of Environmental Measurement and Evaluation
EPA Region New England
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1.0 **Scope and Application:**

- 1.1 The procedure contained herein is applicable to all EPA Region I chemists performing 1,4-Dioxane [123-91-9] analysis by GC/MS with purge and trap.
- 1.2 This method is applicable for analysis of drinking water, ground water and surface water.

Table I - Target Compound List

<u>Analyte</u>	<u>RL, ug/L</u>
1,4-Dioxane	2.0

- 1.3 This SOP is to be used in conjunction with EIA-VOAGCMS7.SOP.

2.0 **Summary of Method:**

1,4-dioxane is purged from the sample matrix by bubbling helium through a 5-mL sample. The high solubility of 1,4-dioxane in water results in low purge efficiency. Heating the sample to 80°C prior to purging increases the purge efficiency. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and back flushed with helium to desorb the trapped sample components on to a capillary gas chromatographic (GC) column interfaced to a mass spectrometer (MS). The column is temperature programmed to separate the method analytes that are then detected with the mass spectrometer. Comparing their measured mass spectra and retention times to reference spectra and retention times in the database identifies compounds eluting from the GC columns. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. The concentration of each identified component is measured by relating the mass spectrometric response of the quantitation ion produced by that compound to the response

of the quantitation ion produced by the associated internal standard. Surrogates, whose concentrations are known in every sample, are measured with the same internal standard quantitation procedure. Removing an aliquot of the sample and diluting to the needed total volume with reagent water accomplish dilutions.

3.0 Definitions:

- 3.1 FIELD DUPLICATES (FD1 and FD2): Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.
- 3.2 Laboratory Duplicate (LD1 and LD2): Two injections from the same sample. The analyses of LD1 and LD2 give a measure of the precision associated with the laboratory procedure.
- 3.4 LABORATORY REAGENT BLANK (LRB): An aliquot of VOA free reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 3.5 STOCK STANDARD SOLUTION: A concentrated solution containing one or more method analytes (in methanol) prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.
- 3.6 WORKING STANDARD SOLUTION: A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare aqueous calibration solutions and other needed analyte solutions.
- 3.7 SECONDARY STANDARD: A standard from another vendor or a different lot number that is used to check the primary standard used for quantitation.

4.0 Health and Safety Warnings:

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses. For example, concentrated sulfuric acid presents some hazards and is moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs flush with large volumes of water.

4.2 Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

4.3 Some method analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled with suitable protection to skin, eyes, etc.

5.0 Interferences:

5.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that leads to discrete artifacts and/or elevated baselines in the chromatograms. All of these materials must routinely be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks.

5.2 Matrix interferences may be caused by contaminants that co-elute with the target compounds. The extent of matrix interferences will vary considerably from source to source.

5.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry

over, VOA free water blank should be analyzed following an unusually concentrated sample to assure that the syringe is clean.

6.0 Personnel Qualifications:

- 6.1 The analyst should have at least a four -year degree in a physical science.
- 6.2 The analyst must have a satisfactory IDC/MDL in place before analyzing samples.
- 6.3 All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function.

7.0 Equipment and Supplies:

- 7.1 ThermoFinnigan PolarisQ ion -trap MS with a ThermoFinnigan Trace 2000 GC.
- 7.2 GC column - 40 meter x 0.18 mm i.d. with a 1.0 um film thickness; Rtx-502.2 (Restek Corp.)
- 7.3 Varian Archon auto sampler with a Tekmar LSC 3100 equipped with a 5-mL fritted sparge vessel and a Tekmar Purge Trap K (VOCARB 3000).
- 7.4 Vial: 40 mL VOA vials with a Teflon lined septum cap.
- 7.5 5,10,25,50,100 and 500 uL micro syringes.
- 7.6 5mL and 100 mL volumetric flasks.

8.0 Standards and Reagents

- 8.1 Methanol - demonstrated to be free of analytes.
- 8.2 Reagent water

8.3 Standard and Secondary Stock Solutions- Solutions are prepared from pure standard materials and purchased by the Region I Chemistry Laboratory. The calibration standard is obtained from Restek, cat#30287, 1,4Dioxane Standard, and 2,000ug/mL in P&T methanol. Secondary (LCS/LFB/CCV) standard is obtained from Ultra Scientific, 1,4-Dioxane Solution, cat# NV-150, 100 ug/ml in methanol. The surrogate standard is prepared from neat compound, 1,4 -Dioxane-d₈, Cambridge Isotope Lab, cat# DLM-28.

The internal standard, 4 -BFB, is obtained from Ultra Scientific, part # STS-110N, 2,000 ug/mL in MeOH.

8.3.1 Standard Preparation and Use:

The working calibration standard is prepared by diluting 250uL of the Restek standard to 5 mL with methanol. This gives the 100 ug/mL working solution. This is used to make the calibration curve by injecting 1,5,10,20,50 and 100 uL into 100 mL of reagent water. This gives standards of 1 - 100 ppb. This aqueous standard is poured into a VOA vial for analysis.

The secondary standard is used as received. The ampule is opened and the standard is transferred to an amber 2 mL vial. This solution is used for the CCV/LCS and spiking. The CCV/LCS is prepped at 10 ppb.

The surrogate standard is prepared by diluting 10 uL of the neat compound to 5 mL in methanol. Then 250 uL of this solution is diluted a second time to 5 mL in methanol. This results in a surrogate solution of ~100 ppb. This solution is added to the same 100 mL flask as the calibration standard.

The internal standard is prepared by diluting 12.5 uL of BFB solution to 5 mL in MeOH. This results in a 5.00 ppb solution that is loaded into the Archon IS/SU vessel. The auto sampler adds 1 uL to each sample for a concentration of 1.0 ppb.

9.0 Calibration:

9.1 Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is

required routinely throughout sample analysis as dictated by results of continuing calibration checks. The relative standard deviation (RSD) of each analyte must be less than 30%. After the initial calibration is successful, a continuing calibration check is required at the beginning of each 12-hour period during which analyses are performed. Samples may also be analyzed following an initial calibration, if time permits. The analytical sequence is closed with a CCV.

9.1.1 Calibration ions:

Compound	Primary ion	Secondary ion(s)
1,4-Dioxane	88	58
1,4-Dioxane- d_8 (SS)	96	64
BFB (IS)	174	176, 95

9.2 Initial Calibration

9.2.1 Mass Spectrometer Calibration and Tuning Procedure

When necessary, calibrate the mass axis and adjust the tuning of the mass spectrometer using the procedures prescribed by the manufacturer with any modifications necessary to meet the requirements in Section 9.2.2.

9.2.2 At the beginning of each 12-hour work shift, during which analyses are performed, introduce into the GC (either by purging a laboratory reagent blank or making a syringe injection) 50 ng of BFB and acquire mass spectra for m/z 35-300 at electron energy equivalent to 70 eV. If the spectrum does not meet all criteria (see Table 1), the mass spectrometer must be retuned and adjusted to meet the EPA program required criteria before proceeding with calibration and sample analysis. An average spectrum across the BFB peak may be used to test the instrument performance. A background-subtracted spectrum must be used to evaluate the performance of the system.

Table 1. Ion Abundance Criteria For 4-Bromofluorobenzene (BFB)

m/z	Relative Abundance Criteria
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	50% to 100% of m/z 95
175	5 to 9% of m/z 174
176	95% to 101% of m/z 174
177	5 to 9% of m/z 176

- 9.2.3 When an initial calibration is required, prepare the calibration standards according to Section 8.9.
- 9.2.4 Analyze the calibration standards using the purge and trap and GC/MS parameters given in Attachment A.
- 9.2.5 Check each standard for good GC performance. Good column performance will produce symmetrical peaks with minimum tailing. If peaks are broad, or if sensitivity is poor, remedial action may be necessary.
- 9.2.6 Calculate the response factors (RF) for 1,4-dioxane and 1,4-dioxane- d_8 . This calculation is supported in the GC/MS data system software. RF is a dimensionless number, but units used to express quantities of analyte and internal standard must be equivalent.
- 9.2.6.1 Calculate the mean RF from the analyses of the calibration solutions. Calculate the standard deviation and the percent relative standard deviation from each mean. If the %RSD exceeds 30%, either analyze additional aliquots of

appropriate calibration solutions to obtain an acceptable %RSD over the entire concentration range, or take action to improve GC/MS performance.

9.2.6.2 Verify the accuracy of the initial calibration by preparing and analyzing a Laboratory Control Sample (LCS).

9.2.6.3 If time remains in the 12-hour time period after meeting the initial calibration QC acceptance criteria, blanks and samples may be analyzed.

9.3 Continuing Calibration

9.3.1 At the beginning of each 12-hour work shift during which analyses are performed, analyze BFB according to the directions in Section 9.2.2. If the spectrum does not meet all criteria the mass spectrometer must be retuned and adjusted to meet all criteria before proceeding with continuing calibration.

9.3.2 Prepare and analyze a 10.0 ppb CCV and analyze with the same GC/MS conditions used during the initial calibration.

9.3.3 Calculate the RFs for 1,4-dioxane and 1,4-dioxane- d_8 from the data measured in the continuing calibration. The RF must be within 30% of the mean value measured in the initial calibration. If these conditions do not exist, corrective action must be taken which may require a new initial calibration.

10.0 Sample Collection, Preservation and Storage

10.1 Sample Collection and Preservation

10.1.1 Samples should be received in glass vials with Teflon-lined septa without headspace. Samples

should not be acid preserved. Handling of samples should be done in accordance with all Chain of Custody procedures being observed within and outside of the laboratory.

- 10.1.2 The samples must be chilled to 4°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be maintained at 4°C on arrival at the laboratory.

10.2 Sample Storage

- 10.2.1 Samples should be preserved by storing them in a dark area at 4°C until analysis. The sample storage area must be free of organic solvent vapors.
- 10.2.2 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and replaced, unless after consultation with project management of the applicable program, it is determined that the analysis may be performed.

11.0 Sample Analysis

- 11.1 See attachment A for all operating conditions.
- 11.2 At the start of each 12-hour period during which standards or samples are to be analyzed, analyze BFB according to the directions of Section 9.2.2.
- 11.3 After achieving a tune and an appropriate calibration, proceed with sample analysis.
- 11.3.1 Run the sample analysis in the following order:

BFB tune check standard (50 ng)

CCV (10.0 PPB)
Method Blank
Field Blank (if any)
Samples
Matrix Spike/ Matrix Spike
Duplicate (when required)
Close out CCV
Load Lastrun method

11.4 Sample vials are placed in Archon auto-sampler. The auto-sampler is started after the GC/MS sequence has been downloaded.

11.5 If the sample concentration exceeds the linear range of the calibration curve sample dilution with reagent water should be carried out.

12.0 Identification and Quantitation:

12.1 Identification of Analyses

Identify a sample component by comparison of its mass spectrum (after background subtraction) to reference spectra. The GC retention time of the sample component should be within +/- 30 sec of the predicted time.

12.1.1 In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within a 20% range. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10 to 50%. Some ions, particularly the molecular ion, are of special importance, and should be evaluated even if they are below 10% relative abundance.

12.1.2 Identification requires expert judgment when sample components are not chromatographically resolved and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample

component (i.e., broadened peak with shoulder(s) between two or more maxima), appropriate analyte spectra and background spectra can be selected. When analytes co-elute (i.e., only one GC peak is apparent), the identification criteria can be met, but each analytes mass spectrum will contain extraneous ions contributed by the co-eluting compound. With the gas chromatographic conditions of this method, 1,4-dioxane and the internal standard, 1,4-dioxane- d_8 , are not fully resolved. To avoid the possibility of false negatives for 1,4-dioxane, the analyst must examine and submit mass chromatograms of m/z 88 centered at the expected retention time of 1,4-dioxane.

12.2 Calculations

12.2.1 complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations if unique ions with adequate intensities are available for quantitation.

12.2.2 Use following calculation for analyte concentration.

$$C_x = \frac{(A_x)(C_{IS})}{(A_{IS})(RF_{CC})(V_o)}$$

where:

C_x = concentration of analyte in micrograms per liter in the water sample

A_x = peak area of the quantitation ion of the analyte in the sample

A_{IS} = peak area of the quantitation ion of the internal standard in the sample

C_{IS} = total quantity (in micrograms) of internal standard added to the water sample

RF_{CC} = response factor of analyte from the calibration. The RF used is dependent upon which calibration is current, i.e. the initial or

continuing (daily). For samples analyzed immediately after an initial calibration, the RF from the Level 3 standard is used for quantitation.

V_o = Volume of water extracted (L)

- 12.2.3 Alternatively, use the GC/MS system software or other proven software to compute the concentration of analyte.
- 12.2.4 Calculations should utilize all available digits of precision, but final reported concentrations should be rounded to two significant figures.

13.0 Quality Control:

See TABLE 2 for criteria.

13.1 Initial Demonstration of Precision and Accuracy

- 13.1.1 Prepare a QC reference sample containing 1,4-dioxane at 5.0 ppb. The calibration standards and the QC standards are prepared from solutions obtained from different vendors.
- 13.1.2 Analyze the QC reference sample at least four consecutive times following the calibration and analysis procedures of Sections 9 and 11. Accept results only if routine data validation procedures are acceptable.
- 13.1.3 Tabulate all results and calculate the average recovery in micrograms per liter and in percent values, the standard deviation in $\mu\text{g/L}$, and the relative standard deviation (RSD) in percent values.
- 13.1.4 The data is acceptable if the average recovery is in

the range of 60-140%.

13.2 Determination of Method Detection Limits (MDLs)
Analyze seven replicates of a calibration standard containing 2 µg/L of 1,4-dioxane and 1,4-dioxane-d₈.

13.2.1 Calculate and tabulate all results of 1,4-dioxane and 1,4-dioxane-d₈, including the number of acceptable results (n), the average recovery in µg/L and in percent values, and the standard deviation in µg/L.

13.3 Daily Quality Control

13.3.1 The GC/MS mass and abundance scales must be calibrated and tuned using PFTBA if the BFB criteria cannot be met. On a routine basis BFB is analyzed daily to confirm the calibration and tune. No sample analysis may be initiated without an acceptable 50-ng BFB tune check, laboratory blank results, and continuing calibration standard RFs. Acceptable tune checks from BFB, RFs from continuing calibrations and blank results are required in every 12-hour analysis set. The blank analysis should demonstrate that interferences from the analytical system, glassware, and reagents are under control.

13.3.2 BFB (50 ng) is analyzed with mass spectra acquired for m/z 35-300. If the spectrum does not meet all criteria analyses cannot proceed until corrective action is taken and acceptable mass spectra is obtained.

13.3.3 No analysis may be initiated without an appropriate five-point calibration curve. Each analyte and surrogate must have an RSD less than or equal to 30%. For each analyte or surrogate with an RSD exceeding 30%, flag the analyte in all associated data.

13.3.4 A continuing calibration standard meeting criteria must be analyzed every 12-hours. If the continuing calibration standard's response factors meet criteria the analysis can continue.

13.3.4.1 Acceptance criteria for continuing calibration standard results are:

(a) Percent differences (%D) between the RF of the daily standard and the average of the five-point initial calibration curve are less than 30%. The percent difference is defined in the following equation:

$$\%D = \frac{RF_{cc} - \overline{RF}_{IC}}{\overline{RF}_{IC}} \times 100$$

where:

RF_{cc} = Response factor from continuing calibration
 \overline{RF}_{IC} = Average response factor from the most recent initial calibration meeting the technical acceptance criteria

(b) If a compound with an unacceptable %RSD or %D is detected in a sample, the analysis should be repeated, if the holding time will not be exceeded.

(c) If the %D for the RFs are deemed unacceptable for the target, sample analysis cannot be initiated, and corrective action must be taken. Either another continuing calibration standard or a five-point initial calibration curve must be analyzed.

13.3.5 A method blank is tested after the BFB check and the continuing calibration and for every 12-hour interval in which samples are analyzed. The method blank must contain the target at levels less than its RL.

13.3.6 Internal Standards

The absolute area of the internal standard's quantitation ion will be tabulated in a run sequence as part of sample analysis records. The internal standard areas from any sample may vary within the range of 50% to 200% of the corresponding areas from the day's continuing calibration standard. The internal standard retention times from any sample may vary within the range of .30 seconds of the corresponding retention times from the day's continuing calibration standard. When any internal standard response exceeds the -50% to +100% area variation or when any retention time exceeds the .30 second variation, the sample should be re-analyzed.

13.3.7 Surrogate Recoveries

Each sample, standard and blank must contain 1,4-dioxane- d_8 at 10 $\mu\text{g/L}$ as a surrogate. Acceptable recoveries are 40% - 160%. As data are accumulated, the acceptable recovery range may be revised as appropriate. Surrogate percent recoveries will be reported with each sample.

13.3.8 Matrix Spike/Matrix Spike Duplicate

With each project a matrix spike and matrix spike duplicate will be analyzed. The spiked samples must be carried through all stages of the sample preparation and analysis steps. Recoveries must be within the range of 60% to 140%. The samples are spiked at 10 ppb. As data are accumulated, the acceptable recovery range may be revised as appropriate.

13.3.9 Laboratory Control Sample

A Laboratory Control Sample (LFB/LCS/CCV) is prepared at 10.0 ppb. Recoveries of 1,4-dioxane must be in the range of 60% to 140%. As data are accumulated, the acceptable recovery range may be revised as appropriate. The spike solution for this sample is made up from a different source than the standards

used for the initial calibration.

13.3.10 Reporting level

The reporting level of 2.0 ppb is based on a factor of 3x the calculated MDL. Due to the relative low response of this compound any positive value between 2.0 and 5.0 ppb will have the signal to noise ratio determined. If the signal to noise ratio is found to be less than 3, the reported value will be qualified with a J as estimated.

14.0 Data and Records Management:

14.1 All work performed for the analyses of samples will be entered into the appropriate logbook.

15.0 Waste Management and Pollution Prevention

NERL encourages all chemist and biologists to investigate micro-analytical techniques, innovative technologies, and chemical substitution in laboratory processes to reduce waste and prevent pollution. As analytical SOPs are reviewed, on an annual basis, the responsible chemist or biologist will incorporate waste minimization practices where practicable and where these practices have been demonstrated to return data of equivalent quality.

Chemists and biologists must refer to the Waste Management Program SOP for proper disposal of laboratory waste. Personnel should contact the

Environmental, Safety and Health Department if changes in the analytical SOP will generate new waste streams. Questions regarding the proper disposal of laboratory waste and purchase of new reagents should be directed to the Environmental, Safety and Health Department in advance of actually initiating a change in the analytical method.

15. **References:**

1. "Standard Operating Procedure for Measurement of Purgeable 1,4-Dioxane and THF in Water By Wide-Bore Capillary Column Gas Chromatography/Mass Spectrometry,@ CRL SOP GCMS023, Rev. 2, Jan 9, 2002, USEPA, Region V, Chicago, Illinois.
2. "Method 524.2, Rev. 3.0, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry", 1989, USEPA, EMSL, Cincinnati, Ohio.
3. Federal Register, 40 CFR Part 136 Appendix B, Friday, October 26, 1984, Vol. 49 No. 209, p. 149-150 and 198-199.
4. Incos 50 GC/MS Operation for Volatile Organic Compounds@ EIA-VOAGCMS7.SOP,04/29/02, Office of Environmental Measurement and Evaluation(OEME) U.S. Environmental Protection Agency, Region I.

ATTACHMENT A

PURGE AND TRAP CONDITIONS
LSC 3100 Method 2

Trap: Tekmar Purge Trap K (VOCARB 3000).
Purge Flow = 30 mL/min of helium
Line, Valve Temperature of Tekmar 2000 concentrator = 100°C
Purge Ready (Standby) Temp = 35°C
Sample Heater = ON
Sample Preheat Time = 2 min
Sample Preheat Temp = 80°C
Purge Time = 11 min
Dry Purge Time = 2.0 min
Desorb Preheat Time = 245°C
Desorb Time = 4 min
Desorb Temp = 250°C
Sample Drain = ON
Bake Time = 15 min
Bake Temp = 260°C
Bake Gas Bypass = OFF
Bake Gas Bypass Delay = 0.0 min

Archon Method

Sample Volume: 5mL
Rinse Volume: 5 mL
#Rinses: 3
Standard: 1 (Load IS/SU into vial #1, left side)
Syringe Flushes: 3

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GC/MS CONDITIONS
p-dioxane Method

OVEN: Initial oven temperature of 40 °C for 5 minutes; a
rate of 10 °C/min to a final temperature of 220 °C
for 3 min. Run time = 26.0 min.
INLET: Injection port temperature = 180°C.
COLUMN: 40 meter x 0.18 mm i.d. with a 1.0 um film
thickness; Rtx-502.2 (Restek Corp.)
MS transfer line temperature = 220°C.

MS Method:

Acquisition Time: GC Run time
Cal Gas: Off
Reagent Gas: Off
Acquire Profile: No
Acq Threshold: 0
Source Temp: 200 C

Segment 1
Start Time: 3.00 minutes
Polarity: POS
Mult Offset: + 100 volts
Tune File: enviromentalbfb
Reagent gas flow: Off

Scan Event 1
Micro Scans: 2
Max Ion Time: 25
Mass Defect: 0.00
Scan Mode: Segmented scan
Contiguous Ranges: Yes
Custom Inj RF: Yes
Custom Gate Lens: No
Single AGC prescan: No
First Mass: 35 Last: 70 Inj RF: 25.00 Inj Scale: 220
First Mass: 71 Last: 120 Inj RF: 25.00 Inj Scale: 110
First Mass: 121 Last: 300 Inj RF: 29.00 Inj Scale: 100

TABLE 2: QC Acceptance criteria

AUDIT	FREQUENCY	LIMIT	CORRECTIVE ACTION
BFB	Before analysis and every 12 hrs.	See sec 9.2.2	Repeat BFB Tune MS Clean source
Initial Cal	When cont cal fails or as method requires	%RSD </+ 30., Sec 9.2	Eval chromatogram and integration Re- Calibrate
Method Blk	Before analysis and every 12 hours	Free of all target analytes > RL. sec 13.3.5	Check blank source Check reagent source Bake trap Clean purge vessel Rinse syringes
Trip Blk	One/set of samples		Check storage blk Contact project officer
Lab Fortified Blk (LCS)	Before analysis and every 12 hrs.	$\pm 40\%$, sec 13.3.9	Check peak integration Test for leaks Repeat
Matrix Spikes	One MS/MSD for every project up to 20 samples	60 - 140 % sec 13.3.8	Areas of internal std Explain in report narrative
Internal Std	Every sample including std, blk and QC samples	-50% - 200%, sec 13.3.6	Re-run the sample Check the solution

Surrogate Recoveries	Every sample including std, blk, and QC sample	60-140% sec 13.3.7	Eval integration Quality the report
Low level positive results	With positive hits between 2.0 and 5.0 ppb	signal to noise of >3 sec 13.3.10	Qualify data with a J

Analyst's Agreement Form

I have read and understand the revision of the Standard Operating Procedure listed above and agree to follow the procedures as written. If specific SOP requirements can not be met due to uncontrollable factors or if the client requests a departure from the SOP, the policy for departures from SOPs outlined in the current laboratory QA Plan will be adhered to.

Dan Boudreau
PRINT NAME


SIGNATURE

11/30/04
DATE

Joseph Montanaro
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DATE

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