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Innovative Technology Verification Report

XRF Technologies for Measuring Trace Elements in Soil and Sediment

Oxford X-Met 3000TX XRF Analyzer

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Oxford X-Met 3000TX XRF Analyzer

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Notice

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Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's natural resources. Under the mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's Office of Research and Development (ORD) provides data and scientific support that can be used to solve environmental problems, build the scientific knowledge base needed to manage ecological resources wisely, understand how pollutants affect public health, and prevent or reduce environmental risks.

The National Exposure Research Laboratory is the Agency's center for investigation of technical and management approaches for identifying and quantifying risks to human health and the environment. Goals of the laboratory's research program are to (1) develop and evaluate methods and technologies for characterizing and monitoring air, soil, and water; (2) support regulatory and policy decisions; and (3) provide the scientific support needed to ensure effective implementation of environmental regulations and strategies.

EPA's Superfund Innovative Technology Evaluation (SITE) Program evaluates technologies designed for characterization and remediation of contaminated Superfund and Resource Conservation and Recovery Act (RCRA) sites. The SITE Program was created to provide reliable cost and performance data to speed acceptance and use of innovative remediation, characterization, and monitoring technologies by the regulatory and user community.

Effective monitoring and measurement technologies are needed to assess the degree of contamination at a site, provide data that can be used to determine the risk to public health or the environment, and monitor the success or failure of a remediation process. One component of the EPA SITE Program, the Monitoring and Measurement Technology (MMT) Program, demonstrates and evaluates innovative technologies to meet these needs.

Candidate technologies can originate within the federal government or the private sector. Through the SITE Program, developers are given an opportunity to conduct a rigorous demonstration of their technologies under actual field conditions. By completing the demonstration and distributing the results, the Agency establishes a baseline for acceptance and use of these technologies. The MMT Program is managed by ORD's Environmental Sciences Division in Las Vegas, Nevada.

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Abstract

The Oxford Instruments Analytical, Ltd., (Oxford) X-Met 3000 TX (X-Met) XRF Services x-ray fluorescence (XRF) analyzer was demonstrated under the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) Program. The field portion of the demonstration was conducted in January 2005 at the Kennedy Athletic, Recreational and Social Park (KARS) at Kennedy Space Center on Merritt Island, Florida. The demonstration was designed to collect reliable performance and cost data for the X-Met analyzer and seven other commercially available XRF instruments for measuring trace elements in soil and sediment. The performance and cost data were evaluated to document the relative performance of each XRF instrument.

This innovative technology verification report describes the objectives and the results of that evaluation and serves to verify the performance and cost of the X-Met analyzer. Separate reports have been prepared for the other XRF instruments that were evaluated as part of the demonstration.

The objectives of the evaluation included determining each XRF instrument's accuracy, precision, sample throughput, and tendency for matrix effects. To fulfill these objectives, the field demonstration incorporated the analysis of 326 prepared samples of soil and sediment that contained 13 target elements. The prepared samples included blends of environmental samples from nine different sample collection sites as well as spiked samples with certified element concentrations. Accuracy was assessed by comparing the XRF instrument's results with data generated by a fixed laboratory (the reference laboratory). The reference laboratory performed element analysis using acid digestion and inductively coupled plasma – atomic emission spectrometry (ICP-AES), in accordance with EPA Method 3050B/6010B, and using cold vapor atomic absorption (CVAA) spectroscopy for mercury only, in accordance with EPA Method 7471A.

The X-Met portable XRF analyzer features a miniature, rugged x-ray tube excitation source for analyzing a wide variety of elements and sample materials, including alloys, environmental solids, and other analytical samples. Other features of the X-Met include: multiple x-ray beam filters, multiple calibration methods, and adjustable tube voltages and currents.

The analyzer weighs 4.5 pounds and can be powered in the field with a lithium-ion battery or 110-volt alternating current (AC). The X-Met XRF analyzer utilizes a Hewlett-Packard (HP) iPAQ personal data assistant (PDA) for data storage of up to 10,000 tests with spectra in its 64 megabyte memory. The iPAQ has a color, high resolution display with variable backlighting and can be fitted with Bluetooth® wireless printing and data downloading, an integrated bar-code reader, and wireless data and file transfer accessories. The X-Met analyzer can analyze elements from potassium to uranium in suites of 25 elements simultaneously.

This report describes the results of the evaluation of the X-Met analyzer based on the data obtained during the demonstration. The method detection limits, accuracy, and precision of the instrument for each of the 13 target analytes are presented and discussed. The cost of element analysis using the X-Met analyzer is compiled and compared to both fixed laboratory costs and average XRF instrument costs.

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Acronyms, Abbreviations, and Symbols

µg	Micrograms
µA	Micro-amps
AC	Alternating current
ADC	Analog to digital converter
Ag	Silver
Am	Americium
ARDL	Applied Research and Development Laboratory, Inc.
As	Arsenic
ASARCO	American Smelting and Refining Company
BN	Burlington Northern
C	Celsius
Cd	Cadmium
CFR	Code of Federal Regulations
cps	Counts per second
CPU	Central processing unit
Cr	Chromium
CSV	Comma-separated value
Cu	Copper
CVAA	Cold vapor atomic absorption
EDXRF	Energy dispersive XRF
EDD	Electronic data deliverable
EPA	U.S. Environmental Protection Agency
ERA	Environmental Research Associates
ESA	Environmental site assessment
ESD	Environmental Sciences Division
ETV	Environmental Technology Verification (Program)
eV	Electron volts
Fe	Iron
FPT	Fundamental Parameters Technique
FWHM	Full width of peak at half maximum height
GB	Gigabyte
Hg	Mercury
Hz	Hertz

Acronyms, Abbreviations, and Symbols (Continued)

ICP-AES	Inductively coupled plasma-atomic emission spectrometry
ICP-MS	Inductively coupled plasma-mass spectrometry
IR	Infrared
ITVR	Innovative Technology Verification Report
KARS	Kennedy Athletic, Recreational and Social (Park)
keV	Kiloelectron volts
kg	Kilograms
KSC	Kennedy Space Center
kV	Kilovolts
LEAP	Light Element Analysis Program
LiF	Lithium fluoride
LIMS	Laboratory information management system
LOD	Limit of detection
mA	Milli-amps
MB	Megabyte
MBq	Mega Becquerels
MCA	Multi-channel analyzer
mCi	Millicuries
MDL	Method detection limit
mg/kg	Milligrams per kilogram
MHz	Megahertz
mm	Millimeters
MMT	Monitoring and Measurement Technology (Program)
Mo	Molybdenum
MS	Matrix spike
MSD	Matrix spike duplicate
NASA	National Aeronautics and Space Administration
NELAC	National Environmental Laboratory Accreditation Conference
NERL	National Exposure Research Laboratory
Ni	Nickel
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute for Standards and Technology
NRC	Nuclear Regulatory Commission
NSWC	Naval Surface Warfare Center
ORD	Office of Research and Development
OSWER	Office of Solid Waste and Emergency Response

Acronyms, Abbreviations, and Symbols (Continued)

P	Phosphorus
Pb	Lead
PC	Personal computer
PDA	Personal digital assistant
PCB	Polychlorinated biphenyls
Pd	Palladium
PE	Performance evaluation
PeT	Pentaerythritol
ppb	Parts per billion
ppm	Parts per million
Pu	Plutonium
QA	Quality assurance
QAPP	Quality assurance project plan
QC	Quality control
r^2	Correlation coefficient
RCRA	Resource Conservation and Recovery Act
Rh	Rhodium
RPD	Relative percent difference
RSD	Relative standard deviation
%RSD	Percent relative standard deviation
SAP	Sampling and analysis plan
SBMM	Sulphur Bank Mercury Mine
Sb	Antimony
Se	Selenium
Si	Silicon
SITE	Superfund Innovative Technology Evaluation
SOP	Standard operating procedure
SRM	Standard reference material
SVOC	Semivolatile organic compound
TAP	Thallium acid phthalate
Tetra Tech	Tetra Tech EM Inc.
Ti	Titanium
TSA	Technical systems audit
TSP	Total suspended particulates
TXRF	Total reflection x-ray fluorescence spectroscopy
U	Uranium
USFWS	U.S. Fish and Wildlife Service

Acronyms, Abbreviations, and Symbols (Continued)

V	Vanadium
V	Volts
VOC	Volatile organic compound
W	Watts
WDXRF	Wavelength-dispersive XRF
WRS	Wilcoxon Rank Sum
XRF	X-ray fluorescence
Zn	Zinc

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Chapter 1
Introduction

The U.S. Environmental Protection Agency (EPA), Office of Research and Development (ORD) conducted a demonstration to evaluate the performance of innovative x-ray fluorescence (XRF) technologies for measuring trace elements in soil and sediment. The demonstration was conducted as part of the EPA Superfund Innovative Technology Evaluation (SITE) Program.

Eight field-portable XRF instruments, which were provided and operated by six XRF technology developers, were evaluated as part of the demonstration. Each of these technology developers and their instruments are listed in Table 1-1. The technology developers brought each of these instruments to the demonstration site during the field portion of the demonstration. The instruments were used to analyze a total of 326 prepared soil and sediment samples that contained 13 target elements. The same sample set was analyzed by a fixed laboratory (the reference laboratory) using established EPA reference methods. The results obtained using each XRF instrument in the field were compared with the results obtained by the reference laboratory to assess instrument accuracy. The results of replicate sample analysis were utilized to assess the precision and the detection limits that each XRF instrument could achieve. The results of these evaluations, as well as technical observations and cost information, were then documented in an Innovative Technology Verification Report (ITVR) for each instrument.

This ITVR documents EPA's evaluation of the Oxford X-Met 3000 TX XRF analyzer based on the results of the demonstration.

1.1 Organization of this Report

This report is organized to first present general information pertinent to the demonstration. This information is common to all eight ITVRs that were developed from the XRF demonstration. Specifically, this information includes an introduction (Chapter 1), the locations where the field samples were collected (Chapter 2), the field demonstration (Chapter 3), the evaluation design (Chapter 4), and the reference laboratory results (Chapter 5).

The second part of this report provides information relevant to the specific instrument that is the subject of this ITVR. This information includes a description of the instrument (Chapter 6), a performance evaluation (Chapter 7), a cost analysis (Chapter 8), and a summary of the demonstration results (Chapter 9).

Table 1-1. Participating Technology Developers and Instruments

Developer Full Name	Distributor in the United States	Developer Short Name	Instrument Full Name	Instrument Short Name
Elvatech, Ltd.	Xcalibur XRF Services	Xcalibur	ElvaX	ElvaX
Innov-X Systems	Innov-X Systems	Innov-X	XT400 Series	XT400
NITON Analyzers, A Division of Thermo Electron Corporation	NITON Analyzers, A Division of Thermo Electron Corporation	Niton	XLi 700 Series XLt 700 Series	XLi XLt
Oxford Instruments Analytical, Ltd.	Oxford Instruments Analytical, Ltd.	Oxford	X-Met 3000 TX ED2000	X-Met ED2000
Rigaku, Inc.	Rigaku, Inc.	Rigaku	ZSX Mini II	ZSX Mini II
RÖNTEC AG (acquired by Bruker AXS, 11/2005	RÖNTEC USA	Rontec	PicoTAX	PicoTAX

References are provided in Chapter 10. A verification statement for the instrument is provided as Appendix A. Comments from the instrument developer on the demonstration and any exceptions to EPA's evaluation are presented in Appendix B. Appendices C, D, and E contain the data validation summary report for the reference laboratory data and detailed evaluations of instrument versus reference laboratory results.

1.2 Description of the SITE Program

Performance verification of innovative environmental technologies is an integral part of EPA's regulatory and research mission. The SITE Program was established by the EPA Office of Solid Waste and Emergency Response and ORD under the Superfund Amendments and Reauthorization Act of 1986. The overall goal of the SITE Program is to conduct performance verification studies and to promote acceptance of innovative technologies that may be used to achieve long-term protection of human health and the environment. The program is designed to meet three primary objectives: (1) identify and remove obstacles to development and commercial use of innovative technologies; (2) demonstrate promising innovative technologies and gather reliable information on performance and cost to support site characterization and cleanup; and (3) maintain an outreach program to operate existing technologies and identify new opportunities for their use. Additional information on the SITE Program is available on the EPA ORD web site (www.epa.gov/ord/SITE).

The intent of a SITE demonstration is to obtain representative, high-quality data on the performance and cost of one or more innovative technologies so that potential users can assess a technology's suitability for a specific application. The SITE Program includes the following program elements:

- Monitoring and Measurement Technology (MMT) Program – Evaluates technologies that sample, detect, monitor, or measure hazardous and toxic substances. These technologies are expected to provide better, faster, or more cost-effective methods for producing real-time data during site characterization and remediation studies than can conventional technologies.

- Remediation Technology Program – Demonstrates innovative treatment technologies to provide reliable data on performance, cost, and applicability for site cleanups.

- Technology Transfer Program – Provides and disseminates technical information in the form of updates, brochures, and other publications that promote the SITE Program and the participating technologies.

The demonstration of XRF instruments was conducted as part of the MMT Program, which is administered by the Environmental Sciences Division (ESD) of the National Exposure Research Laboratory (NERL) in Las Vegas, Nevada. Additional information on the NERL ESD is available on the EPA web site (www.epa.gov/nerlesd1/). Tetra Tech EM Inc. (Tetra Tech), an EPA contractor, provided comprehensive technical support to the demonstration.

1.3 Scope of the Demonstration

Conventional analytical methods for measuring the concentrations of inorganic elements in soil and sediment are time-consuming and costly. For this reason, field-portable XRF instruments have been proposed as an alternative approach, particularly where rapid and cost-effective assessment of a site is a goal. The use of a field XRF instrument for elemental analysis allows field personnel to quickly assess the extent of contamination by target elements at a site. Furthermore, the near instantaneous data provided by field-portable XRF instruments can be used to quickly identify areas where there may be increased risks and allow development of a more focused and cost-effective sampling strategy for conventional laboratory analysis.

EPA-sponsored demonstrations of XRF technologies have been under way for more than a decade. The first SITE MMT demonstration of XRF occurred in 1995, when six instruments were evaluated for their ability to analyze 10 target elements. The results of this demonstration were published in individual reports for each instrument (EPA 1996a, 1996b, 1998a, 1998b, 1998c, and 1998d). In 2003, two XRF instruments were included in a demonstration of field methods for analysis of mercury in soil and sediment.

Individual ITVRs were also prepared for each of these two instruments (EPA 2004a, 2004b).

Although XRF spectrometry is now considered a mature technology for elemental analysis, field-portable XRF instruments have evolved considerably over the past 10 years, and many of the instruments that were evaluated in the original demonstration are no longer manufactured. Advances in electronics and data processing, coupled with new x-ray tube source technology, have produced substantial improvements in the precision and speed of XRF analysis. The current demonstration of XRF instruments was intended to evaluate these new technologies, with an expanded set of target elements, to provide information to potential users on current state-of-the-art instrumentation and its associated capabilities.

During the demonstration, performance data regarding each field-portable XRF instrument were collected through analysis of a sample set that included a broad range of soil/sediment types and target element concentrations. To develop this sample set, soil and sediment samples that contain the target elements of concern were collected in bulk quantities at nine sites from across the U.S. These bulk samples of soil and sediment were homogenized, characterized, and packaged into demonstration samples for the evaluation. Some of the batches of soil and sediment were spiked with selected target elements to ensure that representative concentration ranges were included for all target elements and that the sample design was robust. Replicate samples of the material in each batch were included in the final set of demonstration samples to assess instrument precision and detection limits. The final demonstration sample set therefore included 326 samples.

Each developer analyzed all 326 samples during the field demonstration using its XRF instrument and in accordance with its standard operating procedure. The field demonstration was conducted during the week of January 24, 2005, at the Kennedy Athletic, Recreational and Social (KARS) Park, which is part of the Kennedy Space Center on Merritt Island, Florida. Observers were assigned to each XRF instrument during the field demonstration to collect detailed information on the instrument and operating procedures, including sample processing times, for subsequent evaluation. The reference laboratory also analyzed a complete set of the demonstration samples for the target elements using acid digestion and inductively coupled plasma-atomic emission spectrometry (ICP-AES), in accordance with EPA Method 3050B/6010B, and using cold vapor atomic absorption (CVAA) spectroscopy (for mercury only) in accordance with EPA Method 7471A. By assuming that the results from the reference laboratory were essentially “true” values, instrument accuracy was assessed by comparing the results obtained using the XRF instrument with the results from the reference laboratory. The data obtained using the XRF instrument were also assessed in other ways, in accordance with the objectives of the demonstration, to provide information on instrument precision, detection limits, and interferences.

1.4 General Description of XRF Technology

XRF spectroscopy is an analytical technique that exposes a solid sample to an x-ray source. The x-rays from the source have the appropriate excitation energy that causes elements in the sample to emit characteristic x-rays. A qualitative elemental analysis is possible from the characteristic energy, or wavelength, of the fluorescent x-rays emitted. A quantitative elemental analysis is possible by counting the number (intensity) of x-rays at a given wavelength.

Three electron shells are generally involved in emissions of x-rays during XRF analysis of samples: the K, L, and M shells. Multiple-intensity peaks are generated from the K, L, or M shell electrons in a typical emission pattern, also called an emission spectrum, for a given element. Most XRF analysis focuses on the x-ray emissions from the K and L shells because they are the most energetic lines. K lines are typically used for elements with atomic numbers from 11 to 46 (sodium to palladium), and L lines are used for elements above atomic number 47 (silver). M-shell emissions are measurable only for metals with an atomic number greater than 57 (lanthanum).

As illustrated in Figure 1-1, characteristic radiation arises when the energy from the x-ray source exceeds the absorption edge energy of inner-shell electrons, ejecting one or more electrons. The vacancies are filled by electrons that cascade in from the outer shells. The energy states of the electrons in the outer shells are higher than those of the inner-shell electrons, and the outer-shell electrons emit energy in the form of x-rays as they cascade down. The energy of this x-ray radiation is unique for each element.

An XRF analyzer consists of three major components: (1) a source that generates x-rays (a radioisotope or x-ray tube); (2) a detector that converts x-rays emitted from the sample into measurable electronic signals; and (3) a data processing unit that records the emission or fluorescence energy signals and calculates the elemental concentrations in the sample.

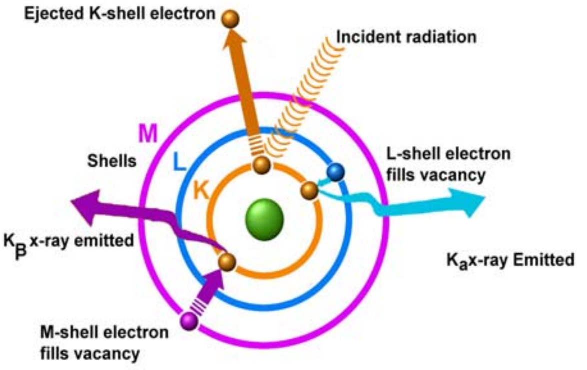

Figure 1-1. The XRF process.

Measurement times vary (typically ranging from 30 to 600 seconds), based primarily on data quality objectives. Shorter analytical measurement times (30 seconds) are generally used for initial screening, element identification, and hot-spot delineation, while longer measurement times (300 seconds or more) are typically used to meet higher goals for precision and accuracy. The length of the measuring time will also affect the detection limit; generally, the longer the measuring time, the lower the detection limit. However, detection limits for individual elements may be increased because of sample heterogeneity or the presence of other elements in the sample that fluoresce with similar x-ray energies.

The main variables that affect precision and accuracy for XRF analysis are:

1. Physical matrix effects (variations in the physical character of the sample).
2. Chemical matrix effects (absorption and enhancement phenomena) and Spectral interferences (peak overlaps).
3. Moisture content above 10 percent, which affects x-ray transmission.

Because of these variables, it is important that each field XRF characterization effort be guided by a well-considered sampling and analysis plan. Sample preparation and homogenization, instrument calibration, and laboratory confirmation analysis are all important aspects of an XRF sampling and analysis plan. EPA SW-846 Method 6200 provides additional guidance on sampling and analytical methodology for XRF analysis.

1.5 Properties of the Target Elements

This section describes the target elements selected for the technology demonstration and the typical characteristics of each. Key criteria used in selecting the target elements included:

- The frequency that the element is determined in environmental applications of XRF instruments.
- The extent that the element poses an environmental consequence, such as a potential risk to human or environmental receptors.
- The ability of XRF technology to achieve detection limits below typical remediation goals and risk assessment criteria.
- The extent that the element may interfere with the analysis of other target elements.

In considering these criteria, the critical target elements selected for this study were antimony, arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, vanadium, and zinc. These 13 target elements are of significant concern for site cleanups and human health risk assessments because most are highly toxic or interfere with the analysis of other elements. The demonstration therefore focused on the analysis of these 13 elements in evaluating the various XRF instruments.

1.5.1 *Antimony*

Naturally occurring antimony in surface soils is typically found at less than 1 to 4 milligrams per kilogram (mg/kg). Antimony is mobile in the environment and is bioavailable for uptake by plants; concentrations greater than 5 mg/kg are potentially phytotoxic, and concentrations above 31 mg/kg in soil may be hazardous to humans. Antimony may be found along with arsenic in mine wastes, at shooting ranges, and at industrial facilities. Typical detection limits for field-portable XRF instruments range from 10 to 40 mg/kg. Antimony is typically analyzed with success by ICP-AES; however, recovery of antimony in soil matrix spikes is often below quality control (QC) limits (50 percent or less) as a result of loss through volatilization during acid digestion. Therefore, results using ICP-AES may be lower than are obtained by XRF.

1.5.2 *Arsenic*

Naturally occurring arsenic in surface soils typically ranges from 1 to 50 mg/kg; concentrations above 10 mg/kg are potentially phytotoxic. Concentrations of arsenic greater than 0.39 mg/kg may cause carcinogenic effects in humans, and concentrations above 22 mg/kg may result in adverse noncarcinogenic effects. Typical detection limits for field-portable XRF instruments range from 10 to 20 mg/kg arsenic. Elevated concentrations of arsenic are associated with mine wastes and industrial facilities. Arsenic is successfully analyzed by ICP-AES; however, spectral interferences between peaks for arsenic and lead can affect detection limits and accuracy in XRF analysis when the ratio of lead to arsenic is 10 to 1 or more. Risk-based screening levels and soil screening levels for arsenic may be lower than the detection limits of field-portable XRF instruments.

1.5.3 *Cadmium*

Naturally occurring cadmium in surface soils typically ranges from 0.6 to 1.1 mg/kg; concentrations greater than 4 mg/kg are potentially phytotoxic. Concentrations of cadmium that exceed 37 mg/kg may result in adverse effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 50 mg/kg. Elevated concentrations of cadmium are associated with mine wastes and industrial facilities. Cadmium is successfully analyzed by both ICP-AES and field-portable XRF; however, action levels for cadmium may be lower than the detection limits of field-portable XRF instruments.

1.5.4 *Chromium*

Naturally occurring chromium in surface soils typically ranges from 1 to 1,000 mg/kg; concentrations greater than 1 mg/kg are potentially phytotoxic, although specific phytotoxicity levels for naturally occurring chromium have not been documented. The variable oxidation states of chromium affect its behavior and toxicity. Concentrations of hexavalent chromium above 30 mg/kg and of trivalent chromium above 10,000 mg/kg may cause adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 50 mg/kg. Hexavalent chromium is typically associated with metal plating or other industrial facilities. Trivalent chromium may be found in mine waste and at industrial facilities. Neither ICP-AES nor field-portable XRF can distinguish between oxidation states for chromium (or any other element).

1.5.5 *Copper*

Naturally occurring copper in surface soils typically ranges from 2 to 100 mg/kg; concentrations greater than 100 mg/kg are potentially phytotoxic. Concentrations greater than 3,100 mg/kg may result in adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 50 mg/kg. Copper is mobile and is a common contaminant in soil and sediments. Elevated concentrations of copper are associated with mine wastes and industrial facilities. Copper is successfully analyzed by ICP-AES and XRF; however, spectral interferences between peaks for copper and zinc may affect the detection limits and accuracy of the XRF analysis.

1.5.6 *Iron*

Although iron is not considered an element that poses a significant environmental consequence, it interferes with measurement of other elements and was therefore included in the study. Furthermore, iron is often used as a target reference element in XRF analysis.

Naturally occurring iron in surface soils typically ranges from 7,000 to 550,000 mg/kg, with the iron content originating primarily from parent rock. Typical detection limits for field-portable XRF instruments are in the range of 10 to 60 mg/kg. Iron is easily analyzed by both ICP-AES and XRF; however, neither technique can distinguish among iron species in soil. Although iron in soil may pose few environmental consequences, high levels of iron may interfere with analyses of other elements in both techniques (ICP-AES and XRF). Spectral interference from iron is mitigated in ICP-AES analysis by applying inter-element correction factors, as required by the analytical method. Differences in analytical results between ICP-AES and XRF for other target elements are expected when concentrations of iron are high in the soil matrix.

1.5.7 Lead

Naturally occurring lead in surface soils typically ranges from 2 to 200 mg/kg; concentrations greater than 50 mg/kg are potentially phytotoxic. Concentrations greater than 400 mg/kg may result in adverse effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 20 mg/kg. Lead is a common contaminant at many sites, and human and environmental exposure can occur through many routes. Lead is frequently found in mine waste, at lead-acid battery recycling facilities, at oil refineries, and in lead-based paint. Lead is successfully analyzed by ICP-AES and XRF; however, spectral interferences between peaks for lead and arsenic in XRF analysis can affect detection limits and accuracy when the ratio of arsenic to lead is 10 to 1 or more. Differences between ICP-AES and XRF results are expected in the presence of high concentrations of arsenic, especially when the ratio of lead to arsenic is low.

1.5.8 Mercury

Naturally occurring mercury in surface soils typically ranges from 0.01 to 0.3 mg/kg; concentrations greater than 0.3 mg/kg are potentially phytotoxic. Concentrations of mercury greater than 23 mg/kg and concentrations of methyl mercury above 6.1 mg/kg may result in adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 20 mg/kg. Elevated concentrations of mercury are associated with amalgamation of gold and with mine waste and industrial facilities. Native surface soils are commonly enriched by anthropogenic sources of mercury. Anthropogenic sources include coal-fired power plants and metal smelters. Mercury is too volatile to withstand both the vigorous digestion and extreme temperature involved with ICP-AES analysis; therefore, the EPA-approved technique for laboratory analysis of mercury is CVAA spectroscopy. Mercury is successfully measured by XRF, but differences between results obtained by CVAA and XRF are expected when mercury levels are high.

1.5.9 Nickel

Naturally occurring nickel in surface soils typically ranges from 5 to 500 mg/kg; a concentration of 30 mg/kg is potentially phytotoxic. Concentrations greater than 1,600 mg/kg may result in adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 60 mg/kg. Elevated concentrations of nickel are associated with mine wastes and industrial facilities. Nickel is a common environmental contaminant at metal processing sites. It is successfully analyzed by both ICP-AES and XRF with little interference; therefore, a strong correlation between the methods is expected.

1.5.10 Selenium

Naturally occurring selenium in surface soils typically ranges from 0.1 to 2 mg/kg; concentrations greater than 1 mg/kg are potentially phytotoxic. Its toxicities are well documented for plants and livestock; however, it is also considered a trace nutrient. Concentrations above 390 mg/kg may result in adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 20 mg/kg. Most selenium is associated with sulfur or sulfide minerals, where concentrations can exceed 200 mg/kg. Selenium can be measured by both ICP-AES and XRF; however, detection limits using XRF usually exceed the ecological risk-based screening levels for soil. Analytical results for selenium using ICP-AES and XRF are expected to be comparable.

1.5.11 Silver

Naturally occurring silver in surface soils typically ranges from 0.01 to 5 mg/kg; concentrations greater than 2 mg/kg are potentially phytotoxic. In addition, concentrations that exceed 390 mg/kg may result in adverse effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 45 mg/kg. Silver is a common contaminant in mine waste, in photographic film processing wastes, and at metal processing sites. It is successfully analyzed by ICP-AES and XRF; however, recovery may be reduced in ICP-AES analysis because insoluble silver chloride may form during acid digestion. Detection limits using XRF may exceed the risk-based screening levels for silver in soil.

1.5.12 Vanadium

Naturally occurring vanadium in surface soils typically ranges from 20 to 500 mg/kg; concentrations greater than 2 mg/kg are potentially phytotoxic, although specific phytotoxicity levels for naturally occurring vanadium have not been documented. Concentrations above 550 mg/kg may result in adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 50 mg/kg. Vanadium can be associated with manganese, potassium, and organic matter and is typically concentrated in organic shales, coal, and crude oil. It is successfully analyzed by both ICP-AES and XRF with little interference.

1.5.13 Zinc

Naturally occurring zinc in surface soils typically ranges from 10 to 300 mg/kg; concentrations greater than 50 mg/kg are potentially phytotoxic. Zinc at concentrations above 23,000 mg/kg may result in adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 30 mg/kg. Zinc is a common contaminant in mine waste and at metal processing sites. In addition, it is highly soluble, which is a common concern for aquatic receptors. Zinc is successfully analyzed by ICP-AES; however, spectral interferences between peaks for copper and zinc may influence detection limits and the accuracy of the XRF analysis.

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Chapter 2
Field Sample Collection Locations

Although the field demonstration took place at KARS Park on Merritt Island, Florida, environmental samples were collected at other sites around the country to develop a demonstration sample that incorporated a variety of soil/sediment types and target element concentrations. This chapter describes these sample collection sites, as well as the rationale for the selection of each.

Several criteria were used to assess potential sample collection sites, including:

- The ability to provide a variety of target elements and soil/sediment matrices.
- The convenience and accessibility of the location to the sampling team.
- Program support and the cooperation of the site owner.

Nine sample collection sites were ultimately selected for the demonstration; one was the KARS Park site itself. These nine sites were selected to represent variable soil textures (sand, silt, and clay) and iron content, two factors that significantly affect instrument performance.

Historical operations at these sites included mining, smelting, steel manufacturing, and open burn pits; one, KARS Park, was a gun range. Thus, these sites incorporated a wide variety of metal contaminants in soils and sediments. Both contaminated and uncontaminated (background) samples were collected at each site.

A summary of the sample collection sites is presented in Table 2-1, which describes the types of metal-contaminated soils or sediments that were found at each site. This information is based on the historical data that were provided by the site owners or by the EPA remedial project managers.

2.1 Alton Steel Mill Site

The Alton Steel Mill site (formerly the Laclede Steel site) is located at 5 Cut Street in Alton, Illinois. This 400-acre site is located in Alton's industrial corridor. The Alton site was operated by Laclede Steel Company from 1911 until it went bankrupt in July 2001. The site was purchased by Alton Steel, Inc., from the bankruptcy estate of Laclede Steel in May 2003. The Alton site is heir to numerous environmental concerns from more than 90 years of steel production; site contaminants include polychlorinated biphenyls (PCBs) and heavy metals. Laclede Steel was cited during its operating years for improper management and disposal of PCB wastes and electric arc furnace dust that contained heavy metals such as lead and cadmium. A Phase I environmental site assessment (ESA) was conducted at the Alton site in May 2002, which identified volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), total priority pollutant metals, and PCBs as potential contaminants of concern at the site.

Based on the data gathered during the Phase I ESA and on discussions with Alton personnel, several soil samples were collected for the demonstration from two areas at the Alton site, including the Rod Patenting Building and the Tube Mill Building. The soil in the areas around these two buildings had not been remediated and was known to contain elevated concentrations of arsenic, cadmium, chromium, lead, nickel, zinc, and iron. The matrix of the contaminated soil samples was a fine to medium sand; the background soil sample was a sand loam.

Table 2-2 presents historical analytical data (the maximum concentrations) for some of the target elements detected at the Alton site.

Table 2-1. Nature of Contamination in Soil and Sediment at Sample Collection Sites

Sample Collection Site	Source of Contamination	Matrix	Site-Specific Metals of Concern for XRF Demonstration											
			Sb	As	Cd	Cr	Cu	Fe	Pb	Hg	Ni	Se	Ag	Zn
Alton Steel, Alton, IL	Steel manufacturing facility with metal arc furnace dust. The site also includes a metal scrap yard and a slag recovery facility.	Soil		X	X	X		X	X		X			X
Burlington Northern–ASARCO Smelter Site, East Helena, MT	Railroad yard staging area for smelter ores. Contaminated soils resulted from dumping and spilling concentrated ores.	Soil		X	X				X					
KARS Park – Kennedy Space Center, Merritt Island, FL	Impacts to soil from historical facility operations and a former gun range.	Soil	X	X		X	X		X					X
Leviathan Mine Site/Aspen Creek, Alpine County, CA	Abandoned open-pit sulfur and copper mine that has contaminated a 9-mile stretch of mountain creeks, including Aspen Creek, with heavy metals.	Soil and Sediment		X	X	X	X	X			X			
Naval Surface Warfare Center, Crane Division, Crane, IN	Open disposal and burning of general refuse and waste associated with aircraft maintenance.	Soil	X	X	X	X	X	X	X	X	X		X	X
Ramsay Flats–Silver Bow Creek, Butte, MT	Silver Bow Creek was used as a conduit for mining, smelting, industrial, and municipal wastes.	Soil and Sediment		X	X		X	X	X					X
Sulphur Bank Mercury Mine	Inactive mercury mine. Waste rock, tailings, and ore are distributed in piles throughout the property.	Soil	X	X					X	X				
Torch Lake Site (Great Lakes Area of Concern), Houghton County, MI	Copper mining produced mill tailings that were dumped directly into Torch Lake, contaminating the lake sediments and shoreline.	Sediment		X		X	X		X	X		X	X	X
Wickes Smelter Site, Jefferson City, MT	Abandoned smelter complex with contaminated soils and mineral-processing wastes, including remnant ore piles, decomposed roaster brick, slag piles and fines, and amalgamation sediments.	Soil	X	X	X	X	X	X	X		X			X

Notes (in order of appearance in table):

Sb:	Antimony	Cr:	Chromium	Pb:	Lead	Se:	Selenium
As:	Arsenic	Cu:	Copper	Hg:	Mercury	Ag:	Silver
Cd:	Cadmium	Fe:	Iron	Ni:	Nickel	Zn:	Zinc

Note: Vanadium was not a chemical of concern at any of the sites and so does not appear on the table.

Table 2-2. Historical Analytical Data, Alton Steel Mill Site

Metal	Maximum Concentration (mg/kg)
Arsenic	80.3
Cadmium	97
Chromium	1,551
Lead	3,556

2.2 Burlington Northern-ASARCO Smelter Site

The Burlington Northern (BN)-ASARCO Smelter site is located in the southwestern part of East Helena, Montana. The site was an active smelter for more than 100 years and closed in 2002. Most of the ore processed at the smelter was delivered on railroad cars. An area west of the plant site (the BN property) was used for temporary staging of ore cars and consists of numerous side tracks to the primary railroad line into the smelter. This site was selected to be included in the demonstration because it had not been remediated and contained several target elements in soil.

At the request of EPA, the site owner collected samples of surface soil in this area in November 1997 and April 1998 and analyzed them for arsenic, cadmium, and lead; elevated concentrations were reported for all three metals. The site owner collected 24 samples of surface soil (16 in November 1997 and 8 in April 1998). The soils were found to contain up to 2,018 parts per million (ppm) arsenic, 876 ppm cadmium, and 43,907 ppm lead. One sample of contaminated soil and one sample of background soil were collected. The contaminated soil was a light brown sandy loam with low organic carbon content. The background soil was a medium brown sandy loam with slightly more organic material than the contaminated soil sample. Table 2-3 presents the site owner's data for arsenic, cadmium, and lead (the maximum concentrations) from the 1997 and 1998 sampling events.

Table 2-3. Historical Analytical Data, BN-ASARCO Smelter Site

Metal	Maximum Concentration (ppm)
Arsenic	2,018
Cadmium	876
Lead	43,907

2.3 Kennedy Athletic, Recreational and Social Park Site

Soil and sediment at the KARS Park site were contaminated from former gun range operations and contain several target elements for the demonstration. The specific elements of concern for the KARS Park site include antimony, arsenic, chromium, copper, lead, and zinc.

The KARS Park site is located at the Kennedy Space Center on Merritt Island, Florida. KARS Park was purchased in 1962 and has been used by employees of the National Aeronautics and Space Administration (NASA), other civil servants, and guests as a recreational park since 1963. KARS Park occupies an area of Kennedy Space Center just outside the Cape Canaveral base. Contaminants in the park resulted from historical facility operations and impacts from the former gun range. The land north of KARS is owned by NASA and is managed by the U.S. Fish and Wildlife Service (USFWS) as part of the Merritt Island National Wildlife Refuge.

Two soil and two sediment samples were collected from various locations at the KARS Park site for the XRF demonstration. The contaminated soil sample was collected from an impact berm at the small arms range. The background soil sample was collected from a forested area near the gun range. The matrix of the contaminated and background soil samples consisted of fine to medium quartz sand. The sediment samples were collected from intermittently saturated areas within the skeet range. These samples were organic rich sandy loams. Table 2-4 presents historical analytical data (the maximum concentrations) for soil and sediment at KARS Park.

Table 2-4. Historical Analytical Data, KARS Park Site

Metal	Maximum Concentration (mg/kg)
Antimony	8,500
Arsenic	1,600
Chromium	40.2
Copper	290,000
Lead	99,000
Zinc	16,200

2.4 Leviathan Mine Site

The Leviathan Mine site is an abandoned copper and sulfur mine located high on the eastern slopes of the Sierra Nevada Mountain range near the California-Nevada border. Development of the Leviathan Mine began in 1863, when copper sulfate was mined for use in the silver refineries of the Comstock Lode. Later, the underground mine was operated as a copper mine until a mass of sulfur was encountered. Mining stopped until about 1935, when sulfur was extracted for use in refining copper ore. In the 1950s, the mine was converted to an open-pit sulfur mine. Placement of excavated overburden and waste rock in nearby streams created acid mine drainage and environmental impacts in the 1950s. Environmental impacts noted at that time included large fish kills.

Historical mining distributed waste rock around the mine site and created an open pit, adits, and solution cavities through mineralized rock. Oxygen in contact with the waste rock and mineralized rock in the adits oxidizes sulfur and sulfide minerals, generating acid. Water contacting the waste rock and flowing through the mineralized rock mobilizes the acid into the environment. The acid dissolves metals, including arsenic, copper, iron, and nickel, which creates conditions toxic to insects and fish in Leviathan, Aspen, and Bryant Creeks, downstream of the Leviathan Mine. Table 2-5 presents historical analytical data (the maximum concentrations) for the target elements detected at elevated concentrations in sediment samples collected along the three creeks. Four sediment and one soil sample were collected. One of the sediment samples was collected from the iron precipitate terraces formed from the acid mine drainage. The matrix of this sample appeared to be an orange silty clay loam. A second sediment sample was collected from the settling pond at the wastewater treatment system. The matrix of this sample was orange clay. A third sample was collected from the salt crust at the settling pond. This sample incorporated white crystalline material. One background sediment and one background soil sample were collected upstream of the mine. These samples consisted of light brown sandy loam.

Table 2-5. Historical Analytical Data, Leviathan Mine Site

Metal	Maximum Concentration (mg/kg)
Arsenic	2,510
Cadmium	25.7
Chromium	279
Copper	837
Nickel	2,670

2.5 Navy Surface Warfare Center, Crane Division Site

The Old Burn Pit at the Naval Surface Warfare Center (NSWC), Crane Division, was selected to be included in the demonstration because 6 of the 13 target elements were detected at significant concentration in samples of surface soil previously collected at the site.

The NSWC, Crane Division, site is located near the City of Crane in south-central Indiana. The Old Burn Pit is located in the northwestern portion of NSWC and was used daily from 1942 to 1971 to burn refuse. Residue from the pit was buried along with noncombustible metallic items in a gully north of the pit. The burn pit was covered with gravel and currently serves as a parking lot for delivery trailers. The gully north of the former burn pit has been revegetated. Several soil samples were collected from the revegetated area for the demonstration because the highest concentrations of the target elements were detected in soil samples collected previously from this area. The matrix of the contaminated and background soil samples was a sandy loam. The maximum concentrations of the target elements detected in surface soil during previous investigations are summarized in Table 2-6.

Table 2-6. Historical Analytical Data, NSWC Crane Division-Old Burn Pit

Metal	Maximum Concentration (mg/kg)
Antimony	301
Arsenic	26.8
Cadmium	31.1
Chromium	112
Copper	1,520
Iron	105,000
Lead	16,900
Mercury	0.43
Nickel	62.6
Silver	7.5
Zinc	5,110

2.6 Ramsay Flats-Silver Bow Creek Site

The Ramsay Flats-Silver Bow Creek site was selected to be included in the demonstration because 6 of the 13 target elements were detected in samples of surface sediment collected previously at the site. Silver Bow Creek originates north of Butte, Montana, and is a tributary to the upper Clark Fork River.

More than 100 years of nearly continuous mining have altered the natural environment surrounding the upper Clark Fork River. Early wastes from mining, milling, and smelting were dumped directly into Silver Bow Creek and were subsequently transported downstream. EPA listed Silver Bow Creek and a contiguous portion of the upper Clark Fork River as a Superfund site in 1983.

A large volume of tailings was deposited in a low-gradient reach of Silver Bow Creek in the Ramsay Flats area. Tailings at Ramsay Flats extend several hundred feet north of the Silver Bow Creek channel. About 18 inches of silty tailings overlie texturally stratified natural sediments that consist of low-permeability silt, silty clay, organic layers, and stringers of fine sand.

Two sediment samples were collected from the Ramsay Flats tailings area and were analyzed for a suite of metals using a field-portable XRF. The contaminated sediment sample was collected in Silver Bow Creek adjacent to the mine tailings. The matrix of this sediment sample was orange-brown silty fine sand with interlayered black organic material. The background sediment sample was collected upstream of Butte, Montana. The matrix of this sample was organic rich clayey silt with approximately 25 percent fine sand. The maximum concentrations of the target elements in the samples are summarized in Table 2-7.

Table 2-7. Historical Analytical Data, Ramsay Flats-Silver Bow Creek Site

Metal	Maximum Concentration (mg/kg)
Arsenic	176
Cadmium	141
Copper	1,110
Iron	20,891
Lead	394
Zinc	1,459

2.7 Sulphur Bank Mercury Mine Site

The Sulphur Bank Mercury Mine (SBMM) is a 160-acre inactive mercury mine located on the eastern shore of the Oaks Arm of Clear Lake in Lake County, California, 100 miles north of San Francisco. Between 1864 and 1957, SBMM was the site of underground and open-pit mining at the hydrothermal vents and hot springs. Mining disturbed about 160 acres of land at SBMM and generated large quantities of waste rock (rock that did not contain economic concentrations of mercury and was removed to gain access to ore), tailings (the waste material from processes that removed the mercury from ore), and ore (rock that contained economic concentrations of mercury that was mined and stockpiled for mercury extraction). The waste rock, tailings, and ore are distributed in piles throughout the property.

Table 2-8 presents historical analytical data (the maximum concentrations) for the target elements detected at elevated concentrations in surface samples collected at SBMM. Two contaminated soil samples and one background soil sample were collected at various locations for the demonstration project. The mercury sample was collected from the ore stockpile and consisted of medium to coarse sand. The second contaminated soil sample was collected from the waste rock pile and consisted of coarse sand and gravel with trace silt. The matrix of the background soil sample was brown sandy loam.

Table 2-8. Historical Analytical Data, Sulphur Bank Mercury Mine Site

Metal	Maximum Concentration (mg/kg)
Antimony	3,724
Arsenic	532
Lead	900
Mercury	4,296

2.8 Torch Lake Superfund Site

The Torch Lake Superfund site was selected because native and contaminated sediment from copper mining, milling, and smelting contained the elements targeted for the demonstration. The specific metals of concern for the Torch Lake Superfund site included arsenic, chromium, copper, lead, mercury, selenium, silver, and zinc.

The Torch Lake Superfund site is located on the Keweenaw Peninsula in Houghton County, Michigan. Wastes were generated at the site from the 1890s until 1969. The site was included on the National Priorities List in June 1986. Approximately 200 million tons of mining wastes were dumped into Torch Lake and reportedly filled about 20 percent of the lake's original volume. Contaminated sediments are believed to be up to 70 feet thick in some locations. Wastes occur both on the uplands and in the lake and are found in four forms, including poor rock piles, slag and slag-enriched sediments, stamp sands, and abandoned settling ponds for mine slurry.

EPA initiated long-term monitoring of Torch Lake in 1999; the first monitoring event (the baseline study) was completed in August 2001. Table 2-9 presents analytical data (the maximum concentrations) for eight target elements in sediment samples collected from Torch Lake during the baseline study. Sediment samples were collected from the Torch Lake site at various locations for the demonstration. The matrix of the sediment samples was orange silt and clay.

Table 2-9. Historical Analytical Data, Torch Lake Superfund Site

Metal	Maximum Concentration (mg/kg)
Arsenic	40
Chromium	90
Copper	5,850
Lead	325
Mercury	1.2
Selenium	0.7
Silver	6.2
Zinc	630

2.9 Wickes Smelter Site

The roaster slag pile at the Wickes Smelter site was selected to be included in the demonstration because 12 of the 13 target elements were detected in soil samples collected previously at the site.

The Wickes Smelter site is located in the unincorporated town of Wickes in Jefferson County, Montana. Wastes at the Wickes Smelter site include waste rock, slag, flue bricks, and amalgamation waste. The wastes are found in discrete piles and are mixed with soil. The contaminated soil sample was collected from a pile of roaster slag at the site. The slag was black, medium to coarse sand and gravel. The matrix of the background soil sample was a light brown sandy loam. Table 2-10 presents historical analytical data (maximum concentrations) for the roaster slag pile.

Table 2-10. Historical Analytical Data, Wickes Smelter Site-Roaster Slag Pile

Metal	Maximum Concentration (mg/kg)
Antimony	79
Arsenic	3,182
Cadmium	70
Chromium	13
Copper	948
Iron	24,780
Lead	33,500
Nickel	7.3
Silver	83
Zinc	5,299

Chapter 3
Field Demonstration

The field demonstration required a sample set and a single location (the demonstration site) where all the technology developers could assemble to analyze the sample set under the oversight of the EPA/Tetra Tech field team. This chapter describes how the sample set was created, how the demonstration site was selected, and how the field demonstration was conducted. Additional detail regarding these topics is available in the *Demonstration and Quality Assurance Project Plan* (Tetra Tech 2005).

3.1 Bulk Sample Processing

A set of samples that incorporated a variety of soil and sediment types and target element concentrations was needed to conduct a robust evaluation. The demonstration sample set was generated from the bulk soil and sediment samples that were collected from the nine sample collection sites described in Chapter 2. Both contaminated (environmental) and uncontaminated (background) bulk samples of soil and sediment were collected at each sample collection site. The background sample was used as source material for a spiked sample when the contaminated sample did not contain the required levels of target elements. By incorporating a spiked background sample into the sample set, the general characteristics of the soil and sediment sample matrix could be maintained. At the same time, this spiked sample assured that all target elements were present at the highest concentration levels needed for a robust evaluation.

3.1.1 Bulk Sample Collection and Shipping

Large quantities of soil and sediment were needed for processing into well-characterized samples for this demonstration. As a result, 14 soil samples and 11 sediment samples were collected in bulk quantity from the nine sample collection sites across the U.S. A total of approximately 1,500 kilograms of unprocessed soil and sediment was collected, which yielded more than 1,000 kilograms of soil and sediment after the bulk samples had been dried.

Each bulk soil sample was excavated using clean shovels and trowels and then placed into clean, plastic 5-gallon (19-liter) buckets at the sample collection site. The mass of soil and sediment in each bucket varied, but averaged about 25 kilograms per bucket. As a result, multiple buckets were needed to contain the entire quantity of each bulk sample.

Once it had been filled, a plastic lid was placed on each bucket, the lid was secured with tape, and the bucket was labeled with a unique bulk sample number. Sediment samples were collected in a similar method at all sites except at Torch Lake, where sediments were collected using a Vibracore or Ponar sediment sampler operated from a boat. Each 5-gallon bucket was overpacked in a plastic cooler and was shipped under chain of custody via overnight delivery to the characterization laboratory, Applied Research and Development Laboratory (ARDL).

3.1.2 Bulk Sample Preparation and Homogenization

Each bulk soil or sediment sample was removed from the multiple shipping buckets and then mixed and homogenized to create a uniform batch. Each bulk sample was then spread on a large tray at ARDL's laboratory to promote uniform air drying. Some bulk samples of sediment required more than 2 weeks to dry because of the high moisture content.

The air-dried bulk samples of soil and sediment were sieved through a custom-made screen to remove coarse material larger than about 1 inch. Next, each bulk sample was mechanically crushed using a hardened stainless-steel hammer mill until the particle size was sub-60-mesh sieve (less than 0.2 millimeters). The particle size of the processed bulk soil and sediment was measured after each round of crushing using standard sieve technology, and the particles that were still larger than 60-mesh were returned to the crushing process. The duration of the crushing process for each bulk sample varied based on soil type and volume of coarse fragments.

After each bulk sample had been sieved and crushed, the sample was mixed and homogenized using a

Model T 50A Turbula shaker-mixer. This shaker was capable of handling up to 50 gallons (190 liters) of sample material; thus, this shaker could handle the complete volume of each bulk sample. Bulk samples of smaller volume were mixed and homogenized using a Model T 10B Turbula shaker-mixer that was capable of handling up to 10 gallons (38 liters). Aliquots from each homogenized bulk sample were then sampled and analyzed in triplicate for the 13 target elements using ICP-AES and CVAA. If the relative percent difference between the highest and lowest result exceeded 10 percent for any element, the entire batch was returned to the shaker-mixer for additional homogenization. The entire processing scheme for the bulk samples is shown in Figure 3-1.

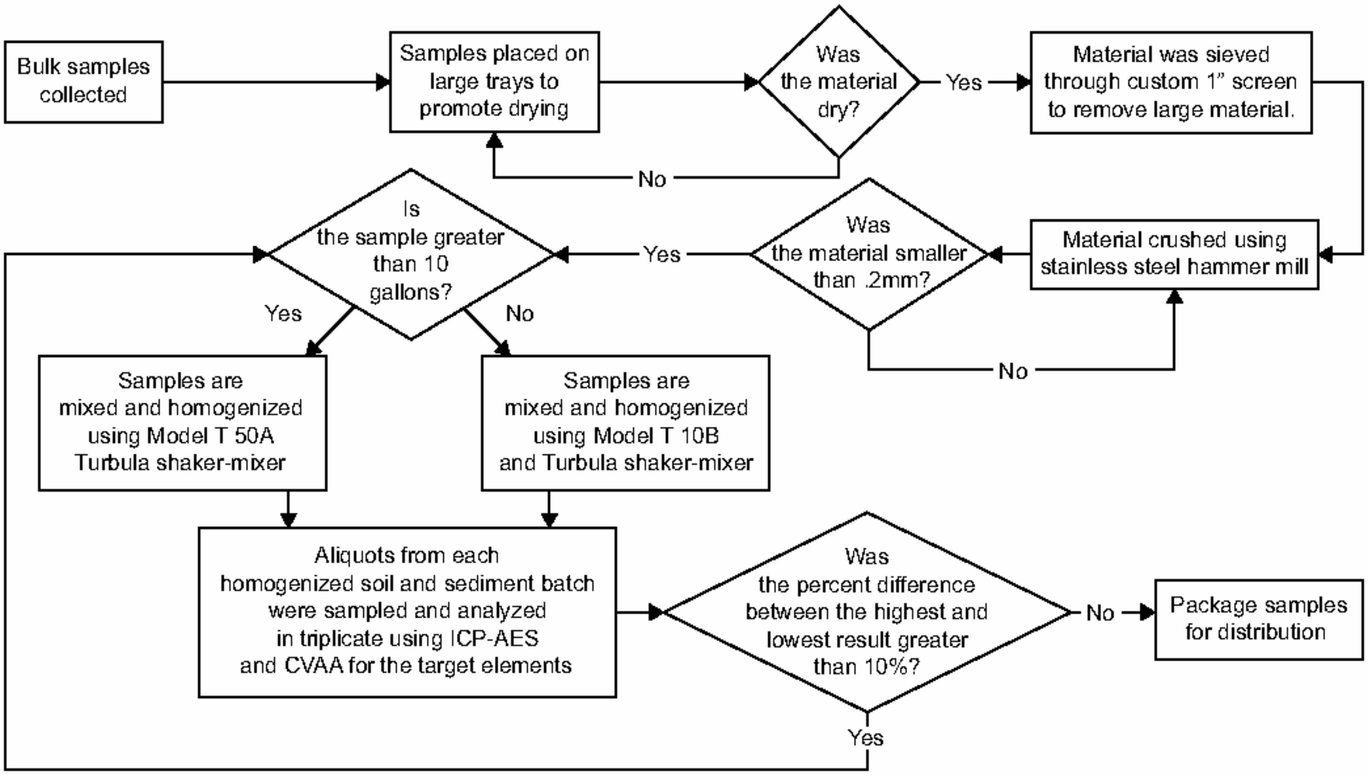


Figure 3-1. Bulk sample processing diagram.

3.2 Demonstration Samples

After the bulk soil and sediment sample material had been processed into homogenized bulk samples for the demonstration, the next consideration was the concentrations of target elements. The goal was to create a demonstration sample set that would cover the concentration range of each target element that may be reasonably found in the environment. Three concentration levels were identified as a basis for assessing both the coverage of the environmental samples and the need to generate spiked samples. These three levels were: (1) near the detection limit, (2) at intermediate concentrations, and (3) at high concentrations. A fourth concentration level (very high) was added for lead, iron, and zinc in soil and for iron in sediment. Table 3-1 lists the numerical ranges of the target elements for each of these levels (1 through 4).

3.2.1 Environmental Samples

A total of 25 separate environmental samples were collected from the nine sample collection sites described in Chapter 2. This bulk environmental sample set included 14 soil and 11 sediment samples. The concentrations of the target elements in some of these samples, however, were too high or too low to be used for the demonstration. Therefore, the initial analytical results for each bulk sample were used to establish different sample blends for each sampling location that would better cover the desired concentration ranges.

The 14 bulk soil samples were used to create 26 separate sample blends and the 11 bulk sediment samples were used to create 19 separate sample blends. Thus, there were 45 environmental sample blends in the final demonstration sample set. Either five or seven replicate samples of each sample blend were included in the sample set for analysis during the demonstration. Table 3-2 lists the number of sample blends and the number of demonstration samples (including replicates) that were derived from the bulk environmental samples for each sampling location.

3.2.2 Spiked Samples

Spiked samples that incorporated a soil and sediment matrix native to the sampling locations were created by adding known concentrations of target elements to the background samples. The spiked concentrations were selected to ensure that a minimum of three samples was available for all concentration levels for each target element.

After initial characterization at ARDL's laboratory, all bulk background soil and sediment samples were shipped to Environmental Research Associates (ERA) to create the spiked samples. The spiked elements were applied to the bulk sample in an aqueous solution, and then each bulk spiked sample was blended for uniformity and dried before it was repackaged in sample bottles.

Six bulk background soil samples were used at ERA's laboratory to create 12 separate spiked sample blends, and four bulk sediment samples were used to create 13 separate spiked sample blends. Thus, a total of 10 bulk background samples were used to create 25 spiked sample blends. Three or seven replicate samples of each spiked sample blend were included in the demonstration sample set. Table 3-3 lists the number of sample blends and the number of demonstration samples (including replicates) that were derived from the bulk background samples for each sampling location.

3.2.3 Demonstration Sample Set

In total, 70 separate blends of environmental and spiked samples were created and a set of 326 samples was developed for the demonstration by including three, five, or seven replicates of each blend in the final demonstration sample set. Thirteen sets of the demonstration samples, consisting of 326 individual samples in 250-milliliter clean plastic sample bottles, were prepared for shipment to the demonstration site and reference laboratory.

Table 3-1. Concentration Levels for Target Elements in Soil and Sediment

Analyte	Level 1 Target Range (mg/kg)	Level 2 Target Range (mg/kg)	Level 3 Target Range (mg/kg)	Level 4 Target Range (mg/kg)
SOIL				
Antimony	40 – 400	400 – 2,000	>2,000	
Arsenic	20 – 400	400 – 2,000	>2,000	
Cadmium	50 – 500	500 – 2,500	>2,500	
Chromium	50 – 500	500 – 2,500	>2,500	
Copper	50 – 500	500 – 2,500	>2,500	
Iron	60 – 5,000	5,000 – 25,000	25,000 – 40,000	>40,000
Lead	20 – 1,000	1,000 – 2,000	2,000 – 10,000	>10,000
Mercury	20 – 200	200 – 1,000	>1,000	
Nickel	50 – 250	250 – 1,000	>1,000	
Selenium	20 – 100	100 – 200	>200	
Silver	45 – 90	90 – 180	>180	
Vanadium	50 – 100	100 – 200	>200	
Zinc	30 – 1,000	1,000 – 3,500	3,500 – 8,000	>8,000
SEDIMENT				
Antimony	40 – 250	250 – 750	>750	
Arsenic	20 – 250	250 – 750	>750	
Cadmium	50 – 250	250 – 750	>750	
Chromium	50 – 250	250 – 750	>750	
Copper	50 – 500	500 – 1,500	>1,500	
Iron	60 – 5,000	5,000 – 25,000	25,000 – 40,000	>40,000
Lead	20 – 500	500 – 1,500	>1,500	
Mercury	20 – 200	200 – 500	>500	
Nickel	50 – 200	200 – 500	>500	
Selenium	20 – 100	100 – 200	>200	
Silver	45 – 90	90 – 180	>180	
Vanadium	50 – 100	100 – 200	>200	
Zinc	30 – 500	500 – 1,500	>1,500	

Table 3-2. Number of Environmental Sample Blends and Demonstration Samples

Sampling Location	Number of Sample Blends	Number of Demonstration Samples
Alton Steel Mill Site	2	10
Burlington Northern-ASARCO East Helena Site	5	29
Kennedy Athletic, Recreational and Social Park Site	6	32
Leviathan Mine Site	7	37
Naval Surface Warfare Center, Crane Division Site	1	5
Ramsay Flats—Silver Bow Creek Superfund Site	7	37
Sulphur Bank Mercury Mine Site	9	47
Torch Lake Superfund Site	3	19
Wickes Smelter Site	5	31
TOTAL *	45	247

* Note: The totals in this table add to those for the spiked blends and replicates as summarized in Table 3-3 to bring the total number of blends to 70 and the total number of samples to 326 for the demonstration.

Table 3-3. Number of Spiked Sample Blends and Demonstration Samples

Sampling Location	Number of Spiked Sample Blends	Number of Demonstration Samples
Alton Steel Mill Site	1	3
Burlington Northern-ASARCO East Helena Site	2	6
Leviathan Mine Site	5	15
Naval Surface Warfare Center, Crane Division Site	2	6
Ramsey Flats—Silver Bow Creek Superfund Site	6	22
Sulphur Bank Mercury Mine Site	3	9
Torch Lake Superfund Site	4	12
Wickes Smelter Site	2	6
TOTAL *	25	79

* Note: The totals in this table add to those for the unspiked blends and replicates as summarized in Table 3-2 to bring the total number of blends to 70 and the total number of samples to 326 for the demonstration.

3.3 Demonstration Site and Logistics

The field demonstration occurred during the week of January 24, 2005. This section describes the selection of the demonstration site and the logistics of the field demonstration, including sample management.

3.3.1 Demonstration Site Selection

The demonstration site was selected from among the list of sample collection sites to simulate a likely field deployment. The following criteria were used to assess which of the nine sample collection sites might best serve as the demonstration site:

- Convenience and accessibility to participants in the demonstration.
- Ease of access to the site, with a reasonably sized airport that can accommodate the travel schedules for the participants.
- Program support and cooperation of the site owner.
- Sufficient space and power to support developer testing.
- Adequate conference room space to support a visitors day.
- A temperate climate so that the demonstration could occur on schedule in January.

After an extensive search for candidates, the site selected for the field demonstration was KARS Park, which is part of the Kennedy Space Center on Merritt Island, Florida. KARS Park was selected as the demonstration site for the following reasons:

- Access and Site Owner Support — Representatives from NASA were willing to support the field demonstration by providing access to the site, assisting in logistical support during the demonstration, and hosting a visitors day.

- Facilities Requirements and Feasibility — The recreation building was available and was of sufficient size to accommodate all the demonstration participants. Furthermore, the recreation building had adequate power to operate all the XRF instruments simultaneously and all the amenities to fully support the demonstration participants, as well as visitors, in reasonable comfort.

- Ease of Access to the Site — The park, located about 45 minutes away from Orlando International Airport, was selected because of its easy accessibility by direct flight from many airports in the country. In addition, many hotels are located within 10 minutes of the site along the coast at Cocoa Beach, in a popular tourist area. Weather in this area of central Florida in January is dry and sunny, with pleasant daytime temperatures into the 70s (F) and cool nights.

3.3.2 Demonstration Site Logistics

The field demonstration was held in the recreation building, which is just south of the gunnery range at KARS Park. Photographs of the KARS Park recreation building, where all the XRF instruments were set up and operated, are shown in Figures 3-2 and 3-3.

A visitors day was held on January 26, 2005 when about 25 guests came to the site to hear about the demonstration and to observe the XRF instruments in operation. Visitors day presentations were conducted in a conference building adjacent to the recreation building at KARS Park (see Figure 3-4). Presentations by NASA and EPA representatives were followed by a tour of the XRF instruments in the recreation building while demonstration samples were being analyzed.

Figure 3-2. KARS Park recreation building.

Figure 3-3. Work areas for the XRF instruments in the recreation building.

Figure 3-4. Visitors day presentation.

3.3.3 EPA Demonstration Team and Developer Field Team Responsibilities

Each technology developer sent its instrument and a field team to the demonstration site for the week of January 24, 2005. The developer's field team was responsible for unpacking, setting up, calibrating, and operating the instrument. The developer's field team was also responsible for any sample preparation for analysis using the XRF instrument.

The EPA/Tetra Tech demonstration team assigned an observer to each instrument. The observer sat beside the developer's field team, or was nearby, throughout the field demonstration and observed all activities involved in setup and operation of the instrument. The observer's specific responsibilities included:

- Guiding the developer's field team to the work area in the recreation building at KARS Park and assisting with any logistical issues involved in instrument shipping, unpacking, and setup.
- Providing the demonstration sample set to the developer's field team in accordance with the sample management plan.
- Ensuring that the developer was operating the instrument in accordance with standard procedures and questioning any unusual practices or procedures.
- Communications with the developer's field team regarding schedules and fulfilling the requirements of the demonstration.
- Recording information relating to the secondary objectives of the evaluation (see Chapter 4) and for obtaining any cost information that could be provided by the developer's field team.
- Receiving the data reported by the developer's field team for the demonstration samples, and loading these data into a temporary database on a laptop computer.

Overall, the observer was responsible for assisting the developer's field team throughout the field demonstration and for recording all pertinent information and data for the evaluation. However, the observer was not allowed to advise the developer's field team on sample processing or to provide any feedback based on preliminary inspection of the XRF instrument data set.

3.3.4 Sample Management during the Field Demonstration

The developer's field team analyzed the demonstration sample set with its XRF instrument during the field demonstration. Each demonstration sample set was shipped to the demonstration site with only a reference number on each bottle as an identifier. The reference number was tied to the source information in the EPA/Tetra Tech database, but no information was provided on the sample label that might provide the developer's field team any insight as to the nature or content of the sample. Spiked samples were integrated with the environmental samples in a random manner so that the spiked samples could not be distinguished.

The demonstration sample set was divided into 13 subsets, or batches, for tracking during the field demonstration. The samples provided to each developer's field team were randomly distributed in two fashions. First, the order of the jars within each batch was random, so that the sample order for a batch was different for each developer's field team. Second, the distribution of sample batches was random, so that each developer's field team received the sample batches in a different order.

The observer provided the developer's field team with one batch of samples at a time. When the developer's field team reported that analysis of a batch was complete, the observer would reclaim all the unused sample material from that batch and then provide the next batch of samples for analysis. Chain-of-custody forms were used to document all sample transfers. When the analysis of all batches was complete, the observer assisted the developer's field team in cleanup of the work area and repackaging the instrument and any associated equipment. The members of the developer's field team were not allowed to take any part of the demonstration samples with them when they left the demonstration site.

Samples that were not in the possession of the developer's field team during the demonstration were held in a secure storage room adjacent to the demonstration work area (see Figure 3-5). The storage room was closed and locked except when the observer retrieved samples from the room. Samples were stored at room temperature during the demonstration, in accordance with the quality assurance/quality control (QA/QC) requirements established for the project.

Figure 3-5. Sample storage room.

3.3.5 Data Management

Each of the developer's field teams was able to complete analysis of all 326 samples during the field demonstration (or during the subsequent week, in one case when the developer's field team arrived late at the demonstration site because of delays in international travel). The data produced by each developer's field team were submitted during or at the end of the field demonstration in a standard Microsoft Excel® spreadsheet. (The EPA/Tetra Tech field team had provided a template.) Since each instrument provided data in a different format, the developer's field team was responsible for reducing the data before they were submitted and for transferring the data into the Excel spreadsheet.

The observer reviewed each data submittal for completeness, and the data were then uploaded into a master Excel spreadsheet on a laptop computer for temporary storage. Only the EPA/Tetra Tech field team had access to the master Excel spreadsheet during the field demonstration.

Once the EPA/Tetra Tech field team returned to their offices, the demonstration data were transferred to an Microsoft Access® database for permanent storage. Each developer's data, as they existed in the Access database, were then provided to the developer for review. Any errors the developers identified were corrected, and the database was then finalized. All statistical analysis and data evaluation took place on this final database.

Chapter 4
Evaluation Design

This chapter presents the approach for evaluating the performance of the XRF instruments. Specifically, the sections below describe the objectives of the evaluation and the experimental design.

The *Demonstration and Quality Assurance Project Plan* (Tetra Tech 2005) provides additional details on the overall demonstration approach. However, some deviations from the plan, involving data evaluation and laboratory audits, occurred after the demonstration plan was written. For completeness, the primary changes to the written plan are documented in the final section of this chapter.

4.1 Evaluation Objectives

The overall purpose of the XRF technology demonstration was to evaluate the performance of various field XRF instruments in detecting and quantifying trace elements in soils and sediments from a variety of sites around the U.S. The performance of each XRF instrument was evaluated in accordance with primary and secondary objectives. Primary objectives are critical to the evaluation and require the use of quantitative results to draw conclusions about an instrument's performance. Secondary objectives pertain to information that is useful but that will not necessarily require use of quantitative results to draw conclusions about an instrument's performance.

The primary and secondary objectives for the evaluation are listed in Table 4-1. These objectives were based on:

- Input from MMT Program stakeholders, including developers and EPA staff.
- General expectations of users of field measurement instruments.
- The time available to complete the demonstration.
- The capabilities of the instruments that the developers participating in the demonstration intended to highlight.

4.2 Experimental Design

To address the first four primary objectives, each XRF instrument analyzed the demonstration sample set for the 13 target elements. The demonstration samples originated from multiple sampling locations across the country, as described in Chapter 2, to provide a diverse set of soil and sediment matrices. The demonstration sample set included both blended environmental samples and spiked background samples, as described in Chapter 3, to provide a wide range of concentrations and combinations of elements.

When the field demonstration was completed, the results obtained using the XRF instruments were compared with data from a reference laboratory to evaluate the performance of each instrument in terms of accuracy and comparability (Primary Objective 2). The results for replicate samples were used to evaluate precision in various concentration ranges (Primary Objective 3) and the method detection limits (MDL) (Primary Objective 1). Each of these quantitative evaluations of instrument performance was carried out for each target element. The effect of chemical and spectral interferences and of soil characteristics (Primary Objectives 4 and 5) were evaluated to help explain extreme deviations or outliers observed in the XRF results when compared with the reference laboratory results.

A second important comparison involved the average performance of all eight XRF instruments that participated in the demonstration. For the first three primary objectives (MDL, accuracy, precision), the performance of each individual instrument was compared to the overall average performance of all eight instruments. Where the result of the instrument under consideration was less than 10 percent different than the average result for all eight instruments, the result was considered "equivalent." A similar comparison was conducted with respect to cost (Primary Objective 7). These comparisons were intended to illustrate the performance of each XRF instrument in relation to its peers.

The evaluation design for meeting each objective, including data analysis procedures, is discussed in more detail in the sections below. Where specific deviations from these procedures were necessary for the data set associated with specific instruments, these deviations are described as part of the performance evaluation in Chapter 7.

4.2.1 Primary Objective 1 — Method Detection Limits

The MDL for each target element was evaluated based on the analysis of sets of seven replicate samples that contained the target element at concentrations near the detection limit. The MDL was calculated using the procedures found in Title 40 Code of Federal Regulations (CFR) Part 136, Appendix B, Revision 1.11. The following equation was used:

$$\mathrm{MDL} = t_{(n-1,1-\alpha=0.99)}(s)$$

where

MDL = method detection limit
t = Student's t value for a 99 percent confidence level and a standard deviation estimate with *n-1* degrees of freedom
n = number of samples
s = standard deviation.

Table 4-1. Evaluation Objectives

Objective	Description
Primary Objective 1	Determine the MDL for each target element.
Primary Objective 2	Evaluate the accuracy and comparability of the XRF measurement to the results of laboratory reference methods for a variety of contaminated soil and sediment samples.
Primary Objective 3	Evaluate the precision of XRF measurements for a variety of contaminated soil and sediment samples.
Primary Objective 4	Evaluate the effect of chemical and spectral interference on measurement of target elements.
Primary Objective 5	Evaluate the effect of soil characteristics on measurement of target elements.
Primary Objective 6	Measure sample throughput for the measurement of target elements under field conditions.
Primary Objective 7	Estimate the costs associated with XRF field measurements.
Secondary Objective 1	Document the skills and training required to properly operate the instrument.
Secondary Objective 2	Document health and safety concerns associated with operating the instrument.
Secondary Objective 3	Document the portability of the instrument.
Secondary Objective 4	Evaluate the instrument's durability based on its materials of construction and engineering design.
Secondary Objective 5	Document the availability of the instrument and of associated customer technical support.

Based on the data provided by the characterization laboratory before the demonstration, a total of 12 sample blends (seven for soil and five for sediment) were identified for use in the MDL determination.

The demonstration approach specified the analysis of seven replicates for each of these sample blends by both the developer and the reference laboratory. It was predicted that these blends would allow the determination of a minimum of one MDL for soil and one MDL for sediment for each element, with the exception of iron. This prediction was based on the number of sample blends that contained concentrations less than 50 percent lower or higher than the lower limit of the Level 1 concentration range (from 20 to 50 ppm, depending on the element), as presented in Table 3-1.

After the field demonstration, the data sets obtained by the developers and the reference laboratory for the MDL sample blends were reviewed to confirm that they were appropriate to use in calculating MDLs. The requirements of 40 CFR 136, Appendix B, were used as the basis for this evaluation. Specifically, the CFR states that samples to be used for MDL determinations should contain concentrations in the range of 1 to 5 times the predicted MDL. On this basis, and using a nominal predicted reporting limit of 50 ppm for the target elements based on past XRF performance and developer information, a concentration of 250 ppm (5 times the "predicted" nominal MDL) was used as a threshold in selecting samples to calculate the MDL. Thus, each of the 12 MDL blends that contained mean reference laboratory concentrations less than 250 ppm were used in calculating MDLs for a given target element. Blends with mean reference laboratory concentrations greater than 250 ppm were discarded for evaluating this objective.

For each target element, an MDL was calculated for each sample blend with a mean concentration within the prescribed range. If multiple MDLs could be calculated for an element from different sample blends, these results were averaged to arrive at an overall mean MDL for the demonstration. The mean MDL for each target element was then categorized as either low (MDL less than 20 ppm), medium (MDL between 20 and 100 ppm), or high (MDL exceeds 100 ppm). No blends were available to calculate a detection limit for iron because all the blends contained substantial native concentrations of iron.

4.2.2 Primary Objective 2 — Accuracy

Accuracy was assessed based on a comparison of the results obtained by the XRF instrument with the results from the reference laboratory for each of the 70 blends in the demonstration sample set. The results from the reference laboratory were essentially used as a benchmark in this comparison, and the accuracy of the XRF instrument results was judged against them. The limitations of this approach should be recognized, however, because the reference laboratory results were not actually "true values." Still, there was a high degree of confidence in the reference laboratory results for most elements, as described in Chapter 5.

The following data analysis procedure was followed for each of the 13 target elements to assess the accuracy of an XRF instrument:

1. The results for replicate samples within a blend were averaged for both the data from the XRF instrument and the reference laboratory. Since there were 70 sample blends, this step created a maximum of 70 paired results for the assessment.

2. A blend that exhibited one or more non-detect values in either the XRF instrument or the reference laboratory analysis was excluded from the evaluation.

3. A blend was excluded from the evaluation when the average result from the reference laboratory was below a minimum concentration. The minimum concentration for exclusion from the accuracy assessment was identified as the lower limit of the lowest concentration range (Level 1 in Table 3-1), which is about 50 ppm for most elements.

4. The mean result for a blend obtained with the XRF instrument was compared with the corresponding mean result from the reference laboratory by calculating a relative percent difference (RPD). This comparison was carried out for each of the paired XRF and reference laboratory results included in the evaluation (up to 70 pairs) as follows:

$$\text{RPD} = \frac{(M_R - M_D)}{\text{average}(M_R, M_D)}$$

where

M_R = the mean reference laboratory measurement
M_D = the mean XRF instrument measurement.

5. Steps 1 through 4 provided a set of up to 70 RPDs for each element (70 sample blends minus the number excluded in steps 1 and 2). The absolute value of each of the RPDs was taken and summary statistics (minimum, maximum, mean and median) were then calculated.

6. The accuracy of the XRF instrument for each target element was then categorized, based on the median of the absolute values of the RPDs, as either excellent (RPD less than 10 percent), good (RPD between 10 percent and 25 percent), fair (RPD between 25 percent and 50 percent), or poor (RPD above 50 percent).

7. The set of absolute values of the RPDs for each instrument and element was further evaluated to assess any trends in accuracy versus concentration. These evaluations involved grouping the RPDs by concentration range (Levels 1 through 3 and 4, as presented in Table 3-1), preparing summary statistics for each range, and assessing differences among the grouped RPDs.

The absolute value of the RPDs was taken in step 5 to provide a more sensitive indicator of the extent of differences between the results from the XRF instrument and the reference laboratory. However, the absolute value of the RPDs does not indicate the direction of the difference and therefore does not reflect bias.

The populations of mean XRF and mean reference laboratory results were assessed through linear correlation plots to evaluate bias. These plots depict the linear relationships between the results for the XRF instrument and reference laboratory for each target element using a linear regression calculation with an associated correlation coefficient (r^2). These plots were used to evaluate the existence of general bias between the data sets for the XRF instrument and the reference laboratory.

4.2.3 Primary Objective 3 — Precision

The precision of the XRF instrument analysis for each target element was evaluated by comparing the results for the replicate samples in each blend. All 70 blends in the demonstration sample set (including environmental and spiked samples) were included in at least triplicate so that precision could be evaluated across all concentration ranges and across different matrices.

The precision of the data for a target element was evaluated for each blend by calculating the mean relative standard deviation (RSD) with the following equation:

$$RSD = \left| \frac{SD}{\overline{C}} \right| x\ 100$$

where

RSD = Relative standard deviation
SD = Standard deviation
$\overline{C}$ = Mean concentration.

The standard deviation was calculated using the equation:

$$SD = \left[\frac{1}{n-1} \sum_{k=1}^{n} \left(C_k - \overline{C} \right)^2 \right]^{\frac{1}{2}}$$

where

SD = Standard deviation
n = Number of replicate samples
C_k = Concentration of sample K
$\overline{C}$ = Mean concentration.

The following specific procedure for data analysis was followed for each of the 13 target elements to assess XRF instrument precision:

1. The RSD for the replicate samples in a blend was calculated for both data from the XRF instrument and the reference laboratory. Since there were 70 sample blends, this step created a maximum of 70 paired RSDs for the assessment.

2. A blend that exhibited one or more non-detect values in either the XRF or the reference laboratory analysis was excluded from the evaluation.

3. A blend was excluded from the evaluation when the average result from the reference laboratory was below a minimum concentration. The minimum concentration for exclusion from the precision assessment was identified as the lower limit of the lowest concentration range (Level 1 in Table 3-1), which was about 50 ppm for most elements.

4. The RSDs for the various blends for both the XRF instrument and the reference laboratory were treated as a statistical population. Summary statistics (minimum, maximum, mean and median) were then calculated and compared for the data set as a whole and for the different concentration ranges (Levels 1 through 3 or 4).

5. The precision of the XRF instrument for each target element was then categorized, based on the median RSDs, as either excellent (RSD less than 5 percent), good (RSD between 5 percent and 10 percent), fair (RSD between 10 percent and 20 percent), or poor (RSD above 20 percent).

One primary evaluation was a comparison of the mean RSD for each target element between the XRF instrument and the reference laboratory. Using this comparison, the precision of the XRF instrument could be evaluated against the precision of accepted fixed-laboratory methods. Another primary evaluation was a comparison of the mean RSD for each target element between the XRF instrument and the overall average of all XRF instruments. Using this comparison, the precision of the XRF instrument could be evaluated against its peers.

4.2.4 Primary Objective 4 — Impact of Chemical and Spectral Interferences

The potential in the XRF analysis for spectral interference between adjacent elements on the periodic table was evaluated for the following element pairs: lead/arsenic, nickel/copper, and copper/zinc. The demonstration sample set included multiple blends where the concentration of one of these elements was greater than 10 times the concentration of the other element in the pair to facilitate this evaluation. Interference effects were identified through evaluation of the RPDs for these sample blends, which were calculated according to the equation in Section 4.2.2, since spectral interferences would occur only in the XRF data and not in the reference laboratory data.

Summary statistics for RPDs (mean, median, minimum, and maximum) were calculated for each potentially affected element for the sample blends with high relative concentrations (greater than 10 times) of the potentially interfering element. These summary statistics were compared with the RPD statistics for sample blends with lower concentrations of the interfering element. It was reasoned that spectral interference should be directly reflected in increased RPDs for the interference samples when compared with the rest of the demonstration sample set.

In addition to spectral interferences (caused by overlap of neighboring spectral peaks), the data sets were assessed for indications of chemical interferences. Chemical interferences occur when the x-rays characteristic of an element are absorbed or emitted by another element within the sample, causing low or high bias. These interferences are common in samples that contain high levels of iron, where low biases for copper and high biases for chromium can result. The evaluations for Primary Objective 4 therefore included RPD comparisons between sample blends with high concentrations of iron (more than 50,000 ppm) and other sample blends. These RPD comparisons were performed for the specific target elements of interest (copper, chromium, and others) to assess chemical interferences from iron. Outliers and subpopulations in the RPD data sets for specific target elements, as identified through graphical means (probability plots and box plots), were also examined for potential interference effects.

The software that is included with many XRF instruments can correct for chemical interferences. The results of this evaluation were intended to differentiate the instruments that incorporated effective software for addressing chemical interferences.

4.2.5 Primary Objective 5 — Effects of Soil Characteristics

The demonstration sample set included soil and sediment samples from nine locations across the U.S. and a corresponding variety of soil types and lithologies. The accuracy and precision statistics (RPD and RSD) were grouped by soil type (sample location) and the groups were compared to assess the effects of soil characteristics. Outliers and subpopulations in the RPD data sets, as identified through graphical means (correlation plots and box plots), were also examined for matrix effects.

4.2.6 Primary Objective 6 — Sample Throughput

Sample throughput is a calculation of the total number of samples that can be analyzed in a specified time. The primary factors that affect sample throughput are the time required to prepare a sample for analysis, to conduct the analytical procedure for each sample, and to process and tabulate the resulting data. The time required to prepare and to analyze demonstration samples was recorded each day that demonstration samples were analyzed.

Sample throughput can also be affected by the time required to set up and calibrate the instrument as well as the time required for quality control. The time required to perform these activities was also recorded during the field demonstration.

An overall mean processing time per sample and an overall sample throughput rate was calculated based on the total time required to complete the analysis of the demonstration sample set from initial instrument setup through data reporting. The overall mean processing time per sample was then used as the primary basis for comparative evaluations.

4.2.7 Primary Objective 7 — Technology Costs

The costs for analysis are an important factor in the evaluation and include the cost for the instrument, analytical supplies, and labor. The observer collected information on each of these costs during the field demonstration.

Based on input from each technology developer and from distributors, the instrument cost was established for purchase of the equipment and for daily, weekly, and monthly rental. Some of the technologies are not yet widely available, and the developer has not established rental options. In these cases, an estimated weekly rental cost was derived for the summary cost evaluations based on the purchase price for the instrument and typical rental to purchase price ratios for similar instruments. The costs associated with leasing agreements were also specified in the report, if available.

Analytical supplies include sample cups, spoons, x-ray film, Mylar®, reagents, and personal protective equipment. The rate that the supplies are consumed was monitored and recorded during the field demonstration. The cost of analytical supplies was estimated per sample from these consumption data and information on unit costs.

Labor includes the time required to prepare and analyze the samples and to set up and dismantle the equipment. The labor hours associated with preparing and analyzing samples and with setting up and dismantling the equipment were recorded during the demonstration. The labor costs were calculated based on this information and typical labor rates for a skilled technician or chemist.

In addition to the assessment of the above-described individual cost components, an overall cost for a field effort similar to the demonstration was compiled and compared to the cost of fixed laboratory analysis. The results of the cost evaluation are presented in Chapter 8.

4.2.8 Secondary Objective 1 — Training Requirements

Each XRF instrument requires that the operator be trained to safely set up and operate the instrument. The relative level of education and experience that is appropriate to operate the XRF instrument was assessed during the field demonstration.

The amount of specific training required depends on the complexity of the instrument and the associated software. Most developers have established training programs. The time required to complete the developer's training program was estimated and the content of the training was identified.

4.2.9 Secondary Objective 2 — Health and Safety

The health and safety requirements for operation of the instrument were identified, including any that are associated with potential exposure from radiation and to reagents. Not included in the evaluation were potential risks from exposure to site-specific hazardous materials or physical safety hazards associated with the demonstration site.

4.2.10 Secondary Objective 3 — Portability

The portability of the instrument depends on size, weight, number of components, power requirements, and reagents required. The size of the instrument, including physical dimensions and weight, was recorded (see Chapter 6). The number of components, power requirements, support structures, and reagent requirements were also recorded. A qualitative assessment of portability was conducted based on this information.

4.2.11 Secondary Objective 4 — Durability

The durability of the instrument was evaluated by gathering information on the warranty and expected lifespan of the radioactive source or x-ray tube. The ability to upgrade software or hardware also was evaluated. Weather resistance was evaluated if the instrument is intended for use outdoors by examining the instrument for exposed electrical connections and openings that may allow water to penetrate.

4.2.12 Secondary Objective 5 — Availability

The availability of the instrument from the developer, distributors, and rental agencies was documented. The availability of replacement parts and instrument-specific supplies was also noted.

4.3 Deviations from the Demonstration Plan

Although the field demonstration and subsequent data evaluations generally followed the *Demonstration and Quality Assurance Project Plan* (Tetra Tech 2005), there were some deviations as new information was uncovered or as the procedures were reassessed while the plan was executed. These deviations are documented below for completeness and as a supplement to the demonstration plan:

1. An in-process audit of the reference laboratory was originally planned while the laboratory was analyzing the demonstration samples. However, the reference laboratory completed all analysis earlier than expected, during the week of the field demonstration, and thereby created a schedule conflict. Furthermore, it was decided that the original pre-award audit was adequate for assessing the laboratory's procedures and competence.

2. The plan suggested that each result for spiked samples from the reference laboratory would be replaced by the "certified analysis" result, which was quantitative based on the amount of each element spiked, whenever the RPD between these two results was greater than 10 percent. The project team agreed that 10 percent was too stringent for this evaluation, however, and decided to use 25 percent RPD as the criterion for assessing reference laboratory accuracy against the spiked samples. Furthermore, it was found during the data evaluations that replacing individual reference laboratory results using this criterion would result in a mixed data set. Therefore, the 25 percent criterion was applied to the overall mean RPD for each element, and the "certified analysis" data set for a specific target element was used as a supplement to the reference laboratory result when this criterion was exceeded.

3. Instrument accuracy and comparability in relation to the reference laboratory (Primary Objective 2) was originally planned to be assessed based on a combination of percent recovery (instrument result divided by reference laboratory result) and RPD. It was decided during the data analysis, however, that the RPD was a much better parameter for this assessment. Specifically, it was found that the mean or median of the absolute values of the RPD for each blend was a good discriminator of instrument performance for this objective.

4. Although this step was not described in the plan, some quantitative results for each instrument were compared with the overall average of all XRF instruments. Since there were eight instruments, it was believed that a comparison of

this type did not violate EPA's agreement with the technology developers that one instrument would not be compared with another. Furthermore, this comparison provides an easy-to-understand basis for assessing instrument performance.

5. The plan proposed statistical testing in support of Primary Objectives 4 and 5. Specifically, the Wilcoxon Rank Sum (WRS) test was proposed to assist in evaluating interference effects, and the Rosner outlier test was proposed in evaluating other matrix effects on XRF data quality (EPA 2000; Gilbert 1987). However, these statistical tests were not able to offer any substantive performance information over and above the evaluations based on RPDs and regression plots because of the limited sample numbers and scatter in the data. On this basis, the use of these two statistical tests was not further explored or presented.

Chapter 5
Reference Laboratory

As described in Chapter 4, a critical part of the evaluation was the comparison of the results obtained for the demonstration sample set by the XRF instrument with the results obtained by a fixed laboratory (the reference laboratory) using conventional analytical methods. Therefore, a significant effort was undertaken to ensure that data of the highest quality were obtained as the reference data for this demonstration. This effort included three main activities:

- Selection of the most appropriate methods for obtaining reference data,
- Selection of a high-quality reference laboratory, and
- Validation of reference laboratory data and evaluation of QA/QC results.

This chapter describes the information that confirms the validity, reliability, and usability of the reference laboratory data based on each of the three activities listed above (Sections 5.1, 5.2, and 5.3). Finally, this chapter presents conclusions (Section 5.4) on the level of data quality and the usability of the data obtained by the reference laboratory.

5.1 Selection of Reference Methods

Methods for analysis of elements in environmental samples, including soils and sediments, are well established in the environmental laboratory industry. Furthermore, analytical methods appropriate for soil and sediment samples have been promulgated by EPA in the compendium of methods, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW-846) (EPA 1996c). Therefore, the methods selected as reference methods for the demonstration were the SW-846 methods most typically applied by environmental laboratories to soil and sediment samples, as follows:

- Inductively coupled plasma-atomic emission spectroscopy (ICP-AES), in accordance with EPA SW-846 Method 3050B/6010B, for all target elements except mercury
- Cold vapor atomic absorption (CVAA) spectroscopy, in accordance with EPA SW-846 Method 7471A, for mercury only

Selection of these analytical methods for the demonstration was supported by the following additional considerations: (1) the methods are widely available and widely used in current site characterizations, remedial investigations, risk assessments, and remedial actions; (2) substantial historical data are available for these methods to document that their accuracy and precision are adequate to meet the objectives of the demonstration; (3) these methods have been used extensively in other EPA investigations where confirmatory data were compared with XRF data; and (4) highly sensitive alternative methods were less suitable given the broad range of concentrations that were inherent in the demonstration sample set. Specific details on the selection of each method are presented below.

Element Analysis by ICP-AES. Method 6010B (ICP-AES) was selected for 12 of the target elements because its demonstrated accuracy and precision meet the requirements of the XRF demonstration in the most cost-effective manner. The ICP-AES method is available at most environmental laboratories and substantial data exist to support the claim that the method is both accurate and precise enough to meet the objectives of the demonstration.

Inductively coupled plasma-mass spectrometry (ICP-MS) was considered as a possible analytical technique; however, fewer data were available to support the claims of accuracy and precision. Furthermore, it was available in less than one-third of the laboratories solicited for this project. Finally, ICP-MS is a technique for analysis of trace elements and often requires serial dilutions to mitigate the effect of high concentrations of interfering ions or other matrix interferences. These dilutions can introduce the possibility of error and contaminants that might bias the results. Since the matrices (soil and sediment) for this demonstration are designed to contain high concentrations of elements and interfering ions, ICP-AES was selected over ICP-MS as the instrumental method best suited to meet the project objectives. The cost per analysis is also higher for ICP-MS in most cases than for ICP-AES.

Soil/Sediment Sample Preparation by Acid Digestion. The elements in soil and sediment samples must be dissolved from the matrix into an aqueous solution by acid digestion before analysis by ICP-AES. Method 3050B was selected as the preparation method and involves digestion of the matrix using a combination of nitric and hydrochloric acids, with the addition of hydrogen peroxide to assist in degrading organic matter in the samples. Method 3050B was selected as the reference preparation method because extensive data are available that suggest it efficiently dissolves most elements, as required for good overall recoveries and method accuracy. Furthermore, this method was selected over other digestion procedures because it is the most widely used dissolution method. In addition, it has been used extensively as the digestion procedure in EPA investigations where confirmatory data were compared with XRF data.

The ideal preparation reference method would completely digest silicaceous minerals. However, total digestion is difficult and expensive and is therefore seldom used in environmental analysis. More common strong acid-based extractions, like that used by EPA Method 3050B, recover most of the heavy element content. In addition, stronger and more vigorous digestions may produce two possible drawbacks: (1) loss of elements through volatilization, and (2) increased dissolution of interfering species, which may result in inaccurate concentration values.

Method 3052 (microwave-assisted digestion) was considered as an alternative to Method 3050B, but was not selected because it is not as readily available in environmental laboratories.

Soil/Sediment Sample Preparation for Analysis of Mercury by CVAA. Method 7471A (CVAA) is the only method approved by EPA and promulgated for analysis of mercury. Method 7471A includes its own digestion procedure because more vigorous digestion of samples, like that incorporated in Method 3050B, would volatilize mercury and produce inaccurate results. This technique is widely available, and extensive data are available that support the ability of this method to meet the objectives of the demonstration.

5.2 Selection of Reference Laboratory

The second critical step in ensuring high-quality reference data was selection of a reference laboratory with proven credentials and quality systems. The reference laboratory was procured via a competitive bid process. The procurement process involved three stages of selection: (1) a technical proposal, (2) an analysis of performance audit samples, and (3) an on-site laboratory technical systems audit (TSA). Each stage was evaluated by the project chemist and a procurement specialist.

In Stage 1, 12 analytical laboratories from across the U.S. were invited to bid by submitting extensive technical proposals. The technical proposals included:

- A current statement of qualifications.
- The laboratory quality assurance manual.
- Standard operating procedures (SOP) (including sample receipt, laboratory information management, sample preparation, and analysis of elements).
- Current instrument lists.
- Results of recent analysis of performance evaluation samples and audits.
- Method detection limit studies for the target elements.
- Professional references, laboratory personnel experience, and unit prices.

Nine of the 12 laboratories submitted formal written proposals. The proposals were scored based on technical merit and price, and a short list of five laboratories was identified. The scoring was weighed heavier for technical merit than for price. The five laboratories that received the highest score were advanced to stage 2.

In stage 2, each of the laboratories was provided with a set of six samples to analyze. The samples consisted of three certified reference materials (one soil and two sediment samples) at custom spiking concentrations, as well as three pre-demonstration soil samples. The results received from each laboratory were reviewed and assessed. Scoring at this stage was based on precision (reproducibility of results for the three pre-demonstration samples), accuracy (comparison of results to certified values for the certified reference materials), and completeness of the data package (including the hard copy and electronic data deliverables). The two laboratories that received the highest score were advanced to stage 3.

In stage 3, the two candidate laboratories were subjected to a thorough on-site TSA by the project chemist. The audit consisted of a direct comparison of the technical proposal to the actual laboratory procedures and conditions. The audit also tracked the pre-demonstration samples through the laboratory processes from sample receipt to results reporting. When the audit was conducted, the project chemist verified sample preparation and analysis for the three pre-demonstration samples. Each laboratory was scored on identical checklists.

The reference laboratory was selected based on the highest overall score. The weights of the final scoring selection were as follows:

Scoring Element	Relative Importance
Audits (on site)	40%
Performance evaluation samples, including data package and electronic data deliverable	50%
Price	10%

Based on the results of the evaluation process, Shealy Environmental Services, Inc. (Shealy), of Cayce, South Carolina, received the highest score and was therefore selected as the reference laboratory. Shealy is accredited by the National Environmental Laboratory Accreditation Conference (NELAC). Once selected, Shealy analyzed all demonstration samples (both environmental and spiked samples) concurrently with the developers' analysis during the field demonstration. Shealy analyzed the samples by ICP-AES using EPA SW-846 Method 3050B/6010B and by CVAA using EPA SW-846 Method 7471A.

5.3 QA/QC Results for Reference Laboratory

All data and QC results from the reference laboratory were reviewed in detail to determine that the reference laboratory data were of sufficiently high quality for the evaluation. Data validation of all reference laboratory results was the primary review tool that established the level of quality for the data set (Section 5.3.1). Additional reviews included the on-site TSA (Section 5.3.2) and other evaluations (Section 5.3.3).

5.3.1 Reference Laboratory Data Validation

After all demonstration samples had been analyzed, reference data from Shealy were fully validated according to the EPA validation document, *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (EPA 2004c) as required by the *Demonstration and Quality Assurance Project Plan* (Tetra Tech 2005). The reference laboratory measured 13 target elements, including antimony, arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, vanadium, and zinc. The reference laboratory reported results for 22 elements at the request of EPA; however, only the data for the 13 target elements were validated and included in data comparisons for meeting project objectives. A complete summary of the validation findings for the reference laboratory data is presented in Appendix C.

In the data validation process, results for QC samples were reviewed for conformance with the acceptance criteria established in the demonstration plan. Based on the validation criteria specified in the demonstration plan, all reference laboratory data were declared valid (were not rejected). Thus, the completeness of the data set was 100 percent. Accuracy and precision goals were met for most of the QC samples, as were the criteria for comparability, representativeness, and sensitivity. Thus, all reference laboratory data were deemed usable for comparison to the data obtained by the XRF instruments.

Only a small percentage of the reference laboratory data set was qualified as undetected as a result of blank contamination (3.3 percent) and estimated because of matrix spike and matrix spike duplicate (MS/MSD) recoveries (8.7 percent) and serial dilutions results (2.5 percent). Table 5.1 summarizes the number of validation qualifiers applied to the reference laboratory data according to QC type. Of the three QC types, only the MS/MSD recoveries warranted additional evaluation. The MS/MSD recoveries for antimony were marginally low (average recovery of 70.8 percent) when compared with the QC criterion of 75 to 125 percent recovery. It was concluded that low recoveries for antimony are common in analysis of soil and sediment by the prescribed methods and likely result from volatilization during the vigorous acid digestion process or spectral interferences found in soil and sediments matrices (or both). In comparison to antimony, high or low recoveries were observed only on an isolated basis for the other target metals (for example, lead and mercury) such that the mean and median percent recoveries were well within the required range. Therefore, the project team decided to evaluate the XRF data against the reference laboratory data for all 13 target elements and to evaluate the XRF data a second time against the ERA certified spike values for antimony only. These comparisons are discussed in Section 7.1. However, based on the validation of the complete reference data set and the low occurrence of qualified data, the reference laboratory data set as a whole was declared of high quality and of sufficient quality to make valid comparisons to XRF data.

5.3.2 Reference Laboratory Technical Systems Audit

The TSA of the Shealy laboratory was conducted by the project chemist on October 19, 2004, as part of the selection process for the reference laboratory. The audit included the review of element analysis practices (including sample preparation) for 12 elements by EPA Methods 3050B and 6010B and for total mercury by EPA Method 7471A. All decision-making personnel for Shealy were present during the TSA, including the laboratory director, QA officer, director of inorganics analysis, and the inorganics laboratory supervisor. Project-specific requirements were reviewed with the Shealy project team as were all the QA criteria and reporting requirements in the demonstration plan. It was specifically noted that the demonstration samples would be dried, ground, and sieved before they were submitted to the laboratory, and that the samples would be received with no preservation required (specifically, no chemical preservation and no ice). The results of the performance audit were also reviewed.

No findings or nonconformances that would adversely affect data quality were noted. Only two minor observations were noted; these related to the revision dates of two SOPs. Both observations were discussed at the debriefing meeting held at the laboratory after the TSA. Written responses to each of the observations were not required; however, the laboratory resolved these issues before the project was awarded. The auditor concluded that Shealy complied with the demonstration plan and its own SOPs, and that data generated at the laboratory should be of sufficient and known quality to be used as a reference for the XRF demonstration.

5.3.3 Other Reference Laboratory Data Evaluations

The data validation indicated that all results from the reference laboratory were valid and usable for comparison to XRF data, and the pre-demonstration TSA indicated that the laboratory could fully comply with the requirements of the demonstration plan for producing data of high quality. However, the reference laboratory data were evaluated in other ways to support the claim that reference laboratory data are of high quality. These evaluations included the (1) assessment of accuracy based on ERA-certified spike values, (2) assessment of precision based on replicate measurements within the same sample blend, and (3) comparison of reference laboratory data to the initial characterization data that was obtained when the blends were prepared. Each of these evaluations is briefly discussed in the following paragraphs.

Blends 46 through 70 of the demonstration sample set consisted of certified spiked samples that were used to assess the accuracy of the reference laboratory data. The summary statistics from comparing the "certified values" for the spiked samples with the reference laboratory results are shown in Table 5-2. The target for percent recovery was 75 to 125 percent. The mean percent recoveries for 12 of the 13 target elements were well within this accuracy goal. Only the mean recovery for antimony was outside the goal (26.8 percent). The low mean percent recovery for antimony supported the recommendation made by the project team to conduct a secondary comparison of XRF data to ERA-certified spike values for antimony. This secondary evaluation was intended to better understand the impacts on the evaluation of the low bias for antimony in the reference laboratory data. All other recoveries were acceptable. Thus, this evaluation further supports the conclusion that the reference data set is of high quality.

All blends (1 through 70) were prepared and delivered with multiple replicates. To assess precision, percent RSDs were calculated for the replicate sample results submitted by the reference laboratory for each of the 70 blends. Table 5-3 presents the summary statistics for the reference laboratory data for each of the 13 target elements. These summary statistics indicate good precision in that the median percent RSD was less than 10 percent for 11 out of 13 target elements (and the median RSD for the other two elements was just above 10 percent). Thus, this evaluation further supports the conclusion that the reference data set is of high quality.

Table 5-1. Number of Validation Qualifiers

	Number and Percentage of Qualified Results per QC type [1]					
	Method Blank		**MS/MSD**		**Serial Dilution**	
Element	**Number**	**Percent[2]**	**Number**	**Percent[2]**	**Number**	**Percent[2]**
Antimony	5	1.5	199	61.0	8	2.4
Arsenic	12	3.7	3	0.9	10	3.1
Cadmium	13	4.0	0	0	6	1.8
Chromium	0	0	0	0	10	3.1
Copper	1	0.3	0	0	8	2.4
Iron	0	0	0	0	10	3.1
Lead	0	0	34	10.5	11	3.4
Mercury	68	20.9	31	9.5	4	1.2
Nickel	0	0	0	0	10	3.1
Selenium	16	4.9	0	0	3	0.9
Silver	22	6.7	102	31.3	7	2.1
Vanadium	0	0	0	0	9	2.8
Zinc	1	0.3	0	0	10	3.1
Totals	138	3.3	369	8.7	106	2.5

Notes:

MS	Matrix spike.
MSD	Matrix spike duplicate.
QC	Quality control.

[1] This table presents the number of "U" (undetected) and "J" (estimated) qualifiers added to the reference laboratory data during data validation. Though so qualified, these results are considered usable for the demonstration. As is apparent in the "Totals" row at the bottom of this table, the amount of data that required qualifiers for any specific QC type was invariably less than 10 percent. No reference laboratory data were rejected (that is, qualified "R") during the data validation.

[2] Percents for individual elements are calculated based on 326 results per element. Total percents at the bottom of the table are calculated based on the total number of results for all elements (4,238).

ARDL, in Mount Vernon, Illinois, was selected as the characterization laboratory to prepare environmental samples for the demonstration. As part of its work, ARDL analyzed several samples of each blend to evaluate whether the concentrations of the target elements and the homogeneity of the blends were suitable for the demonstration. ARDL analyzed the samples using the same methods as the reference laboratory; however, the data from the characterization laboratory were not validated and were not intended to be equivalent to the reference laboratory data. Rather, the intent was to use the results obtained by the characterization laboratory as an additional quality control check on the results from the reference laboratory.

A review of the ARDL characterization data in comparison to the reference laboratory data indicated that ARDL obtained lower recoveries of several elements. When expressed as a percent of the average reference laboratory result (percent recovery), the median ARDL result was below the lower QC limit of 75 percent recovery for three elements — chromium, nickel, and selenium. This discrepancy between data from the reference laboratory and ARDL was determined to have no significant impact on reference laboratory data quality for three reasons: (1) the ARDL data were obtained on a rapid turnaround basis to evaluate homogeneity — accuracy was not a specific goal, (2) the ARDL data were not validated, and (3) all other quality measurement for the reference laboratory data indicated a high level of quality.

5.4 Summary of Data Quality and Usability

A significant effort was undertaken to ensure that data of high quality were obtained as the reference data for this demonstration. The reference laboratory data set was deemed valid, usable, and of high quality based on the following:

- Comprehensive selection process for the reference laboratory, with multiple levels of evaluation.
- No data were rejected during data validation and few data qualifiers were added.
- The observations noted during the reference laboratory audit were only minor in nature; no major findings or non-conformances were documented.
- Acceptable accuracy (except for antimony, as discussed in Section 5.3.3) of reference laboratory results in comparison to spiked certified values.
- Acceptable precision for the replicate samples in the demonstration sample set.

Based on the quality indications listed above, the reference laboratory data were used in the evaluation of XRF demonstration data. A second comparison was made between XRF data and certified values for antimony (in Blends 46 through 70) to address the low bias exhibited for antimony in the reference laboratory data.

Table 5-2. Percent Recovery for Reference Laboratory Results in Comparison to ERA Certified Spike Values for Blends 46 through 70

Statistic	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg	Ni	Se	Ag	V	Zn
Number of %R values	16	14	20	12	20	NC	12	15	16	23	20	15	10
Minimum %R	12.0	65.3	78.3	75.3	51.7	NC	1.4	81.1	77.0	2.2	32.4	58.5	0.0
Maximum %R	36.1	113.3	112.8	108.6	134.3	NC	97.2	243.8	116.2	114.2	100.0	103.7	95.2
Mean %R[1]	**26.8**	88.7	90.0	94.3	92.1	NC	81.1	117.3	93.8	89.9	78.1	90.4	90.6
Median %R[1]	**28.3**	90.1	87.3	97.3	91.3	NC	88.0	93.3	91.7	93.3	84.4	95.0	91.3

Notes:

[1] Values shown in bold fall outside the 75 to 125 percent acceptance criterion for percent recovery.

ERA = Environmental Resource Associates, Inc.

NC = Not calculated.

%R = Percent recovery.

Source of certified values: Environmental Resource Associates, Inc.

Sb	Antimony
As	Arsenic
Cd	Cadmium
Cr	Chromium
Cu	Copper
Fe	Iron
Pb	Lead
Hg	Mercury
Ni	Nickel
Se	Selenium
Ag	Silver
V	Vanadium
Zn	Zinc

Table 5-3. Precision of Reference Laboratory Results for Blends 1 through 70

Statistic	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg	Ni	Se	Ag	V	Zn
Number of %RSDs	43	69	43	69	70	70	69	62	68	35	44	69	70
Minimum %RSD	1.90	0.00	0.91	1.43	0.00	1.55	0.00	0.00	0.00	0.00	1.02	0.00	0.99
Maximum %RSD	78.99	139.85	40.95	136.99	45.73	46.22	150.03	152.59	44.88	37.30	54.21	43.52	48.68
Mean %RSD[1]	17.29	13.79	12.13	11.87	10.62	10.56	14.52	16.93	10.28	13.24	12.87	9.80	10.94
Median %RSD[1]	11.99	10.01	9.36	8.29	8.66	8.55	9.17	7.74	8.12	9.93	8.89	8.34	7.54

Notes:

[1] Values shown in bold fall outside precision criterion of less than or equal to 25 %RSD.

%RSD = Percent relative standard deviation.

Based on the three to seven replicate samples included in Blends 1 through 70.

Sb	Antimony
As	Arsenic
Cd	Cadmium
Cr	Chromium
Cu	Copper
Fe	Iron
Pb	Lead
Hg	Mercury
Ni	Nickel
Se	Selenium
Ag	Silver
V	Vanadium
Zn	Zinc

Chapter 6
Technology Description

The X-Met 3000TX XRF analyzer is manufactured by the portable division of Oxford Instruments Analytical Ltd. (previously Metorex International). This chapter provides a technical description of the X-Met based on information obtained from Oxford and observation of this analyzer during the field demonstration. This chapter also identifies an Oxford company contact, where additional technical information may be obtained.

6.1 General Description

The X-Met is a portable hand-held XRF analyzer that utilizes a miniature x-ray tube as the excitation source and a Peltier-cooled silicon-PiN diode x-ray detector. The X-Met can be used to detect a wide range of elements in soils, sediment, and solids, including thick homogeneous samples (plastics and metals). The X-Met can analyze elements that would require three isotope sources in traditional XRF analyzers.

The X-Met weighs about 4 pounds (1.8 kg) and is powered in the field with two lithium-ion batteries, or with AC power, if available. The X-Met utilizes an HP iPAQ pocket personal computer (PC) personal data assistant (PDA) for data storage of up to 15,000 tests with spectra in its 64 MB memory. The iPAQ PDA provides a color, high-resolution display, with variable backlighting. Data can be transferred from the iPAQ to another PC using a flash card or using Microsoft ActiveSync software over a USB cable. The iPAQ PDA can also be fitted with Bluetooth© wireless printing and data downloading for wireless data and file transfer.

The X·MET can analyze elements from titanium to uranium simultaneously. Elements from potassium to scandium can also be analyzed with higher detection limits. Typical applications are:

- Environmental samples – Analysis of elements in soils, slurries, liquids, filters, and dust wipes.
- Alloy analysis – Chemistry and grade identification of most alloys, metal powders, sintered alloys, and metallic coatings.
- Process analytical – Elemental analysis of powders, ores, and mining samples; equipment surfaces, coatings, and other samples, including vegetation, oils, water, plastics, ceramics, and glass.

Special features of the X-Met include a sample tray designed for the analyzing soil in plastic bags and a sample tray designed for analyzing soils in polyethylene cups. For bench-top analysis, the X-Met can be inverted and placed in a specially designed fabricated stand. The iPAQ PDA can be connected to the analyzer through a USB cable for easier viewing (Figure 6-2). Other special internal features include multiple x-ray beam filters, adjustable tube voltages and currents, and the selection from several pre-programmed calibration modules. The X-Met comes from the factory with a fundamental parameters calibration program that utilizes Compton scattering intensity to correct for the changing matrices between samples. In addition, the analyzer can be calibrated using site-specific samples in order to provide more accurate results. The customer can select from the standard factory calibrations or can calibrate based on user-generated linear, quadratic, or exponential functions. The X-Met software allows for visual observance and the identification of spectra.

XRF analyses using the X-Met can be fully compliant with EPA Method 6200, “Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.” Since XRF analysis is nondestructive, samples analyzed by XRF can be sent to a fixed analytical laboratory for confirmation of results.

The technical specifications for the X-Met are presented in Table 6-1. The analyzer can be set up either as a hand-held instrument for portable in-situ analysis (Figure 6-1) or as a bench-top instrument using a plastic stand (Figure 6-2).

Table 6-1. Oxford Instruments X-Met 3000TX Analyzer Technical Specifications

Weight:	4 pounds (1.8 kg)
Dimensions:	Hand-held.
Excitation Source:	Miniature x-ray tube; 40 kV, 40 µamps – programmable.
Detector:	Si-PiN Diode.
Element Range:	Titanium to uranium.
Display:	Color TFT 320 x 240 pixels. 65,536 colors.
Memory:	64 MB. Stores a minimum of 15,000 spectra and unlimited results.
Batteries:	(2) Li ion batteries.
Battery Charger/AC Adaptor:	110/220 V AC, 45-65 Hz.
Operating Environment:	Temperature: -10 ºC to +50 ºC.
Safety Features:	IR sample sensor. Failsafe status lights. Safety shield for small parts.
Software Interface:	Windows CE.
Data Transfer:	USB or wireless Bluetooth via Microsoft ActiveSync.
Bench-top Operation:	Bench top instrument stand. PDA cradle and remote extension cable standard.
Warranty:	Instrument – 2 years. X-ray tube – 5 years.

Figure 6-1. Oxford X-Met 3000TX set up for portable in situ analysis.

Figure 6-2. Oxford X-Met 3000TX set up for bench-top analysis.

To analyze soil samples in the in situ mode, the instrument x-ray window is placed directly on the ground or on soils in a plastic bag. In situ testing with the X-Met allows for semi-quantitative assessment of element concentrations at multiple locations or over large areas in a short time. For ex situ analysis, samples are prepared in x-ray sample cups and placed on a sample tray directly beneath the instrument x-ray window. Quantitative ex situ testing involves properly preparing the samples, placing the samples in x-ray sample cups, and analyzing them in a controlled area, typically free from dust and weather extremes. Most field-portable XRF analyses use a combination of in situ and ex situ sample testing.

Oxford Instruments does not have formal published standard operating procedures for X-Met operations, but recommends that users follow EPA Method 6200 and the instrument user's manual to ensure that the appropriate protocol is followed.

6.2 Instrument Operations during the Demonstration

The X-Met can be shipped via regular ground or air transportation. Because the x-ray tube only emits radiation during operation, the analyzer can be transported on aircraft as carry-on baggage. For the field demonstration, the analyzer was packed in a Pelican case and was carried on the plane. One additional large box was needed to hold all the accessories and supplies for routine analysis. A laptop PC is not required for analysis, but was used during the field demonstration for data downloading, manipulations, and storage.

6.2.1 Set Up and Calibration

The Oxford X-Met was set up and operated in the bench-top mode for this demonstration (Figure 6-2). The analyzer was placed in the instrument stand on a table with a sample tray designed for holding small plastic bags of soil (Figure 6-3). The HP iPAQ pocket PC was removed from the PDA cradle and connected to the analyzer with a USB cable. The laptop computer was set up and plugged into the 110-volt power supply. The total time to set up the instrument was less than 30 minutes.

Figure 6-3. Oxford X-Met 3000TX sample container with sample.

The X-Met was previously programmed with both an empirical calibration and fundamental parameters calibration algorithms. One specific empirical calibration model was created for this demonstration. The calibration model was built using self-made standards, National Institute of Standards and Testing (NIST) standards, and the pre-demonstration samples sent to Oxford Instruments prior to the demonstration.

Calibrating the analyzer consisted of selecting the specific empirical calibration program from the PDA menu and analyzing two calibration check standards (two NIST soil standards). The empirical calibration was used for all soil and sediment sample analysis during the demonstration. On the first day of the demonstration, forty samples were analyzed using both the empirical calibration and fundamental parameters calibration models. For the remainder of the demonstration, all soil and sediment samples (286 samples) were analyzed using only the empirical calibration. Samples that contained any target element at concentrations above the empirical calibration range were analyzed a second time using the fundamental parameters calibration. The second analysis was completed as a quality control check. All soil and sediment concentration data reported at the end of the demonstration were produced using the empirical calibration model.

6.2.2 Demonstration Sample Processing

Oxford sent a two-person field team to the demonstration site to prepare and analyze samples using the X-Met. However, it was observed that a single trained individual could have efficiently performed all the required sample processing tasks.

Each soil and sediment sample container was arranged in numeric order and poured from the sample jar into a 6 inch tall by 3.5 inch wide by 0.05 millimeter thick plastic bag. The sample bags used during the demonstration were special bags that are relatively impermeable to organic contaminants. For routine XRF analysis, standard self-sealing plastic sandwich bags (without white labels) can be used. Each soil and sediment sample was analyzed by placing the prepared sample in the sample tray, closing the sample tray lid, and pushing and holding the start button. When the start button was released, analysis stopped and the sample was removed from the sample tray.
Each sample was analyzed for approximately 2 minutes using the 40kV x-ray source.

At the end of each test, the sample concentrations were reviewed to determine if that sample should be analyzed a second time using the fundamental parameters calibration. The analytical results were saved to the iPAQ PDA using the appropriate sample number. Samples containing any target elements at concentrations above the empirical calibration ranges were placed aside to be analyzed at the end of each sample batch. Once per each sample group, a sample was analyzed in triplicate as an internal quality control check on instrument precision. At the end of each day, the data from the PDA were transferred to the laptop PC using a comma separated value (CSV) format.

6.3 General Demonstration Results

Oxford analyzed all 326 soil and sediment samples in 4 days using the empirical calibration for soil and sediment. All analyses were completed in the ex situ mode after the soil and sediment materials were placed in the sample bags. Data processing for the demonstration samples was completed within the iPAQ PDA that is part of the XRF analyzer. The data was transferred to a laptop PC either by using Bluetooth wireless technology or by removing the iPAQ from the analyzer cradle and attaching it to the laptop PC.

6.4 Contact Information

Additional information on Oxford's X-Met 3000TX XRF analyzer is available from the following source:

Dr. John Patterson
Oxford Instruments Analytical Ltd.
945 Busse Road
Elk Grove Village, IL 60007
Telephone: (800) 678-1117
Email: jpatterson@msys.oxinst.com

Chapter 7
Performance Evaluation

As discussed in Chapter 6, Oxford analyzed all 326 demonstration samples of soil and sediment at the field demonstration site between January 24 and 27, 2005. Additional data processing and demobilization occurred for a short time on January 28, 2005. An electronic data set for the X-Met was delivered to the Tetra Tech field team in Microsoft Excel® spreadsheet format as Oxford demobilized from the site. All data provided by Oxford at the close of the demonstration are tabulated and compared with the reference laboratory data and the applicable ERA-certified spike concentrations in Appendix D, Table D-1.

Like all the other developers, Oxford was given the opportunity to correct their data following the field demonstration and prior to receiving the reference laboratory data for comparison. Oxford did not provide any corrections to their data prior to the deadline given. However, following receipt of the reference laboratory data, Oxford notified Tetra Tech of apparent transcription errors in the original data spreadsheet prepared at the site, and submitted a revised data set for mercury, nickel, and selenium. Table D-2 of Appendix D presents this revised data set. These revised data were accepted for inclusion in this report because Oxford's claims regarding transcription errors in the original data set for these three elements appeared to have validity. However, the EPA/Tetra Tech evaluation team cannot provide any guarantee that the revised data submitted by Oxford represents only a correction of transcription errors since Oxford was in possession of the reference laboratory data at the time the corrections were submitted.

The data set for the X-Met was reviewed and evaluated in accordance with the primary and secondary objectives of the demonstration. EPA expected the demonstration data set submitted by each developer to be complete and correct at the time the field demonstration was completed, or to be corrected by the developer prior to receipt of the reference laboratory data. However, Oxford's claims of data transcription errors appeared reasonable based on review of the original and revised data sets, and initial evaluation of the original data sets for mercury, nickel, and selenium relative to the primary objectives indicated very erratic performance for these elements, consistent with the presence of data manipulation errors. Therefore, the evaluations below focus on the revised X-Met data set supplied by Oxford. For completeness purposes, however, separate sets of summary statistics for the original data set (mercury, nickel, and selenium only) in support of the primary objectives are included with the corresponding statistical summaries for the revised data in Appendix E. The original data set is not further discussed or interpreted relative to the primary objectives in this chapter.

7.1 Primary Objective 1 — Method Detection Limits

Samples were selected to calculate MDLs for each target element from the 12 potential MDL sample blends, as described in Section 4.2.1. Oxford reported non-detect values as "0" (Appendix D). In general, only blends for which all seven replicate analyses were reported as detections by Oxford were used for MDL calculation. Because only one blend met this requirement for vanadium, three additional blends were used for MDL calculation where only six of the seven replicates were detections. Iron was not included in the MDL evaluation, as was discussed in Section 4.2.1.

The MDLs calculated for the X-Met from the revised data set are presented in Table 7-1. As shown, the data for the MDL blends allowed the calculation of only three MDLs for antimony, cadmium, and silver. The number of calculated MDLs for the remaining target elements ranged between four (nickel and vanadium) and 10 (chromium). Also shown in Table 7-1 are the mean MDLs calculated for each target element, which are classified as follows:

- Very low (1 to 20 ppm): arsenic and selenium.
- Low (20 to 50 ppm): cadmium, copper, lead, mercury, silver, and zinc.

- Medium (50 to 100 ppm): nickel.

- High (greater than 100 ppm): antimony, chromium, and vanadium

The highest mean MDL of 185 ppm was calculated for vanadium. Blend 8 from the Wickes Smelter site produced anomalously high soil MDLs for vanadium as well as for antimony and cadmium. Table 7-1 indicates that the X-Met concentrations for these elements in Blend 8 were significantly higher than the reference laboratory concentration. Blend 8 was a roaster slag matrix that contained high concentrations of other elements, such as arsenic, copper, lead, and zinc. Other instruments participating in the demonstration also showed poor relative performance for this blend. Generalized biases in the MDL blend concentrations were also apparent for some elements. For example, the X-Met reported detections in multiple MDL blends for selenium that were reported as non-detect by the reference laboratory (which used a more sensitive method), indicating the possibility of a generalized high bias in the XRF data for this element. Conversely, the X-Met reported non detections for nickel in multiple blends where the reference laboratory reported concentrations that should have been detectable by the X-Met (that is, concentrations near or above the final mean MDL calculated for nickel), indicating a potential low bias.

A second set of MDLs was calculated for mercury, nickel, and selenium using the original data set. This set of MDLs is presented in Appendix E (Table E-1).

The mean MDLs calculated for the X-Met are compared in Table 7-2 with the mean MDLs for all instruments that participated in the demonstration and with the mean MDLs derived from performance data presented in EPA Method 6200 (EPA 1998e). As shown, the mean MDLs for the X-Met are significantly lower than were calculated from EPA Method 6200 data for all target elements except antimony. When compared with the overall average results for all eight XRF instruments that participated in the demonstration, the X-Met exhibited high relative mean MDLs for seven of the target elements, including antimony, chromium, copper, mercury, nickel, silver, and vanadium.

Table 7-2. Comparison of X-Met 3000TX MDLs to All-Instrument and EPA Method 6200 Data[1]

Element	X-Met 3000TX Mean MDLs[2]	All Instrument Mean MDLs[3]	EPA Method 6200 Mean Detection Limits[4]
Antimony	177	61	55 [5]
Arsenic	15	26	92
Cadmium	48	70	NR
Chromium	155	83	376
Copper	30	23	171
Lead	32	40	78
Mercury	35	23	NR
Nickel	70	50	100 [5]
Selenium	6	8	NR
Silver	49	42	NR
Vanadium	185	28	NR
Zinc	39	38	89

Notes:

[1] Detection limits are in units of milligrams per kilogram (mg/kg), or parts per million (ppm).

[2] The mean MDLs calculated for this technology demonstration, as presented in Table 7-1.

[3] The mean MDLs calculated for all eight XRF instruments that participated in this technology demonstration.

[4] Mean values calculated from Table 4 of Method 6200 (EPA 1998e, www.epa.gov/sw-846).

[5] Only one value reported.

EPA U.S. Environmental Protection Agency.

MDL Method detection limit.

NR Not reported; no MDLs reported for this element.

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Oxford X-Met 3000TX[1]

Matrix	**Blend No.**	**Antimony**			**Arsenic**			**Cadmium**			**Chromium**		
		X-Met MDL[2]	**X-Met Conc.[3]**	**Ref. Lab Conc[4]**	**X-Met MDL[2]**	**X-Met Conc. [3]**	**Ref. Lab Conc. [4]**	**X-Met MDL[2]**	**X-Met Conc. [3]**	**Ref. Lab Conc. [4]**	**X-Met MDL[2]**	**X-Met Conc. [3]**	**Ref. Lab Conc. [4]**
Soil	2	NC	ND	17	NC	ND	1.5	NC	ND	ND	**141**	187	167
Soil	5	NC	ND	ND	**17**	53	47	NC	ND	1.9	**49**	115	121
Soil	6	NC	ND	8	NC	245	477	NC	ND	12	**165**	104	133
Soil	8	**321**	438	118	NC	5314	3,943	**85**	240	91	NC	ND	55
Soil	10	NC	ND	ND	**13**	45	39	NC	ND	0.96	**154**	149	116
Soil	12	**121**	221	62	NC	533	559	**41**	282	263	**175**	192	101
Soil	18	NC	ND	ND	**11**	14	9	NC	ND	ND	**170**	199	150
Sediment	29	NC	ND	ND	**15**	12	10	NC	ND	ND	**145**	77	63
Sediment	31	**89**	56	ND	**26**	20	11	NC	ND	ND	**267**	303	133
Sediment	32	NC	ND	ND	**11**	37	31	NC	ND	ND	**179**	92	75
Sediment	39	NC	ND	ND	**14**	21	14	NC	ND	ND	**100**	136	102
Sediment	65	NC	ND	11	NC	356	250	**20**	41	44	NC	427	303
Mean X-Met MDL		**177**			**15**			**48**			**155**		

Matrix	**Blend No.**	**Copper**			**Lead**			**Mercury**			**Nickel**		
		X-Met MDL[2]	**X-Met Conc. [3]**	**Ref. Lab Conc. [4]**	**X-Met MDL[2]**	**X-Met Conc. [3]**	**Ref. Lab Conc. [4]**	**X-Met MDL[2]**	**X-Met Conc. [3]**	**Ref. Lab Conc. [4]**	**X-Met MDL[2]**	**X-Met Conc. [3]**	**Ref. Lab Conc. [4]**
Soil	2	**38**	49	47	NC	1273	1,200	NC	ND	ND	**54**	85	83
Soil	5	**30**	51	49	**64**	123	78	NC	ND	ND	NC	ND	60
Soil	6	**55**	182	160	NC	4133	3,986	NC	ND	0.83	NC	ND	70
Soil	8	NC	1710	1,243	NC	43011	33,429	NC	ND	15	NC	ND	57
Soil	10	**11**	30	31	**21**	86	72	NC	ND	0.14	NC	ND	60
Soil	12	NC	965	747	NC	4651	4,214	NC	ND	1.8	NC	ND	91
Soil	18	**15**	43	50	**20**	39	17	**19**	63	56	NC	ND	213
Sediment	29	NC	2074	1,986	**23**	70	33	NC	ND	0.24	NC	ND	72
Sediment	31	NC	1489	1,514	**29**	77	51	**49**	54	ND	NC	ND	196
Sediment	32	**18**	46	36	**32**	64	26	**25**	49	ND	NC	ND	174
Sediment	39	**49**	124	94	**33**	70	27	**43**	89	ND	**62**	82	202
Sediment	65	**26**	108	69	**35**	36	25	**41**	91	32	**92**	181	214
Mean X-Met MDL		**30**			**32**			**35**			**70**		

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Oxford X-Met 3000TX[1] (Continued)

Matrix	Blend No.	Selenium X-Met MDL[2]	Selenium X-Met Conc.[3]	Selenium Ref. Lab Conc.[4]	Silver X-Met MDL[2]	Silver X-Met Conc.[3]	Silver Ref. Lab Conc.[4]	Vanadium X-Met MDL[2]	Vanadium X-Met Conc.[3]	Vanadium Ref. Lab Conc.[4]	Zinc X-Met MDL[2]	Zinc X-Met Conc.[3]	Zinc Ref. Lab Conc.[4]
Soil	2	**6**	4	ND	NC	ND	ND	NC	ND	1.2	NC	ND	24
Soil	5	**7**	3	ND	NC	ND	0.93	**123**[5]	53	55	**26**	180	229
Soil	6	NC	ND	ND	NC	ND	14	NC	ND	56	NC	753	886
Soil	8	NC	ND	ND	**56**	204	144	**323**	250	34	NC	8566	5,657
Soil	10	NC	ND	ND	NC	ND	ND	NC	ND	51	**82**	93	92
Soil	12	**10**	20	15	**43**	55	38	**155**[5]	44	45	NC	2968	2,114
Soil	18	**4**	3	ND	NC	ND	ND	NC	ND	67	**20**	79	90
Sediment	29	**3**	3	ND	NC	ND	ND	NC	ND	96	**26**	226	160
Sediment	31	**8**	6	ND	NC	ND	6.2	NC	ND	76	**51**	252	137
Sediment	32	**4**	10	4.6	NC	ND	ND	**140**[5]	136	57	**27**	80	69
Sediment	39	**3**	9	ND	NC	ND	ND	NC	ND	38	**43**	168	137
Sediment	65	**8**	26	22	**47**	42	41	NC	ND	31	NC	2378	1,843
Mean X-Met MDL		**6**			**49**			**185**			**39**		

Notes:

[1] Detection limits and concentrations are in milligrams per kilogram (mg/kg), or parts per million (ppm).

[2] MDLs calculated from the 12 MDL sample blends for the X-Met 3000TX in this technology demonstration (in bold typeface for emphasis).

[3] This column lists the mean concentration reported for this MDL sample blend by the X-Met 3000TX.

[4] This column lists the mean concentration reported for this MDL sample blend by the reference laboratory.

[5] To increase the number of calculated MDLs for this element, this blend was included despite the fact that detections were reported by the vendor for only six of the seven replicates. This mean concentration and the corresponding MDL were calculated using the six replicated detected concentrations.

Conc.	Concentration.
MDL	Method detection limit.
NC	The MDL was not calculated because reference laboratory concentrations exceeded five times the expected MDL range (approximately 50 ppm, depending on the element) or an insufficient number of detected concentrations were reported.
ND	One or more results for this blend were reported as “Not Detected.” Except where otherwise noted, blends with one or more ND result as reported by the XRF were not used for calculating the MDL for this element.
Ref. Lab.	Reference laboratory.

7.2 Primary Objective 2 — Accuracy and Comparability

The number of demonstration sample blends that met the criteria for evaluation of accuracy, as described in Section 4.2.2, was low for vanadium (8 blends), but was adequate for the remaining target elements, ranging from 19 (nickel) to 69 (iron). RPDs between the mean concentrations obtained from the X-Met and the reference laboratory were calculated for each blend that met the criteria for an element. Table 7-3 presents the median RPDs, along with the number of RPD results used to calculate the median, for each target element. These statistics are provided for all demonstration samples as well as for subpopulations grouped by medium (soil versus sediment) and concentration level (Levels 1 through 4, as documented in Table 3-1). Additional summary statistics for the RPDs (minimum, maximum, and mean) are provided in Appendix E (Table E-2). In addition, a second set of RPD summary statistics was calculated for mercury, nickel, and selenium using the using the original data set. This set of RPD statistics is presented in Appendix E (Table E-3).

Accuracy was classified as follows for the target elements based on the overall median RPDs in the revised data set:

- Very good (median RPD less than 10 percent): cadmium and selenium.
- Good (median RPD between 10 percent and 25 percent): arsenic, iron, lead, and nickel.
- Fair (median RPD between 25 percent and 50 percent): chromium, copper, mercury, silver, vanadium, and zinc.
- Poor (median RPD greater than 50 percent): antimony.

The median RPD was used for this evaluation because it is less affected by extreme values than is the mean. (The initial evaluation of the RPD populations showed that they were right-skewed or lognormal.) However, the classification of the elements based on accuracy generally stayed the same when the mean rather than the median RPD was used for the evaluation (Table E-2).

The ability to discern effects of sample medium (soil versus sediment) or concentration range on the RPDs was limited by the variability of the data set. Slight increases in median RPDs were observed for a few elements (cadmium, iron, lead, and nickel) in Level 1 soil or sediment. In the case of cadmium, the median RPDs for Level 1 soil appeared to be skewed high by the results for sample Blends 7, 8, and 9, which contained high concentrations of a number of target elements (arsenic, lead, copper, zinc, and iron). For the other three elements, the higher RPDs in the Level 1 blends were more generalized and could not be traced to specific blends.

Section 5.3.3 discussed how the reference laboratory data for antimony were consistently biased low when compared with the ERA-certified spike concentrations. This effect may be caused by volatilization of the antimony compounds used for spiking, resulting in loss of antimony during the sample digestion process at the reference laboratory. Therefore, Table 7-3 includes a second evaluation of accuracy for antimony, comparing the results from the X-Met with the ERA-certified values. As shown, this comparison indicates better performance for antimony than does the comparison to the reference laboratory results; the overall median RPD using the ERA-certified values was 45.3 percent, compared with an overall median RPD of 90.1 percent using the reference laboratory data. By compensating for potential laboratory bias, use of the ERA-certified values therefore improved apparent XRF performance for antimony from “poor” to “fair.”

Table 7-3: Evaluation of Accuracy — Relative Percent Differences versus Reference Laboratory Data for the Oxford X-Met 3000TX

	Sample		Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Matrix	Group	Statistic	Ref Lab	ERA Spike												
Soil	Level 1	Number	4	--	11	6	10	16	4	14	5	1	4	3	0	18
		Median	104.0%	--	27.0%	42.5%	48.1%	38.5%	103.4%	44.7%	45.4%	15.3%	10.6%	19.8%	NC	22.4%
	Level 2	Number	5	1	4	7	4	8	13	4	7	3	5	3	0	6
		Median	92.1%	56.2%	7.3%	12.0%	42.7%	28.6%	12.0%	8.0%	31.8%	27.2%	5.4%	24.0%	NC	21.8%
	Level 3	Number	4	3	4	2	2	2	13	8	2	6	4	7	3	9
		Median	75.8%	35.9%	22.3%	10.6%	15.0%	27.5%	24.0%	8.1%	11.1%	16.6%	4.1%	36.7%	28.7%	29.4%
	Level 4	Number	--	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	--	31.1%	17.3%	--	--	--	--	--	--
	All Soil	Number	13	4	19	15	16	26	37	31	14	10	13	13	3	33
		Median	92.1%	46.0%	20.3%	12.0%	43.9%	31.9%	21.9%	18.8%	30.9%	18.1%	5.6%	24.6%	28.7%	25.2%
Sediment	Level 1	Number	1	1	14	3	1	8	3	14	2	0	5	4	0	19
		Median	155.5%	13.0%	20.1%	8.9%	78.0%	38.5%	134.8%	44.9%	115.7%	NC	6.7%	40.1%	NC	40.0%
	Level 2	Number	3	3	4	4	3	4	19	4	4	6	4	4	2	5
		Median	141.2%	13.0%	24.2%	8.2%	34.1%	27.1%	8.7%	7.5%	64.2%	15.3%	10.4%	23.2%	49.8%	18.3%
	Level 3	Number	3	3	2	3	3	10	4	3	3	4	3	3	3	4
		Median	49.9%	49.8%	30.1%	3.8%	24.5%	6.5%	22.5%	17.2%	43.1%	17.9%	8.5%	60.8%	33.8%	19.1%
	Level 4	Number	--	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	--	12.6%	--	--	--	--	--	--	--
	All Sediment	Number	7	7	20	10	7	22	32	21	9	10	12	11	5	28
		Median	76.2%	45.3%	23.1%	6.1%	30.9%	24.8%	10.2%	31.6%	63.4%	15.3%	7.9%	35.4%	39.5%	28.3%
All Samples	X-Met 3000TX	Number	20	11	39	25	23	48	69	52	23	20	25	24	8	61
		Median	90.1%	45.3%	22.5%	8.9%	36.8%	26.8%	14.4%	23.9%	43.1%	16.0%	6.7%	32.4%	36.6%	25.8%
All Samples	All XRF Instruments	Number	206	110	320	209	338	363	558	392	192	403	195	177	218	471
		Median	84.3%	70.6%	26.2%	16.7%	26.0%	16.2%	26.0%	21.5%	58.6%	25.4%	16.7%	28.7%	38.3%	19.4%

Notes:

All median RPDs presented in this table are based on the population of absolute values of the individual RPDs.

--	No samples reported by the reference laboratory in this concentration range.
ERA	Environmental Resource Associates, Inc.
NC	Not calculated.
Number	Number of samples appropriate for accuracy evaluation.
Ref Lab	Reference laboratory (Shealy Environmental Services, Inc.).
RPD	Relative percent difference.

As an additional comparison, Table 7-3 also presents the overall average of the median RPDs for all eight XRF instruments. Additional summary statistics for the RPDs across all eight XRF instruments are included in Appendix E (Tables E-2 and E-3). Table 7-3 indicates that the median RPDs for the X-Met were equivalent to or below the all-instrument medians for seven of the target elements. Antimony, chromium, copper, lead, silver, and zinc displayed slightly higher median RPDs for the X-Met than for all the demonstration instruments combined.

In addition to calculating RPDs, the evaluation of accuracy included preparing linear correlation plots. These plots are presented for the individual target elements in Figures E-1 through E-13 of Appendix E (these plots include the revised data as well as the original data for mercury, nickel, and selenium). The plots include a 45-degree line showing the "ideal" relationship between the X-Met data and the reference laboratory data, as well as a "best fit" linear equation (y = mx + b, where *m* is the slope of the line and *b* is the y-intercept of the line) and correlation coefficient (r^2) to help illustrate the "actual" relationship between the two methods. To be considered accurate, the correlation coefficient should be greater than 0.9, the slope (m) should be between 0.75 and 1.25, and the y-intercept (b) should be relatively (plus or minus the detection limit) close to zero. Table 7-4 lists the results for these three correlation parameters using X-Met's revised data set. This table shows that the results for cadmium, chromium, iron, nickel, and selenium met all these criteria (these elements are shown in bold typeface). The results for copper and mercury also met these criteria except that the intercept was slightly higher than the MDL. The correlation plot for cadmium is displayed in Figure 7-1 as an example of the correlations obtained for these elements.

Figure 7-1. Linear correlation plot for X-MET 3000TX showing high correlation for cadmium.

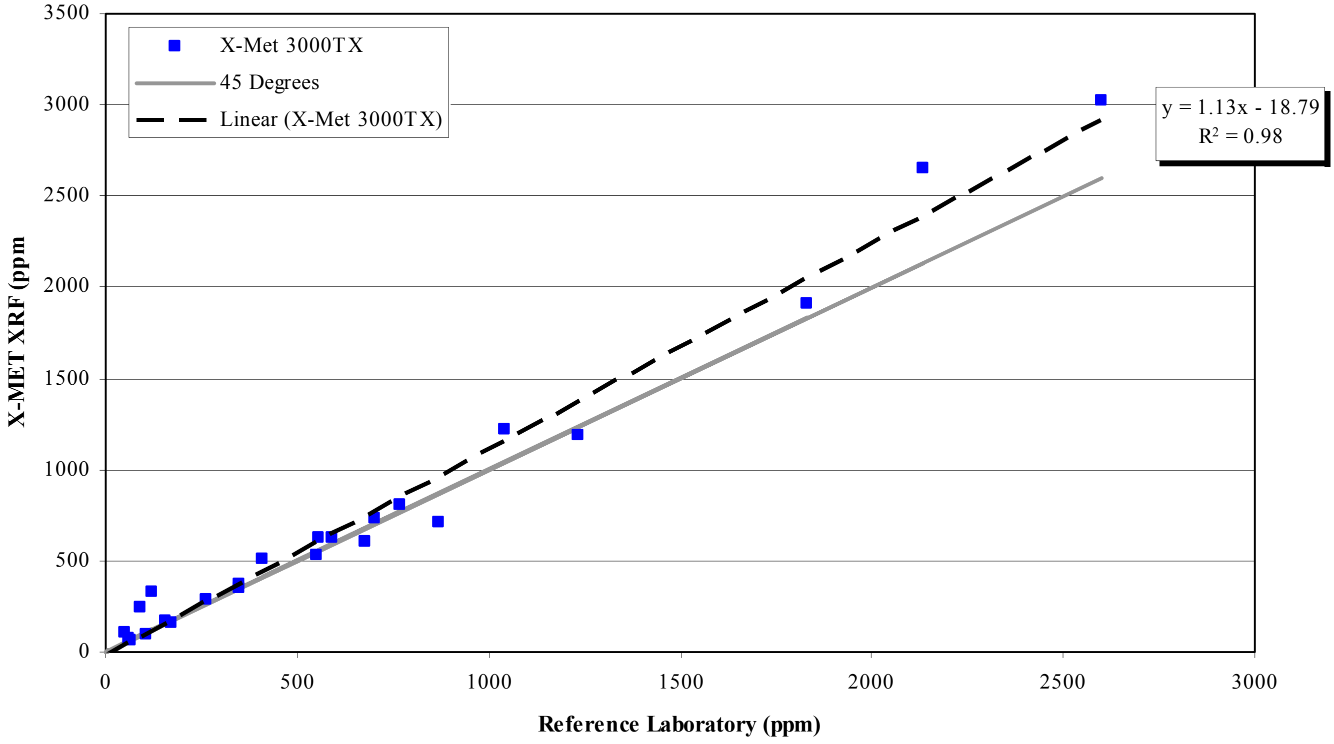


Table 7-4. Summary of Correlation Evaluation for X-Met 3000TX

Target Element	m	b	r^2	Correlation	Bias
Antimony (Ref. Lab) [1]	1.80	193	0.76	Moderate	High
Antimony (Cert. Val.) [1]	0.59	92	0.89	Moderate	Low
Arsenic	1.26	-27	0.99	High	High
Cadmium	**1.13**	**-19**	**0.98**	**High**	**--**
Chromium	**1.23**	**131**	**0.93**	**High**	**--**
Copper	1.14	68	0.95	High	--
Iron	**0.93**	**1098** [2]	**0.94**	**High**	**--**
Lead	1.32	-328 [2]	0.98	High	High
Mercury	0.95	96	0.98	High	--
Nickel	**1.19**	**-66**	**0.97**	**High**	**--**
Selenium	**1.05**	**-0.37**	**0.98**	**High**	**--**
Silver	1.36	26	0.69	Moderate	High
Vanadium	0.97	142	0.41	Low	High
Zinc	1.46	-47	0.98	High	High

Notes:

[1] For antimony, correlation was analyzed for the X-Met versus the reference laboratory data ("Ref. Lab") as well as versus the ERA-certified spike values ("Cert. Val.") for the spiked sample blends.

[2] For iron, no MDL was calculated and the high intercept value was the result of the extreme range of concentrations in the demonstration samples. The extreme range of concentrations in the demonstration samples also affected the intercept for lead.

-- No bias observed.
b Y-intercept of correlation line.
m Slope of correlation line.
r^2 Correlation coefficient of correlation line.

Other general observations from the correlation plots are as follows:

- Mercury exhibited a high r^2 value (0.98). However, this correlation was affected by two extreme Level 4 concentrations (Blends 21 and 22) that were more than three times higher than the next-highest concentrations in the data set for mercury (see Figure E-8). Removing these extreme concentrations from the plots produced a much poorer correlation coefficient in the range of 0.8.

- Large y-intercepts were calculated for lead and iron. Examination of the plots for these elements (Figures E-6 and E-7) reveals that these y-intercepts are small relative to the extreme range of concentrations in the demonstration samples.

- For antimony, a high bias and a low relative degree of correlation (r^2 = 0.76) was observed between the data for the X-Met and the reference laboratory. Table 7-4 and Figure E-1 show a second correlation analysis for antimony, comparing the mean X-Met concentrations for spiked blends with the ERA-certified values. Although a slightly better correlation was observed relative to the ERA-certified values (r^2 = 0.89), a low slope of 0.59 indicates a significant low bias in the X-Met data when compared with these values. This observation was consistent with the RPD evaluation in showing that use of the ERA-certified values produced only moderate improvements in accuracy for antimony.

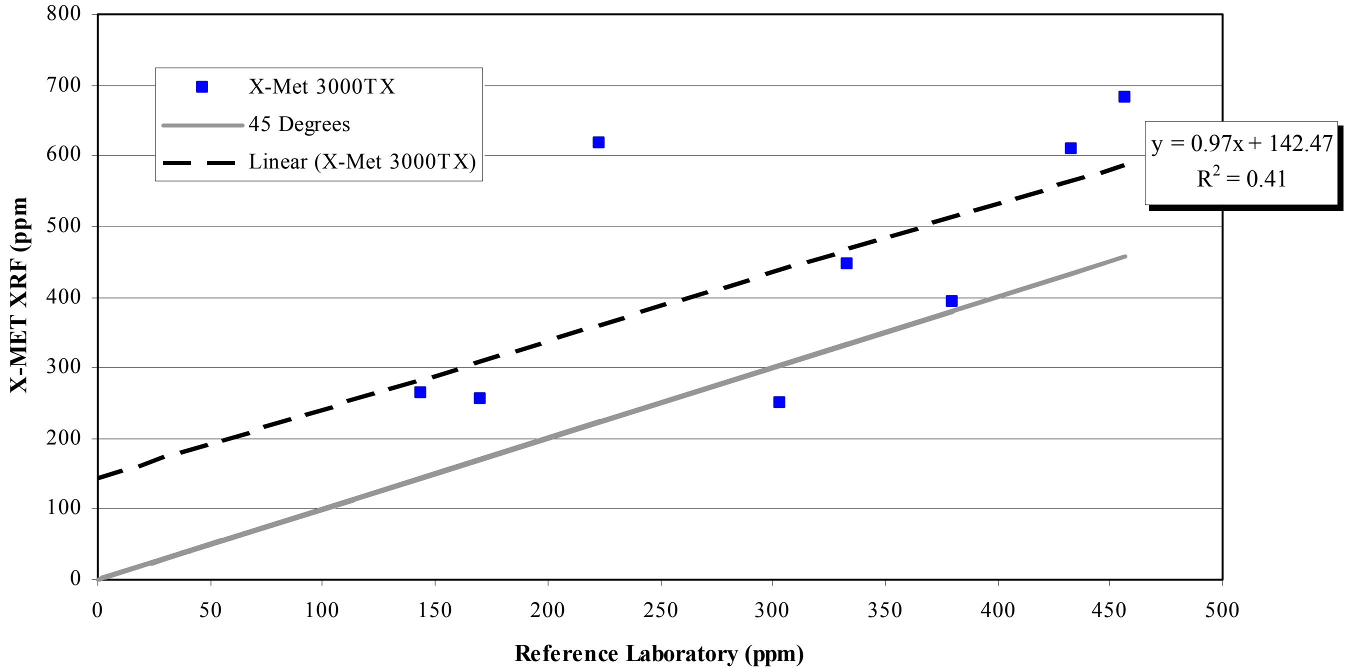
- The lowest degree of correlation between the X-Met and the reference laboratory was observed for vanadium, with an r^2 of 0.41. A high y-intercept further produced a positive bias in the XRF results. This finding agreed with the RPD evaluation, which found poor performance for vanadium. The accuracy evaluation for vanadium may have been affected by the limited number of appropriate sample blends, as only 8 demonstration sample blends met the criteria specified in Section 4.2.2 for inclusion in the accuracy evaluation. The correlation plot for vanadium is displayed in Figure 7-2.

In conclusion, the evaluations of accuracy showed an acceptable overall level of performance by the X-Met for the target elements. Correlations with the reference laboratory were generally high and, for most elements, median RPDs were better than the average of all eight XRF instruments. Oxford's empirical calibration protocol specific to the demonstration may have contributed to the high relative level of accuracy attained. However, the instrument showed poor overall performance for vanadium.

7.3 Primary Objective 3 — Precision

As described in Section 4.2.3, the precision of the X-Met was evaluated by calculating RSDs for the replicate measurements from each sample blend. Median RSDs for the various concentration levels and media (soil and sediment), as well as for the demonstration sample set as a whole, are presented for Oxford's revised data set in Table 7-5. An expanded set of summary statistics for the RSDs (including minimum, maximum, and mean) are provided in Appendix E (Table E-4). Table E-5 of Appendix E provides an additional set of RSD statistics for mercury, nickel, and selenium based on the original data set.

Figure 7-2. Linear correlation plot for X-MET 3000TX showing low correlation and limited data set for vanadium.



The RSD calculation found a high level of precision for the X-Met in that the demonstration-wide median RSDs for the full sample set were below 15 percent for all target elements. The ranges into which the median RSDs fell are summarized below:

- Very low (median RSD between 0 and 5 percent): cadmium, iron, mercury, and selenium.
- Low (median RSD between 5 and 10 percent): arsenic, copper, lead, nickel, silver, and zinc.
- Moderate (median RSD between 10 and 20 percent): antimony, chromium, and vanadium.
- High (median RSD greater than 20 percent): none.

The median RSDs for the soil and sediment subsets were also less than 15 percent with the exception of vanadium in soil (19.8 percent). The median RSDs for soil were slightly larger for some target elements (antimony, chromium, mercury, and vanadium) than the median RSDs for sediment, but this observation may reflect the larger numbers of soil samples included in the precision evaluation.

The high overall level of precision may have been facilitated by the level of processing (homogenizing, sieving, crushing, and drying) on the sample blends before the demonstration (Chapter 3). This observation is consistent with the previous SITE MMT program demonstration of XRF technologies that occurred in 1995 (EPA 1996a, 1996b, 1998a, 1998b, 1998c, and 1998d). The high level of sample processing applied during both XRF technology demonstrations was necessary to minimize the effects of sample heterogeneity on the demonstration results and on comparability with the reference laboratories. During project design, site investigation teams that intend to compare XRF and laboratory data should similarly assess the need for sample processing steps to manage sample heterogeneity and improve data comparability.

Further review of the median RSDs in Table 7-5 based on concentration range reveals slightly higher RSDs (in other words, lower precision) for the target elements in Level 1 samples when compared with the rest of the data set. This effect was observed for multiple target elements in both soil and sediment, but the effect was difficult to further assess due to the limited numbers of samples available (less than 3 sample blends in the Level 1 concentration range were appropriate for precision evaluation in soil or sediment for some of the elements). However, this observation indicates that, to a minor extent, analytical precision for the X-Met results may depend on concentration.

As an additional comparison, Table 7-5 also presents the overall average of the median RSDs calculated for all XRF instruments that participated in the demonstration. Additional summary statistics for the RSDs calculated across all eight XRF instruments are included in Table E-4. Table 7-5 indicates that the median RSDs for the X-Met were slightly above the all-instrument medians for seven of the 13 target elements (antimony, cadmium, copper, lead, silver, vanadium, and zinc).

Table 7-6 presents median RSD statistics for the reference laboratory and compares these to the summary data for the X-Met. (Additional summary statistics are provided in Table E-6 of Appendix E.) Table 7-6 indicates that the median RSDs for the X-Met were lower than the RSDs for the reference laboratory for 9 of the 13 target elements; exceptions included antimony, chromium, silver, and vanadium, which were slightly higher. However, the overall average RSDs for all eight XRF instruments were equivalent to or lower than the reference laboratory RSDs for 11 of the 13 target elements (the exceptions were chromium and vanadium).

Table 7-5. Evaluation of Precision — Relative Standard Deviations for the Oxford X-Met 3000TX

Matrix	Sample Group	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	4	11	6	10	16	4	14	5	1	4	3	0	18
		Median	18.2%	8.2%	8.7%	12.9%	7.9%	1.7%	9.5%	9.8%	15.7%	8.6%	12.3%	NC	8.4%
	Level 2	Number	5	4	7	4	8	13	4	7	3	5	3	0	6
		Median	10.2%	5.2%	2.2%	7.9%	6.0%	2.7%	5.2%	3.8%	9.4%	1.2%	9.7%	NC	3.3%
	Level 3	Number	4	4	2	2	2	13	8	2	7	4	7	3	9
		Median	10.9%	1.1%	3.0%	3.0%	6.3%	1.6%	3.2%	3.1%	3.0%	1.6%	5.7%	19.8%	4.7%
	Level 4	Number	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	1.7%	3.6%	--	--	--	--	--	--
	All Soil	Number	13	19	15	16	26	37	31	14	11	13	13	3	33
		Median	12.2%	5.5%	4.6%	10.9%	7.1%	1.9%	5.3%	4.1%	5.7%	1.8%	9.4%	19.8%	6.6%
Sediment	Level 1	Number	1	14	3	1	8	3	14	2	0	5	4	0	19
		Median	4.8%	9.1%	5.2%	28.0%	9.3%	1.1%	10.8%	8.7%	NC	5.6%	16.3%	NC	6.0%
	Level 2	Number	3	4	4	3	4	19	4	4	6	4	4	2	5
		Median	11.0%	3.2%	4.2%	4.9%	7.3%	2.4%	2.7%	3.2%	12.3%	4.2%	6.8%	11.0%	7.4%
	Level 3	Number	3	2	3	3	10	4	3	3	3	3	3	3	4
		Median	7.2%	4.7%	1.5%	5.5%	2.7%	2.9%	0.6%	3.2%	4.0%	3.2%	3.0%	9.4%	4.7%
	Level 4	Number	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	1.4%	--	--	--	--	--	--	--
	All Sediment	Number	7	20	10	7	22	32	21	9	9	12	11	5	28
		Median	7.2%	7.5%	3.9%	5.5%	5.5%	1.9%	6.4%	3.2%	6.8%	4.9%	8.7%	9.4%	5.7%
All Samples	X-Met 3000TX	Number	20	39	25	23	48	69	52	23	20	25	24	8	61
		Median	11.4%	5.8%	4.6%	10.6%	6.4%	1.9%	5.7%	3.8%	5.8%	3.2%	9.1%	12.6%	6.0%
All Samples	All XRF Instruments	Number	206	320	209	338	363	558	392	192	403	195	177	218	471
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%	7.0%	4.5%	5.2%	8.5%	5.3%

Notes:

--	No samples reported by the reference laboratory in this concentration range.
Number	Number of samples appropriate for precision evaluation.
RSD	Relative standard deviation.

Table 7-6. Evaluation of Precision – Relative Standard Deviations for the Reference Laboratory versus the X-Met 3000TX and All Demonstration Instruments

Matrix	Sample Group	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Ref. Lab	Number	17	23	15	34	26	38	33	16	35	13	13	21	35
		Median	9.8%	12.4%	9.0%	10.6%	9.1%	8.7%	13.2%	6.6%	10.0%	7.1%	7.5%	6.6%	9.1%
Sediment	Ref. Lab	Number	7	24	10	26	21	31	22	10	27	12	10	17	27
		Median	9.1%	9.2%	8.2%	7.5%	8.9%	8.1%	7.4%	6.9%	7.3%	7.6%	6.6%	8.1%	6.9%
All Samples	Ref. Lab	Number	24	47	25	60	47	69	55	26	62	25	23	38	62
		Median	9.5%	9.5%	9.0%	8.4%	8.9%	8.5%	8.6%	6.6%	8.2%	7.4%	7.1%	7.2%	7.4%
All Samples	X-Met 3000TX	Number	20	39	25	23	48	69	52	23	20	25	24	8	61
		Median	11.4%	5.8%	4.6%	10.6%	6.4%	1.9%	5.7%	3.8%	5.8%	3.2%	9.1%	12.6%	6.0%
All Samples	All Instruments	Number	206	320	209	338	363	558	392	192	403	195	177	218	471
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%	7.0%	4.5%	5.2%	8.5%	5.3%

Notes:

Number	Number of samples meeting criteria for precision evaluation (Section (4.2.3).
Ref. Lab	Reference Laboratory.
RSD	Relative standard deviation.

7.4 Primary Objective 4 — Impact of Chemical and Spectral Interferences

The RPD data from the accuracy evaluation were further processed to assess the effects of interferences. The RPD data for elements considered susceptible to interferences were grouped and compared based on the relative concentrations of potentially interfering elements. Of specific interest for the comparison were the potential effects of:

- High concentrations of lead on the RPDs for arsenic
- High concentrations of nickel on the RPDs for copper (and vice versa), and
- High concentrations of zinc on RPDs for copper (and vice versa).

The rationale and approach for evaluation of these interferents are described in Section 4.2.4.

Interferent-to-element ratios were calculated using the mean concentrations the reference laboratory reported for each blend, classified as low (less than 5X), moderate (5 to 10X), or high (greater than 10X). Table 7-7 presents median RPD data for arsenic, nickel, copper, and zinc that are grouped based on this classification scheme. Additional summary statistics are presented in Appendix E (Table E-7), along with another set of summary statistics Oxford's original nickel data set (Table E-8). The tables imply possible interference effects in all of the elements studied. Specifically, high relative concentrations (greater than 10X) of the potential interfering elements invariably correlated with an increase in median RPD (and thus reduced accuracy) for arsenic, nickel, copper, and zinc. For all four elements, increasing concentrations of interferences raised the median RPD from the "good" (less than 25 percent) to the "fair" or "poor" categories (approaching 50% or more). An additional set of summary statistics in Table E-8 indicates that similar interference effects are still observed in the revised data set for the two elements affected by the revisions (nickel and copper).

In presenting statistics for the raw RPDs as well as the absolute values of the RPDs, Table E-7 further shows that the interfering elements appeared to produce an increasing negative bias in the arsenic data (as indicated by larger raw RPDs), and an increasingly positive bias in the data for copper, nickel, and zinc (as indicated by more negative raw RPDs). These interference effects were observed despite the fact that Oxford developed an empirical calibration for the X-Met specifically for use in the field demonstration. The findings for this objective indicate that Oxford's calibration may not adequately address potential interferences, and may require further refinement for analysis of samples containing high element concentrations. On the other hand, the low numbers of samples available for some interferent-to-element ratios (three samples or less; see Table 7-7) creates some uncertainty in the interference evaluation.

7.5 Primary Objective 5 — Effects of Soil Characteristics

The population of RPDs between the results obtained from the X-Met and the reference laboratory was further evaluated against sampling site and soil type. Separate sets of summary statistics were developed for the mean RPDs associated with each sampling site for comparison to the other sites and to the data set for all samples. The site-specific median RPDs are presented in Table 7-8 for the revised X-Met data set, along with descriptions of soil or sediment type from observations during sampling at each site. Additional RPD summary statistics for each soil type (minimum, maximum, and mean) are presented in Table E-9 of Appendix E, and a separate set of summary statistics for the original data set for mercury, nickel, and selenium is presented in Table E-10).

Another perspective on the effects of soil type was developed by graphically assessing outliers and extreme values in the RPD data sets for each target element. This evaluation focused on correlating these values with sample types or locations for multiple elements across the data set. Outliers and extreme values are apparent in the correlation plots (Figures E-1 through E-13) and are further depicted for the various elements on box and whisker plots in Figure E-14.

Table 7-7. Effects of Interferent Elements on the RPDs (Accuracy) for Target Elements, Oxford X-Met 3000TX[1]

Parameter	**Lead Effects on Arsenic**			**Copper Effects on Nickel**			**Nickel Effects on Copper**			**Zinc Effects on Copper**			**Copper Effects on Zinc**		
Interferent/ Element Ratio	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10
Number of Samples	29	7	3	16	1	2	40	0	8	35	2	11	48	3	10
Median RPD of Target Element[2]	22.5%	9.8%	80.9%	19.5%	14.0%	68.4%	21.5%	NC	65.1%	24.1%	25.4%	44.9%	21.3%	63.8%	55.1%
Median Interferent Concentration	86	7946	2944	164	2418	4501	249	NC	1805	259	5903	3969	238	1402	2436
Median Target Element Concentration	140	941	41	365	206	473	1022	NC	225	1293	1161	150	825	220	236

Notes:

[1] Concentrations are reported in units of milligrams per kilogram (mg/kg), or parts per million (ppm).

[2] All median RPDs presented in this table are based on the population of absolute values of the individual RPDs.

< Less than.

> Greater than.

RPD Relative percent difference.

Table 7-8. Effect of Soil Type on the RPDs (Accuracy) for Target Elements, Oxford X-Met 3000TX

Matrix	Site	Matrix Description	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	2	2	3	3	3
			Median	--	--	13.1%	30.0%	18.8%	31.1%	9.3%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	6	5	3	6	7	6
			Median	117.6%	7.5%	6.3%	35.9%	22.0%	9.4%	7.5%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	2	1	3	3	3
			Median	44.9%	44.0%	7.1%	71.7%	32.1%	15.5%	59.3%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	1	--	--	1	2	5	6
			Median	1.8%	--	--	40.7%	36.3%	130.1%	12.1%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	3	9	5	4	4	12	6
			Median	65.8%	17.7%	8.9%	43.5%	38.6%	13.4%	59.0%
Sediment	RF	Silty fine sand (tailings)	Number	2	11	5	4	13	13	11
			Median	53.8%	23.6%	4.3%	27.4%	35.6%	5.9%	22.1%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	4	5	1	5	4	12	6
			Median	90.1%	27.0%	14.9%	36.8%	32.8%	24.5%	60.2%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	1	2	1	7	7	4
			Median	155.5%	116.4%	6.1%	78.0%	4.3%	12.8%	26.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	2	6	3	2	6	7	7
			Median	105.4%	27.6%	89.8%	92.9%	37.0%	8.4%	25.1%
	All		Number	20	39	25	23	48	69	52
			Median	90.1%	22.5%	8.9%	36.8%	26.8%	14.4%	23.9%

Table 7-8. Effect of Soil Type on RPDs (Accuracy) for Target Elements, Oxford X-Met 3000TX (Continued)

Matrix	Site	Matrix Description	Statistic	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	1	1	--	3
			Median	--	--	0.8%	36.7%	--	41.1%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	--	2	4	4	1	7
			Median	--	12.0%	10.6%	28.0%	28.7%	26.8%
Soil	CN	Sandy loam (burn pit residue)	Number	2	1	2	2	--	3
			Median	26.2%	27.7%	17.4%	13.0%	--	22.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	1	--	--	--	1
			Median	--	15.3%	--	--	--	69.7%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	4	6	5	4	4	10
			Median	58.4%	21.7%	9.2%	34.4%	40.1%	30.7%
Sediment	RF	Silty fine sand (tailings)	Number	5	6	5	4	2	13
			Median	79.6%	15.3%	8.5%	48.0%	31.1%	20.5%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	10	2	3	1	--	10
			Median	26.4%	7.4%	2.6%	86.0%	--	14.1%
Sediment	TL	Silt and clay (slag-enriched)	Number	2	--	4	4	--	7
			Median	41.0%	--	10.4%	27.9%	--	40.0%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	2	1	4	1	7
			Median	--	21.0%	5.6%	27.0%	19.6%	33.7%
	All		Number	23	19	24	24	8	61
			Median	79.6%	22.3%	7.9%	32.4%	36.6%	25.8%

Notes:

AS	Alton Steel Mill
BN	Burlington Northern railroad/ASARCO East.
CN	Naval Surface Warfare Center, Crane Division.
KP	KARS Park – Kennedy Space Center.
LV	Leviathan Mine/Aspen Creek.
RF	Ramsey Flats – Silver Bow Creek.
SB	Sulphur Bank Mercury Mine.
TL	Torch Lake Superfund Site.
WS	Wickes Smelter Site.

Other Notes:

--	No samples reported by the reference laboratory in this concentration range.
Number	Number of demonstration samples evaluated.
RPD	Relative percent difference.

Review of Table 7-8 indicates that the median RPDs were highly variable and that trends or differences between sample sites were difficult to discern. Evaluations relative to sampling site were further complicated by the low numbers of samples for many target elements. (Table 7-8 indicates that only 1 to 3 samples were available from many sampling sites for evaluation of specific target elements.) Low relative accuracy was observed for cadmium in blends from the Wickes Smelter site. The median RPD for cadmium in these blends was 89.8 percent compared to a maximum of 14.9 percent in the other blends. The soil matrix from this site was described during the demonstration sample collection program (Chapter 2) as roaster slag, consisting of a black, fairly coarse sand and gravel material. This slag is an intermediate product in processing ore, wherein volatile sulfide materials are thermally removed, leaving concentrated heavy elements. Effects of the Wickes Smelter sample blends on XRF data quality were noted earlier for cadmium and other elements in the MDL and accuracy evaluations (Sections 7.1 and 7.2). Review of the box and whisker plot (Figure E-14) further shows that specific high outliers and extreme values were observed in Wickes Smelter samples (Blends 6, 7, 9, and 52) for arsenic, cadmium, and chromium.

Further review of Table 7-8, Figure E-14, and the correlation plots from the accuracy evaluation revealed that a number of high RPD outliers for iron were from the KARS Park site (Blends 1 through 4, 27, and 28). As discussed in Section 2.3, the KARS Park site was contaminated by former gun range operations. A few high RPD outliers were also observed for multiple elements in samples from the Leviathan Mine site (Blends 36, 54, and 55). Chapter 2 indicates that the matrixes from Leviathan Mine were clay soils that also included precipitates and solids from acid mine leachate and wastewater retention ponds. Many of these blends contained extreme concentrations of iron (in the range of 100,000 to 250,000 ppm).

Overall, the evaluation found that sample matrix had a minor effect on the overall accuracy of the XRF data given that the range of RPDs observed for the target elements was very broad. The spread in the accuracy results is illustrated on the box and whisker plot in Figure E-14. The plot shows the broad distributions of RPDs for many elements, and illustrates that no outliers or extreme values were identified for antimony, mercury, nickel, or selenium.

7.6 Primary Objective 6 — Sample Throughput

The Oxford two-person field team was able to analyze all 326 demonstration samples in 4 days at the demonstration site. Once the X-Met instrument had been set up and operations had been streamlined, the Oxford field team was able to analyze a maximum of 127 samples during an extended work day. This sample throughput was achieved by using the two members of the field team to separately perform sample analysis and data reduction. Without an extended work day, it was estimated that the Oxford field team would have averaged about 72 samples per day.

This estimated sample throughput for a normal working day was slightly higher than the average for all eight instruments that participated in the demonstration (66 samples per day). The higher sample throughput was primarily the result of the lower-than-average times required to complete each analytical step. A detailed discussion of the time required to complete the various steps of sample analysis using the X-Met is included as part of the labor cost analysis in Section 8.3.

7.7 Primary Objective 7 — Technology Costs

The evaluations pertaining to this primary objective are described in Chapter 8, Economic Analysis.

7.8 Secondary Objective 1 — Training Requirements

Technology users must be suitably trained to set up and operate the instrument to obtain the level of data quality required for specific projects. The amount of training required depends on the configuration and complexity of the instrument, along with the associated software.

Oxford recommends that the X-Met operator have a high school diploma and basic on-site operational training. Field or laboratory technicians are generally qualified to operate the X-Met. One Oxford staff member who operated the instrument during the demonstration held a Ph.D. in analytical chemistry, while the other was a degreed engineer. Both individuals had multiple years of experience in operating the X-Met or similar analyzers. The skill level of these operators was higher than is required to operate the X-Met.

Oxford has not established written standard operating procedures (SOPs) for the preparation or analysis of soil or sediment samples using the X-Met. However, the instrument is accompanied by a clear and detailed operating manual that presents the general steps in analyzing soil and other environmental media. Instrument software is also helpful in directing users with intuitive operating menus. Oxford and its distributors offer on-site training and telephone support to instrument users on an informal, as needed basis.

In addition to the general instrument operational instruction and training, the operator and data manager must be familiar with using a PDA and a personal computer (PC) to acquire and manage analytical data obtained from the instrument. Oxford has designed the analyzer around an HP iPAQ Pocket PC. The iPAQ can store a minimum of 10,000 tests with spectra with its 64 MB memory. The iPAQ features a color, high-resolution display with variable backlighting. The data can be transferred from the iPAQ to a personal computer (PC) by inserting the flash card into the PC, where it will appear as an additional removable disk drive or by using Microsoft ActiveSync® software over a USB cable. The iPAQ can be fitted with Bluetooth© wireless printing and data downloading accessories for wireless data and file transfer, thereby maximizing the efficiency of data transfer while eliminating transcription errors.

7.9 Secondary Objective 2 — Health and Safety

Included in the health and safety evaluation were the potential risks from: (1) potential radiation hazards from the instrument itself, and (2) exposure to any reagents used in preparing and analyzing the samples. However, the evaluation did not include potential risks from exposure to site-specific hazardous materials, such as sample contaminants, or to physical safety hazards. These factors were excluded because of the wide and unpredictable range of sites and conditions that could be encountered in the field during an actual project application of the instrument.

The X-Met contains a miniature silver anode x-ray tube. Each instrument is equipped with a fail-safe status lighting system and locking mechanism to manually control tube operation and analysis. However, the developer reports that risks from exposure to radiation are minimal; direct exposure to the x-rays generated by the instrument for the entire life of the battery would not cause limits on radiation exposure to be exceeded.

The second potential source of risk to XRF instrument operators is exposure to reagent chemicals used in sample preparation. However, for the X-Met, there are no risks from this source because no chemical reagents are required for sample preparation.

7.10 Secondary Objective 3 — Portability

Portability depends on the size, weight, number of components, and power requirements of the instrument, and the reagents required. The size of the instrument, including physical dimensions and weight, is presented in Table 6-1. The number of components, power requirements, support structures, and reagent requirements are also listed in Table 6-1. Two distinctions were made during the demonstration regarding portability:

(1) The instrument was considered fully portable if the dimensions were such that the instrument could be easily brought directly to the sample location by one person.

(2) The instrument was considered transportable if the dimensions and power requirements were such that the instrument could be moved to a location near the sampling location, but required a larger and more stable environment (for example, a site trailer with AC power and stable conditions).

Based on its dimensions and power requirements, the X-Met is defined as fully portable. It is a hand-held unit that can be carried directly to the sampling location for analysis of samples. The X-Met is suitable for all types of field analysis, ranging from "point-and-shoot" readings on undisturbed soil surfaces to processed soil samples in plastic bags or sample cups. With the instrument stand, the X-Met can also be used in a hands-free, bench-top mode.

This instrument stand was used during the demonstration.

7.11 Secondary Objective 4 — Durability

Durability was evaluated by gathering information on the instrument's warranty and the expected lifespan of the radioactive source or x-ray tube. The ability to upgrade software or hardware also was evaluated. Weather resistance was evaluated by examining the instrument for exposed electrical connections and openings that may allow water to penetrate (for portable instruments only).

The outer construction of the X-Met consists of a high-density plastic and metal shell, which is weatherproof and impact resistant. The iPAQ operating system is attached via USB port and cable. This connection to the iPAQ allows the operating system to be contained in a remote weather-protected area, thereby reducing the possibility of being compromised by water or dust. Oxford also provides a protective cover for the X-Met to reduce exposure to weather and harmful ambient conditions. However, this mode of operation was not assessed during the demonstration.

Oxford provides a 2-year limited warranty for the X-Met instrument and a 5-year warranty for the x-ray tube. Since x-ray tube sources are new to the world of portable instrumentation, no clear data have been obtained on the useful life that can be assumed. The average lifespan of an x-ray tube in a traditional bench-top device is 3 to 5 years. Therefore, it is generally assumed among developers of portable XRF instrumentation that the useful life of an x-ray tube in these systems will be about 3 to 5 years. The use of a commercially available iPAQ PDA and associated Windows-based Pocket PC software allows for easy upgrades and updates of instrument software and hardware.

7.12 Secondary Objective 5 — Availability

The X-Met is available for purchase or rental from a nationwide network of distributors, many of which can provide on-site training. The instrument can be repaired, maintained, and calibrated by the distributors or at the factory in Finland. Oxford also operates a telephone helpline from 8:00 a.m. to 5:00 p.m. central time, Monday through Friday.

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Chapter 8
Economic Analysis

This chapter provides cost information for the Oxford Instruments X-Met 3000TX Series XRF analyzer. Cost elements that were addressed included instrument purchase or rental, supplies, labor, and ancillary items. Sources of cost information included input from the technology developer and suppliers as well as observations during the field demonstration. Comparisons are provided to average costs for other XRF technologies and for conventional fixed-laboratory analysis to provide some perspective on the relative cost of using the X-Met.

8.1 Equipment Costs

Capital equipment costs include either purchase or rental of the X-Met and any ancillary equipment that is generally needed for sample analysis. Information on purchase price and rental cost for the analyzer and accessories was obtained from Oxford Instruments.

The X-Met used at the demonstration costs approximately $30,000. The cost includes peripherals such as the instrument stand, sample tray, and 110-volt adapter. The use of a laptop computer is also recommended to manipulate the data. The instrument is available for rental by Oxford. Purchased models include a 2-year warranty for the instrument and 5 years for the x-ray tube. The lifespan of this type of x-ray tube is about 2 to 5 years for normal instrument usage.

The purchase price, rental cost, and shipping cost for the X-Met compares favorably with the average costs for all XRF instruments that participated in the demonstration, as shown in Table 8-1. Purchase of the instrument could be justified as more cost effective than rental only for field activities that involve more than about 3 months of total field analysis time.

8.2 Supply Costs

The supplies that were included in the cost estimate include sample containers, Mylar® film, spatulas or scoops, wipes, and disposable gloves. The rate of consumption for these supplies was based on observations during the field demonstration. Unit prices for these supplies were based on price quotes from independent vendors of field equipment. The X-Met was operated for 4 days to complete the analysis of all 326 samples during the field demonstration. The supplies required to process samples were similar for all XRF instruments that participated in the demonstration and were estimated to cost about $245 for 326 samples or $0.75 per sample.

Table 8-1. Equipment Costs

Cost Element	X-Met 3000TX	XRF Demonstration Average [1]
Shipping	$200	$410
Capital Cost (Purchase)	$30,000	$54,300
Weekly Rental	$2,000	$2, 813
Autosampler (for Overnight Analysis)	N/A	N/A

Notes:
[1] Average for all eight instruments in the demonstration
N/A Not available or not applicable for this comparison

8.3 Labor Costs

Labor costs were estimated based on the total time required by the field team to complete the analysis of all 326 samples and the number of people in the field team, while making allowances for field team members that had responsibilities other than sample processing during the demonstration. For example, some developers sent sales representatives to the demonstration to communicate with visitors and provide outreach services; this type of staff time was not included in the labor cost analysis.

While overall labor costs were based on the total time required to process samples, the time required to complete each definable activity was also measured during the field demonstration. These activities included:

- Initial setup and calibration.
- Sample preparation.
- Sample analysis.
- Daily shutdown and startup.
- End of project packing.

The "total processing time per sample" was calculated as the sum of all these activities assuming that the activities were conducted sequentially; therefore, it represents how much time it would take a single trained analyst to complete these activities. However, the "total processing time per sample" does not include activities that were less definable in terms of the amount of time taken, such as data management and procurement of supplies, and is therefore not a true total.

The time to complete each activity using the X-Met is compared with the average of all XRF instruments in Table 8-2 and is compared with the range of all XRF instruments in Figure 8-1. The X-Met compared favorably against the other XRF instruments, exhibiting lower-than-average times for all activities except daily shutdown and startup.

Table 8-2. Time Required to Complete Analytical Activities[1]

Activity	X-Met 3000TX	Average[2]
Initial Setup and Calibration	8	54
Sample Preparation	2.0	3.1
Sample Analysis	5.0	6.7
Daily Shutdown/Startup	20	10
End of Project Packing	7	43
Total Processing Time per Sample	7.1	10.0

Notes:
[1] All estimates are in minutes
[2] Average for all eight XRF instruments in the demonstration

The Oxford field team expended about 36 hours to complete all sample processing activities during the field demonstration using the X-met. This was significantly lower than the overall average of 69 hours for all instruments that participated in the demonstration. However, both the total processing time per sample and the labor hours for the X-Met were in the middle of the range for portable instruments.

8.4 Comparison of XRF Analysis and Reference Laboratory Costs

Two scenarios were evaluated to compare the cost for XRF analysis using the X-Met with the cost of fixed-laboratory analysis using the reference methods. Both scenarios assumed that 326 samples were to be analyzed, as in the field demonstration. The first scenario assumed that only one element was to be measured in a metal-specific project or application (for example, lead in soil, paint, or other solids) for comparison to laboratory per-metal unit costs. The second scenario assumed that 13 elements were to be analyzed, as in the field demonstration, for comparison to laboratory costs for a full suite of metals.

Typical unit costs for fixed-laboratory analysis using the reference methods were estimated using average costs from Tetra Tech's basic ordering agreement with six national laboratories. These unit costs assume a standard turnaround time of 21 days and standard hard copy and electronic data deliverables that summarize results and raw analytical data. No costs were included for field labor that would be specifically associated with off-site fixed laboratory analysis, such as sample packaging and shipment.

The cost for XRF analysis using the X-Met was based on equipment rental for 1 week, along with labor and supplies estimates established during the field demonstration. Labor costs were added for drying, grinding, and homogenizing the samples (estimated at 10 minutes per sample) since these additional steps in sample preparation are required for XRF analysis but not for analysis in a fixed laboratory.

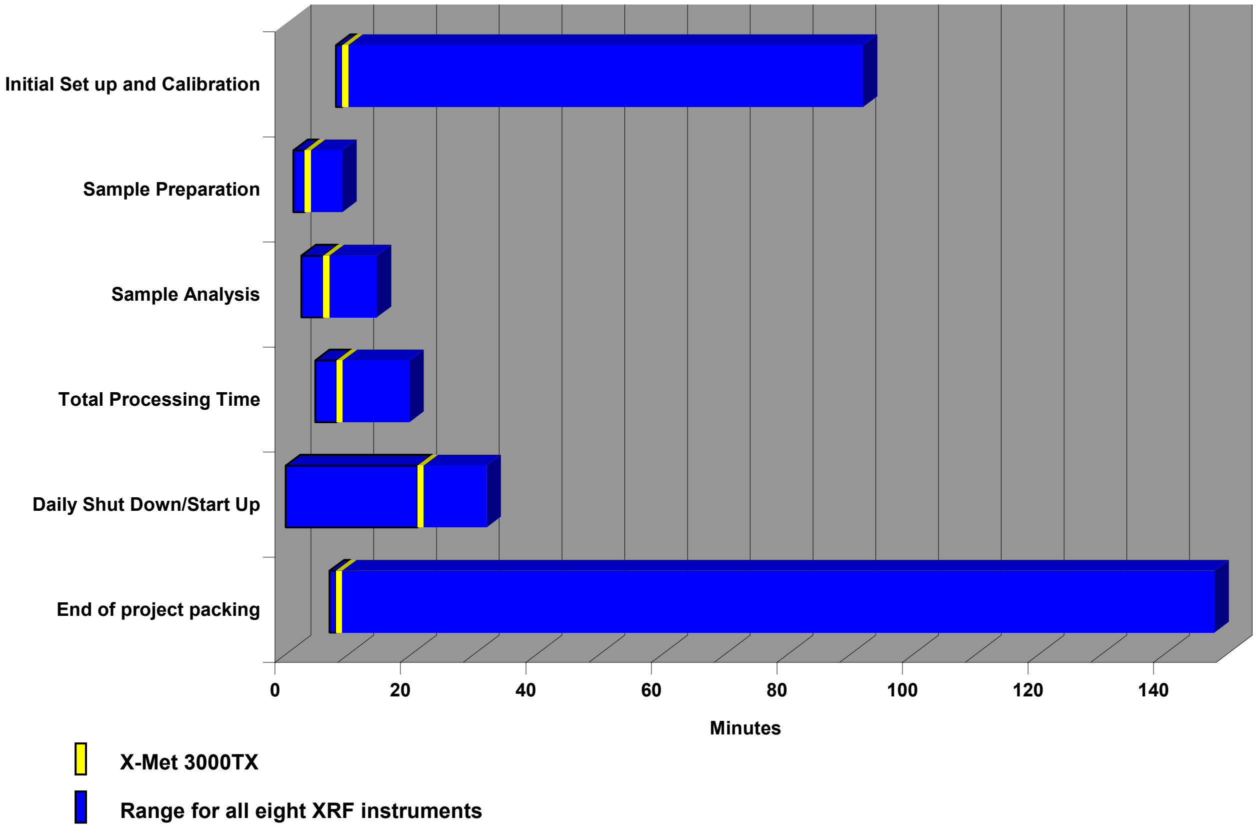

Figure 8-1. Comparison of activity times for the X-Met 3000TX versus other XRF instruments.

A typical cost for managing investigation-derived waste (IDW), including general trash, personal protective equipment, wipes, and soil, was also added to the cost of XRF analysis because IDW costs are included in the unit cost for fixed-laboratory analysis. The IDW management cost was fixed, based on the average IDW disposal cost per instrument during the demonstration, because IDW generation did not vary significantly between instruments during the demonstration. Since the cost for XRF analysis of one element or multiple elements does not vary significantly (all target elements are determined simultaneously when a sample is analyzed), the XRF analysis cost was not adjusted for one element versus 13 elements.

Table 8-3 summarizes the costs for the X-Met versus the cost for analysis in a fixed laboratory. This comparison shows that the X-Met compares favorably to a fixed laboratory in terms of overall cost, particularly when a large number of elements are to be determined. Use of the X-Met will likely produce additional cost savings because analytical results will be available within a few hours after samples are collected, thereby expediting project decisions and reducing or eliminating the need for additional mobilizations.

The total cost for the X-Met in the example scenario (326 samples) was estimated at $7,288, whether one or a number of elements was analyzed. This estimate is less than the average of $8,932 for all XRF instruments that participated in the demonstration. However, it should be noted that bench-top instruments, which typically cost more than hand-held instruments like the X-Met, were included in the calculation of the average cost for all XRF instruments. In comparison to other hand-held XRF instruments, the X-Met cost for the example scenario was similar.

Table 8-3. Comparison of XRF Technology and Reference Method Costs

Analytical Approach	Quantity	Item	Unit Rate	Total
X-Met 3000TX (1 to 13 elements)				
Shipping	1	Roundtrip	$200	$200
Weekly Rental	1	Week	$2,000	$2,000
Supplies	326	Sample	$0.75	$245
Labor	109	Hours	$43.75	$4,753
IDW	N/A	N/A	N/A	$90
Total X-Met 3000TX Analysis Cost (1 to 13 elements)				**$7,288**
Fixed Laboratory (1 element)				
(EPA Method 6010, ICP-AES)	326	Sample	$21	$6,846
Total Fixed Laboratory Costs (1 element)				**$6,846**
Fixed Laboratory (13 elements)				
Mercury (EPA Method 7471, CVAA)	326	Sample	$36	$11,736
All other Elements (EPA Method 6010, ICP-AES)	326	Sample	$160	$52,160
Total Fixed Laboratory Costs (13 elements)				**$63,896**

Chapter 9
Summary of Technology Performance

The preceding chapters of this report document that the evaluation design succeeded in providing detailed performance data for the Oxford X-Met 3000TX XRF analyzer. The evaluation design incorporated 13 target elements, 70 distinct sample blends, and a total of 326 samples. The blends included both soil and sediment samples from nine sampling locations. A rigorous program of sample preparation and characterization, reference laboratory analysis, QA/QC oversight, and data reduction supported the evaluation of XRF instrument performance.

One important aspect of the demonstration was the sample blending and processing procedures (including drying, sieving, grinding, and homogenization) that significantly reduced uncertainties associated with the demonstration sample set. These procedures minimized the impacts of heterogeneity on method precision and on the comparability between XRF data and reference laboratory data. In like manner, project teams are encouraged to assess the effects of sampling uncertainty on data quality and to adopt appropriate sample preparation protocols before XRF is used for large-scale data collection, particularly if the project will involve comparisons to other methods (such as off-site laboratories). An initial pilot-scale method evaluation, carried out in cooperation with an instrument vendor, can yield site-specific standard operating procedures for sample preparation and analysis to ensure that the XRF method will meet data quality needs, such as accuracy and sensitivity requirements. A pilot study can also help the project team develop an initial understanding of the degree of correlation between field and laboratory data. This type of study is especially appropriate for sampling programs that will involve complex soil or sediment matrices with high concentrations of multiple elements because the demonstration found that XRF performance was more variable under these conditions. Initial pilot studies can also be used to develop site-specific calibrations, in accordance with EPA Method 6200, that adjust instrument algorithms to compensate for matrix effects.

The findings of the evaluation of the X-Met for each primary and secondary objective are summarized in Tables 9-1 and 9-2. The X-Met and the average performance of all eight instruments that participated in the XRF technology demonstration are compared in Figure 9-1. The comparison in Figure 9-1 indicates that, when compared with the mean performance of all eight XRF instruments, the X-Met showed:

- Equivalent or better MDLs for five elements, including arsenic, cadmium, lead, selenium, and zinc (iron was not included in the MDL evaluation).
- Equivalent or better accuracy (lower RPDs) for eight of the 13 target elements (exceptions included antimony, chromium, copper, silver, and zinc). Moreover, when RPDs for antimony are calculated versus sample spike levels rather than reference laboratory data (which may be biased low), accuracy for antimony improves to better than the average of all eight instruments.
- Equivalent or better precision (lower RSDs) for six of the target elements (exceptions included antimony, cadmium, copper, lead, silver, vanadium, and zinc).

As a hand-held instrument, the X-Met is fully portable and can be operated in the hand-held mode at a sampling site. Although good overall performance was observed for this instrument, the developer may want to consider whether overall instrument accuracy and interelement interferences could be further improved for environmental applications through refined calibration protocols, quantitation algorithms, or other method modifications.

Table 9-1. Summary of Oxford X-Met 3000TX Performance – Primary Objectives

Objective	Performance Summary
P1: Method Detection Limits	• Mean MDLs for the target elements ranged as follows: ◦ MDLs of 1 to 20 ppm: arsenic and selenium. ◦ MDLs of 20 to 50 ppm: cadmium, copper, lead, mercury, silver, and zinc. ◦ MDLs of 50 to 100 ppm: nickel. ◦ MDLs greater than 100 ppm: antimony, chromium, and vanadium. (Iron was not included in the MDL evaluation.) • A blend of roaster slag from the Wickes Smelter site produced the highest MDLs for some target elements, ranging above 300 ppm for antimony, and vanadium. • For all the target elements except antimony, the MDLs calculated for the X-Met were significantly lower than reference MDL data from EPA Method 6200.
P2: Accuracy and Comparability	• Median RPDs between the XRF and reference laboratory data revealed the following, with lower RPDs indicating greater accuracy: ◦ RPDs of less than 10 percent: cadmium and selenium. ◦ RPDs of 10 to 25 percent: arsenic, iron, lead, and nickel. ◦ RPDs of 25 to 50 percent: chromium, copper, mercury, silver, vanadium, and zinc. ◦ RPDs greater than 50 percent: antimony. • Correlation plots relative to reference laboratory data indicated: ◦ High correlation coefficients (greater than 0.9) for 10 of the 13 target elements. ◦ Low to moderate correlation coefficients for antimony, silver, and vanadium. Further, a moderate degree of correlation for mercury was artificially improved by a few extreme concentrations. ◦ High biases in the XRF data versus the lab data for antimony, arsenic, lead, silver, vanadium, and zinc. • Data review indicated that the reference laboratory results for some spiked demonstration samples may be biased low for antimony due to the volatility of the spiking compounds used. Accuracy (RPDs and correlation) were improved for antimony when calculated relative to certified spike values rather than reference laboratory results. However, this improvement was not as great for the X-Met as for other instruments that participated in the XRF technology demonstration.

Table 9-1. Summary of X-Met 3000TX Performance – Primary Objectives (continued)

Objective	Performance Summary
P3: Precision	• Median RSDs were good for all elements, as follows: o RSDs less than 5 percent: cadmium, iron, mercury, and selenium. o RSDs of 5 to 10 percent: arsenic, copper, lead, nickel, silver, and zinc. o RSDs of 10 and 20 percent: antimony, chromium, and vanadium. o RSDs greater than 20 percent: none. • RSDs were slightly higher (that is, precision was lower) in the lowest concentration sample blends for many of the target elements, indicating a slight concentration dependence for precision. • For nine of the 13 target elements, median RSDs for the X-Met were lower than the RSDs calculated for the reference laboratory data, indicating slightly better precision for the XRF instrument.
P4: Effects of Sample Interferences	• High relative concentrations (greater than 10X) of interfering metals reduced accuracy for arsenic, copper, nickel, and zinc from "good" (median RPDs less than 25 percent) to "fair" or "poor" (median RPDs approaching 50 percent or more). • High concentrations of lead produced increasing negative biases in arsenic results, whereas high concentrations of copper, nickel, and zinc tended to produce tended to produce positive biases in each others' concentrations. • Although the above trends were apparent, the low numbers of samples available for some interferent-to-element ratios created some uncertainty in the interference evaluation.
P5: Effects of Soil Type	• Low relative accuracy was observed for cadmium in blends of roaster slag from the Wickes Smelter site, which contained high overall element concentrations. • Other high outlier RPD values, indicating low relative accuracy, were observed for iron in blends of sandy soil from the KARS Park site, a former gun range. • Overall, sample matrix had little observable effect on overall accuracy for the XRF data.
P6: Sample Throughput	• With an average sample preparation time of 2.0 minutes and an instrument analysis time of 5.0 minutes per sample, the total processing time was 7.1 minutes per sample. • A maximum sample throughput of 127 samples per day was achieved during an extended work day. A more typical sample throughput was estimated to be 72 samples per day for an 8-hour work day.
P7: Costs	• Instrument purchase cost is about \$30,000 with a weekly rental cost of \$2,000. These costs are for the instrument equipped as in the demonstration, including a Hewlett Packard iPAQ PDA-based operating system, instrument stand, and 110 volt AC adapter. • The Oxford field team expended approximately 54 labor hours to complete the processing of the demonstration sample set (326 samples). This was lower than the average for all participating XRF instruments of 69 labor hours. • Using the 1-week rental cost and adding labor and miscellaneous costs (\$445 for shipping and supplies), a total project cost of \$7,288 was estimated for a project the size of the demonstration. In comparison, the project cost averaged \$8,932 for all participating XRF instruments and the cost for fixed-laboratory analysis of all samples for 13 elements was \$63,896.

Table 9-2. Summary of X-Met 3000TX Performance – Secondary Objectives

Objective	Performance Summary
S1: Training Requirements	• Field or laboratory technicians with a high school diploma and basic operational training are generally qualified to operate the X-Met. • Oxford and its distributors offer on-site training on an informal, as needed basis, and provide telephone support through a toll-free number.
S2: Health and Safety	• The X-Met has a fail-safe lighting system and locking mechanism to manually control tube operation and analysis. Further, Oxford states that direct exposure to x-ray tube emissions for the entire life of the battery would not exceed exposure limits. • No chemicals are used during sample preparation or analysis that would pose potential hazards.
S3: Portability	• Based on dimensions, weight, and power requirements, the X-Met is a fully portable instrument. It can be used as a hand-held unit to analyze undisturbed soil or bagged samples. • With an additional instrument stand, the X-Met can be used in a hands-free, bench-top mode.
S4: Durability	• The X-Met has a 2-year limited warranty with a 5-year warranty for the x-ray tube. • The instrument is encased in durable hard-tool plastic and metal, and is largely weatherproof and impact-resistant. Through a USB port and cable, the iPAQ operating system can be used remotely to protect it from water or dust. Oxford also provides a protective cover for the instrument to further reduce exposure to weather and harmful conditions.
S5: Availability	• The X-Met is available for purchase or rental from a nationwide network of distributors. • Instrument repairs, maintenance, and calibration can be performed by the distributors or at the factory in Finland.

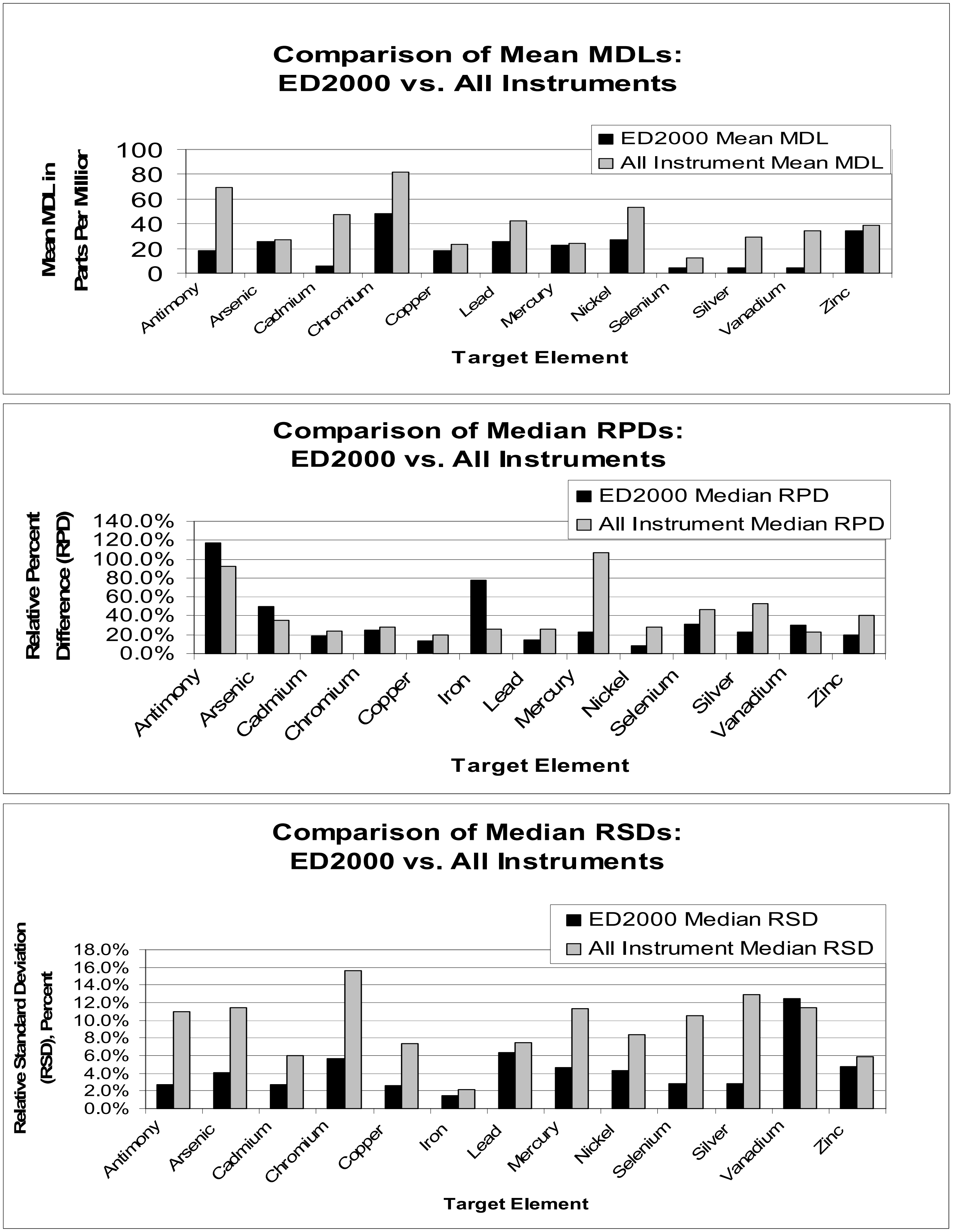

Figure 9-1. Method detection limits (sensitivity), accuracy, and precision of the X-Met 3000TX in comparison to the average of all eight XRF instruments.

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Chapter 10
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APPENDIX A

VERIFICATION STATEMENT

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, DC 20460

SITE Monitoring and Measurement Technology Program Verification Statement

TECHNOLOGY TYPE:	X-ray Fluorescence (XRF) Analyzer
APPLICATION:	MEASUREMENT OF TRACE ELEMENTS IN SOIL AND SEDIMENT
TECHNOLOGY NAME:	X-MET 3000TX XRF Analyzer
COMPANY:	Oxford Instruments Analytical, Inc.
ADDRESS:	Princeton Crossroads Corporate Center 250 Philips Boulevard Ewing, NJ 08618
Telephone:	(609) 406-9000 Ext. 122
Internet:	http://www.oxford-instruments.com
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VERIFICATION PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Superfund Innovative Technology Evaluation (SITE) Monitoring and Measurement Technology (MMT) Program to facilitate deployment of innovative technologies through performance verification and information dissemination. The goal of this program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The program assists and informs those involved in designing, distributing, permitting, and purchasing environmental technologies. This document summarizes the results of a demonstration of the Oxford Instruments Analytical, Inc., (Oxford) X-MET 3000TX portable x-ray fluorescence (XRF) analyzer for the analysis of 13 target elements in soil and sediment, including antimony, arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, vanadium, and zinc.

PROGRAM OPERATION

Under the SITE MMT Program, with the full participation of the technology developers, EPA evaluates and documents the performance of innovative technologies by developing demonstration plans, conducting field tests, collecting and analyzing demonstration data, and preparing reports. The technologies are evaluated under rigorous quality assurance protocols to produce well-documented data of known quality. EPA's National Exposure Research Laboratory, which demonstrates field sampling, monitoring, and measurement technologies, selected Tetra Tech EM Inc. as the verification organization to assist in field testing technologies for measuring trace elements in soil and sediment using XRF technology.

DEMONSTRATION DESCRIPTION

The field demonstration of eight XRF instruments to measure trace elements in soil and sediment was conducted from January 24 through 28, 2005, at the Kennedy Athletic, Recreational and Social (KARS) Park, which is part of the Kennedy Space Center on Merritt Island, Florida. A total of 326 samples were analyzed by each XRF instrument, including the X-MET 3000TX, during the field demonstration. These samples were derived from 70 different blends and spiked blends of soil and sediment collected from nine sites across the U.S. The sample blends were thoroughly dried, sieved, crushed, mixed, and characterized before they were used for the demonstration. Some blends were also spiked to further adjust and refine the concentration ranges of the target elements. Between three and seven replicate samples of each blend were included in the demonstration sample set and analyzed by the technology developers during the field demonstration.

Shealy Environmental Services, Inc. (Shealy), of Cayce, South Carolina, was selected as the reference laboratory to generate comparative data in evaluation of XRF instrument performance. Shealy analyzed all demonstration samples (both environmental and spiked) concurrently with the developers during the field demonstration. The samples were analyzed by inductively coupled plasma–atomic emission spectroscopy (ICP-AES) using EPA SW-846 Method 3050B/6010B and by cold vapor atomic absorption spectroscopy (CVAA) using EPA SW-846 Method 7471A (mercury only).

This verification statement provides a summary of the evaluation results for the X-MET 3000TX XRF instrument. More detailed discussion can be found in the *Innovative Technology Verification Report – XRF Technologies for Measuring Trace Elements in Soil and Sediment: Oxford X-MET 3000TX XRF Analyzer* (EPA/540/R-06/008).

TECHNOLOGY DESCRIPTION

XRF spectroscopy is an analytical technique that exposes a sample (soil, alloy metal, filters, other solids, and thin samples) to an x-ray source. The x-rays from the source have the appropriate excitation energy that causes elements in the sample to emit characteristic x-rays. A qualitative elemental analysis is possible from the characteristic energy, or wavelength, of the fluorescent x-rays emitted. A quantitative elemental analysis is possible from the number (intensity) of x-rays at a given wavelength.

The X-MET is a portable XRF analyzer that utilizes a miniature x-ray tube and a Peltier-cooled silicon-PiN diode x-ray detector. The X·MET can analyze elements from titanium (atomic number [Z] = 22) to uranium (Z = 92) simultaneously in soils, sediment, and other thick homogeneous samples (plastics and metals). Elements from potassium (Z = 19) to scandium (Z = 21) can also be analyzed with higher detection limits.

The analyzer is powered in the field with two lithium-ion batteries, or with AC power, if available. The X-MET utilizes an HP iPAQ personal data assistant (PDA) for data storage of up to 15,000 tests with spectra in its 64 MB memory. The iPAQ PDA provides a color, high-resolution display, with variable backlighting. Data can be transferred from the iPAQ to another PC by using a flash card, a USB cable, or Bluetooth© wireless. Other special internal features include multiple x-ray beam filters, adjustable tube voltages and currents, and selection of several pre-programmed calibration modules.

VERIFICATION OF PERFORMANCE

Method Detection Limit: MDLs were calculated using seven replicate analyses from each of 12 low-concentration sample blends, according to the procedure described in Title 40 Code of Federal Regulations (CFR) Part 136, Appendix B, Revision 1.11. A mean MDL was further calculated for each element. The ranges into which the mean MDLs fell for the X-MET are listed below.

Relative Sensitivity	Mean MDL	Target Elements
High	1 – 20 ppm	Arsenic and Selenium.
Moderate	20 – 50 ppm	Cadmium, Copper, Lead, Mercury, Silver, and Zinc.
Low	50 – 100 ppm	Nickel.
Very Low	100 – 200 ppm	Antimony, Chromium, and Vanadium.

Note: ppm = Parts per million. Iron was not included in the MDL evaluation.

Accuracy: Accuracy was evaluated based on the agreement of the XRF results with the reference laboratory data. Accuracy was assessed by calculating the absolute relative percent difference (RPD) between the mean

XRF and the mean reference laboratory concentration for each blend. Accuracy of the X-MET was classified from high to very low for the various target elements, as indicated in the table below, based on the overall median RPDs calculated for the demonstration.

Relative Accuracy	Median RPD	Target Elements
High	0% - 10%	Cadmium and Selenium.
Moderate	10% - 25%	Arsenic, Iron, Lead, and Nickel.
Low	25% - 50%	Chromium, Copper, Silver, Vanadium, and Zinc.
Very Low	50% - 100%	Antimony and Mercury.

Accuracy was also assessed through correlation plots between the mean X-MET and mean reference laboratory concentrations for the various sample blends. Correlation coefficients (r^2) for linear regression analysis of the plots are summarized below, along with any significant biases apparent from the plots in the XRF data versus the reference laboratory data.

	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Correlation	0.76	0.99	0.98	0.93	0.95	0.94	0.98	0.98	0.97	0.98	0.69	0.41	0.98
Bias	High	High	--	--	--	--	High	--	--	--	High	High	High

Note: -- = No significant bias.

Precision: Replicates were analyzed for all sample blends. Precision was evaluated by calculating the standard deviation of the replicates, dividing by the average concentration of the replicates, and multiplying by 100 percent to yield the relative standard deviation (RSD) for each blend. Precision of the X-MET was classified from high to very low for each target element, as indicated in the table below, based on the overall median RSDs. These results indicated a higher level of precision in the X-MET than in the reference laboratory data for eight of the 13 target elements.

Relative Precision	Median RSD	Target Elements
High	0% - 5%	Cadmium, Iron, and Selenium.
Moderate	5% - 10%	Arsenic, Copper, Lead, Mercury, Nickel, Silver, and Zinc.
Low	10% - 20%	Antimony, Chromium, and Vanadium.
Very Low	20% - 50%	None.

Effects of Interferences: The RPDs from the evaluation of accuracy were further grouped and compared for a few elements of concern (arsenic, nickel, copper, and zinc) based on the relative concentrations of potentially interfering elements. High relative concentrations (greater than 10X) of interfering metals reduced accuracy for arsenic, copper, nickel, and zinc from “moderate” (median RPDs of 25 percent or less) to “very low” (median RPDs greater than 50 percent). Interfering elements increased the high bias of the arsenic data and produced a more negative bias in the data for nickel, copper, and zinc.

Effects of Soil Characteristics: The RPDs from the evaluation of accuracy were also further evaluated in terms of sampling site and soil type. This evaluation found high outlier RPD values, indicating low relative accuracy, for cadmium in blends of roaster slag from the Wickes Smelter site. These blends contained high overall element concentrations. Other high outlier RPD values were observed for multiple target elements in six blends of sandy soil from the KARS Park site, a former gun range. Overall, however, sample matrix had little observable effect on overall accuracy for the XRF data.

Sample Throughput: Field observers timed individual sample batches during the demonstration; the results indicated analysis time as performed by the developer averaged 6.4 minutes per sample for the X-MET. With additional time for other daily activities (such as instrument start-up, shutdown, quality control checks, and breaks), a routine sample throughput of 80 to 120 samples per day was estimated. As noted above, however, the sample blends had undergone rigorous pre-processing before the demonstration. Sample throughput would have decreased if these processing steps (grinding, drying, sieving) had been performed during the demonstration. Sample processing can add from 10 minutes to 2 hours to sample analysis time.

Costs: A cost assessment for the X-MET identified a purchase cost of $30,000 and a weekly rental cost of $2,000, plus $200 shipping, as equipped for the demonstration. A total cost of $6,525 (with a labor cost of $3,991 at $43.75/hr) associated with sample preparation and analysis was estimated for a project similar to the demonstration (326 samples of soil and sediment). In comparison, the project cost averaged $7,271 for all eight XRF instruments participating in the demonstration, and $63,896 for fixed-laboratory analysis of all samples for the 13 target elements.

Skills and Training Required: Field or laboratory technicians with a high school diploma are generally qualified to operate the X-MET. Oxford and its distributors offer on-site training on an informal, as needed basis, and provide telephone support through a toll-free number.

Health and Safety Aspects: The X-MET has a fail-safe lighting system and locking mechanism to manually control tube operation and analysis. Further, Oxford states that direct exposure to x-ray tube emissions for the entire life of the battery would not exceed exposure limits. No chemicals are used during sample preparation or analysis that would pose potential hazards.

Portability: Based on dimensions, weight (4 lbs.), and power requirements, the X-MET is a fully portable instrument. It can be used as a hand-held unit to analyze undisturbed soil or bagged samples. With the instrument stand, the X-MET can be used in a hands-free, bench-top mode.

Durability: Oxford offers a 2-year limited warranty on the X-MET with a 5-year warranty on the x-ray tube. The expected lifespan of an x-ray tube is 2,000 operating hours. The instrument is encased in durable hard-tool plastic and metal. With an available protective cover and USB cable for the iPAQ, the instrument can be used in wet or dusty conditions.

Availability: The X-MET can be purchased, rented, and serviced from the factory in Finland or through a nationwide network of distributors.

RELATIVE PERFORMANCE

The overall performance of the X-MET analyzer relative to the average of all eight XRF instruments that participated in the demonstration is shown below:

	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Sensitivity	○	●	●	○	Same	---	●	○	Same	Same	○	○	Same
Accuracy	●	●	●	○	○	●	●	●	●	●	●	Same	○
Precision	Same	●	●	●	●	Same	●	●	●	●	●	Same	Same

Key: ● Better ○ Worse

NOTICE: Verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always operate as verified. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements.

APPENDIX B

DEVELOPER DISCUSSION

DEVELOPER DISCUSSION

Oxford Instruments was pleased to participate in this SITE program demonstration of "XRF Technologies for Measuring Trace Elements in Soil and Sediment". This demonstration provides significant information to the potential users of XRF interested in the investigation of heavy metal contamination in soil. The instrument used in this demonstration was calibrated using an early version of the Universal Empirical Calibration for heavy metals in soil matrix samples. This calibration is consistent with the EPA Method 6200 and uses Compton Normalization in order to compensate for matrix differences.

The X-MET 3000TXS is a field portable EDXRF instrument which is designed for operation in almost any environment. As such, this analyzer can be used either for direct in-situ screening of samples at the source or it may be set up in Bench-top mode (as it was during this study) for analysis of samples in a field laboratory close to the source of samples.

The results of this SITE study were essentially as expected with the exception of the results for Antimony (discussed below). The MDLs calculated from the data were all within about a factor of two of the theoretical interference free values calculated prior to the start of the study. The two elements in the study (other than Antimony) with high detection limits and moderate precision, Chromium and Vanadium, are low atomic number elements which means that the energy of their characteristic x-rays is low and therefore the excitation of these elements is important. By changing the filter and/or the excitation current and voltage it is possible improve the excitation and therefore substantially improve the MDL and the precision of these two elements. Later versions of the X-MET 3000 TXS will have programmable filters and current to improve the excitation of these two elements and therefore the MDL and precision of the measurement.

While good accuracy of the measurement is desirable, the nature of the matrix and particle size play an important role in determining the value obtained. The samples in this project were very well prepared and the particle size was very small and uniform, so it is unlikely that the sample particle size influenced the values observed. However, the different matrix likely influences the final result. Looking at the results by matrix type there are apparent differences in the accuracy for different matrix types. In some cases the soil matrix results are more accurate while in other case the sediment matrix results are more accurate. In either case, the influence of matrix can be overcome by either performing site specific empirical calibrations or using type standards to determine the relationship between the measured result and the actual value. Thus, as long as the precision of the data is good, the influence of the matrix or particle size can be corrected by appropriate processing of the data.

A site specific empirical calibration uses a set of samples from the location being studied to calibrate the instrument. This can be achieved by taking a well analyzed set of samples from the site and calibrating the instrument using them. This calibration can then be used to measure all the other samples with the same matrix and will provide accurate results as the influence of the matrix will be compensated during the calibration. If a complete set of site specific samples is difficult to obtain, it is possible to correct the data by using a type standard. In this case, one or two samples of known concentrations can be measured and the results can then be used to calculate a correction to the slope of the calibration curve. This correction which is then applied to all measured samples can be calculated either within the instrument software or an offline computer.
Finally, it appears that the calibration for antimony used during this demonstration was erroneous. Due to an oversight in the calibration process incorrect variables were chosen. Due to a lack of adequate test samples this error was not realized until the entire data set was available. The theoretically calculated detection limit for antimony is 23 mg/kg. Given the other results of this demonstration it is reasonable to expect that using the proper calibration equation will yield a MDL of under 50 mg/kg, similar to or lower that that reported in EPA Method 6200.

APPENDIX C

DATA VALIDATION SUMMARY REPORT

Contents

Chapter **Page**

APPENDIX

ABBREVIATIONS AND ACRONYMS

CCV	Continuing calibration verification
CVAA	Cold vapor atomic absorption
DVSR	Data validation summary report
EPA	U.S. Environmental Protection Agency
FAR	Federal acquisition regulations
ICP-AES	Inductively coupled plasma-atomic emission spectroscopy
ICS	Interference check sample
ICV	Initial calibration verification
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
MDL	Method detection limit
mg/kg	Milligram per kilogram
MS	Matrix spike
MSD	Matrix spike duplicate
PARCC	Precision, accuracy, representativeness, completeness, and comparability
PQL	Practical quantitation limit
QA/QC	Quality assurance and quality control
QAPP	Quality assurance project plan
QC	Quality control
RSD	Relative standard deviation
RPD	Relative percent difference
SDG	Sample delivery group
Shealy	Shealy Environmental Services, Inc.
SITE	Superfund Innovative Technology Evaluation
Tetra Tech	Tetra Tech EM Inc.
XRF	X-ray fluorescence

1.0 INTRODUCTION

This data validation summary report (DVSR) summarizes the reference laboratory quality control (QC) data gathered during the x-ray fluorescence (XRF) technologies demonstration conducted under the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) program. The reference laboratory was procured following the federal acquisition regulations (FAR) and an extensive selection process. Shealy Environmental Services, Inc. (Shealy), of Cayce, South Carolina, was selected as the reference laboratory for this project. Thirteen target analytes were measured in reference samples and include antimony, arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, vanadium, and zinc. The laboratory reported results for 22 metals at the request of EPA; however, for the purposes of meeting project objectives, only the data validation for the 13 target analytes is summarized in this document. The objective of the validation is to determine the validity of the reference data, as well as its usability in meeting the primary objective of comparing reference data to XRF data generated during the demonstration. Shealy provided the data to Tetra Tech EM Inc. (Tetra Tech) in electronic and hardcopy formats; a total of 13 sample delivery groups (SDG) contain all the data for this project.

The DVSR consists of seven sections, including this introduction. Section 2.0 presents the data validation methodology. Section 3.0 presents the results of the reference laboratory data validation. Section 4.0 summarizes the precision, accuracy, representativeness, completeness, and comparability (PARCC) evaluation. Section 5.0 presents conclusions about the overall evaluation of the reference data. Section 6.0 lists the references used to prepare this DVSR. Tables are presented following Section 6.0.

2.0 VALIDATION METHODOLOGY

Data validation is the systematic process for reviewing and qualifying data against a set of criteria to ensure that the reference data are adequate for the intended use. The data validation process assesses acceptability of the data by evaluating the critical indicator parameters of PARCC. The laboratory analytical data were validated according to the procedures outlined in the following documents:

- "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" (EPA 2004), hereinafter referred to as the "EPA guidance."

- "Demonstration and Quality Assurance Project Plan, XRF Technologies for Measuring Trace Elements in Soil and Sediment" (Tetra Tech 2005), hereinafter referred to as "the QAPP."

Data validation occurred in the following two stages: (1) a cursory review of analytical reports and quality assurance and quality control (QA/QC) information for 100 percent of the reference data and (2) full validation of analytical reports, QA/QC information, and associated raw data for 10 percent of the reference data as required by the QAPP (Tetra Tech 2005).

QA/QC criteria were reviewed in accordance with EPA guidance (EPA 2004) and the QAPP (Tetra Tech 2005). The cursory review for total metals consisted of evaluating the following requirements, as applicable:

- Holding times

- Initial and continuing calibrations

- Laboratory blank results

- Laboratory control sample (LCS) and laboratory control sample duplicates (LCSD) results

- Matrix spike (MS) and matrix spike duplicate (MSD) results

- Serial dilutions results

In addition to QA/QC criteria described above, the following criteria were reviewed during full validation:

- ICP interference check samples (ICS)

- Target analyte identification and quantitation

- Quantitation limit verification

Section 3.0 presents the results of the both the cursory review and full validation.

During data validation, worksheets were produced for each SDG that identify any QA/QC issues resulting in data qualification. Data validation findings were written in 13 individual data validation reports (one for each SDG). Data qualifiers were assigned to the results in the electronic database in accordance with EPA guidelines (EPA 2004). In addition to data validation qualifiers, comment codes were added to the database to indicate the primary reason for the validation qualifier. Table 1 defines data validation qualifiers and comment codes that are applied to the data set. Details about specific QC issues can be found in the individual SDG data validation reports and accompanying validation worksheets provided in the Appendix.

The overall objective of data validation is to ensure that the quality of the reference data set is adequate for the intended use, as defined by the QAPP (Tetra Tech 2005) for the PARCC parameters. Table 2 provides the QC criteria as defined by the QAPP. PARCC parameters were assessed by completing the following tasks:

- Reviewing precision and accuracy of laboratory QC data

- Reviewing the overall analytical process, including holding time, calibration, analytical or matrix performance, and analyte identification and quantitation

- Assigning qualifiers to affected data when QA/QC criteria were not achieved

- Reviewing and summarizing implications of the frequency and severity of qualifiers in the validated data

Prior to the XRF demonstration, soil and sediment samples were collected from nine locations across the U.S. and then blended, dried, sieved, and homogenized in the characterization laboratory to produce a set of 326 reference samples. Each of these samples were subsequently analyzed by both the reference laboratory and all participating technology vendors. As such, 326 prepared soil/sediment samples were delivered to Shealy for the measurement of total metals. The analytical program included the following analyses and methods:

- Total metal for 22 analytes by inductively coupled plasma atomic emission spectroscopy (ICP-AES) according to EPA Methods 3050B/6010B (EPA 1996)
- Total mercury by cold vapor atomic absorption spectroscopy (CVAA) according to EPA Method 7471A (EPA 1996)

3.0 DATA VALIDATION RESULTS

The parameters listed in Section 2.0 were evaluated during cursory review and full validation of analytical reports for all methods, as applicable. Each of the validation components discussed in this section is summarized as follows:

- Acceptable – All criteria were met and no data were qualified on that basis
- Acceptable with qualification – Most criteria were met, but at least one data point was qualified as estimated because of issues related to the review component

Since no data were rejected, all data were determined to be either acceptable or acceptable with qualification. Sections 3.1 through 3.9 discuss each review component and the results of each. Tables that summarize the data validation findings follow Section 6.0 of this DVSR. Only qualified data are included in the tables. No reference laboratory data were rejected during the validation process. As such, all results are acceptable with the qualification noted in the sections that follow.

3.1 Holding Time

Acceptable. The technical holding times were defined as the maximum time allowable between sample collection and, as applicable, sample extraction, preparation, or analysis. The holding times used for validation purposes were recommended in the specific analytical methods (EPA 1996) and were specified in the QAPP (Tetra Tech 2005).

Because the soil and sediment samples were prepared prior to submission to the reference laboratory, and because the preparation included drying to remove moisture, no chemical or physical (for example ice) preservation was required. The holding time for sample digestion was 180 days for the ICP-AES analyses and 28 days for mercury. All sample digestions and analyses were conducted within the specified holding times. No data were qualified based on holding time exceedances. This fact contributes to the high technical quality of the reference data.

3.2 Calibration

Acceptable. Laboratory instrument calibration requirements were established to ensure that analytical instruments could produce acceptable qualitative and quantitative data for all target analytes. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of an analytical run, while producing a linear curve. Continuing calibration demonstrates that the instrument is capable of repeating the performance established during the initial calibration (EPA 1996).

For total metal analyses (ICP-AES and CVAA), initial calibration review included evaluating criteria for the curve's correlation coefficient and initial calibration verification (ICV) percent recoveries. The ICV percent recoveries verify that the analytical system is operating within the established calibration criteria at the beginning of an analytical run. The continuing calibration review included evaluation of the criteria for continuing calibration verification (CCV) percent recoveries. The CCV percent recoveries verify that the analytical system is operating within the established calibration throughout the analytical run.

All ICV and CCV percent recoveries associated with the reference data were within acceptable limits of 90 to 110 percent. As such, no data were qualified or rejected because of calibration exceedances. This fact contributes to the high technical quality of the data.

3.3 Laboratory Blanks

Acceptable with qualification. No field blanks were required by the QAPP, since samples were prepared after collection and before submission to the reference laboratory. However, laboratory blanks were prepared and analyzed to evaluate the existence and magnitude of contamination resulting from laboratory activities. Blanks prepared and analyzed in the laboratory consisted of calibration and preparation blanks. If a problem with any blank existed, all associated data were carefully evaluated to assess whether the sample data were affected. At a minimum, calibration blanks were analyzed for every 10 analyses conducted on each instrument. Preparation blanks were prepared at a frequency of one per preparation batch per matrix or every 20 samples, whichever is greater (EPA 1996).

When laboratory blank contamination was identified, sample results were compared to the practical quantitation limit (PQL) and the maximum blank value as required by the validation guidelines (EPA 2004). Most of the blank detections were positive results (i.e. greater than the method detection limit [MDL]), but less than the PQL. In these instances, if associated sample results were also less than the PQL, they were qualified as undetected (U); with the comment code "b." In these same instances, if the associated sample results were greater than the PQL, the reviewer used professional judgment to determine if the sample results were adversely affected. If so, then the results were qualified as estimated with the potential for being biased high (J+). If not, then no qualification was required.

In a few cases, the maximum blank value exceeded the PQL. In these cases, all associated sample results less than the PQL were qualified as undetected (U) with the comment code "b." In cases where the associated sample results were greater than the PQL, but less than the blank concentration, the results were also qualified as undetected (U); with the comment code "b." If the associated sample results were greater than both the PQL and the blank value, the reviewer used professional judgment to determine if sample results were adversely affected. If so, then the results were qualified as estimated with the potential for being biased high (J+); with the comment code "b." Sample results significantly above the blank were not qualified.

In addition to laboratory blank contamination, negative drift greater than the magnitude of the PQL was observed in some laboratory blanks. Associated sample data were qualified as undetected (U) if the results were less than the PQL. Professional judgement was used to determine if the negative drift adversely affected associated sample results greater than the PQL. If so, then sample results were qualified as estimated with the potential for being biased low (J-) due to the negative drift of the instrument baseline; with the comment code "b."

Of all target analyte data, 2.6 percent of the data was qualified as undetected because of laboratory blank contamination (U, b), and less than 1 percent of the data was qualified as estimated (either J+, b or J-, b). The low occurrence of results affected by blank contamination indicates that the general quality of the analytical data was not significantly compromised by blank contamination. Table 3 provides all results that were qualified based on laboratory blanks.

3.4 Laboratory Control Samples

Acceptable. LCSs and LCSDs were prepared and analyzed with each batch of 20 or fewer samples of the same matrix. All percent recoveries were within the QC limits of 80 to 120 percent; all relative percent differences (RPD) between the LCD and LCSD values were less than the criterion of 20 percent. No data were qualified or rejected on the basis of LCS/LCSD results. This fact contributes to the high technical quality of the data.

3.5 Matrix Spike Samples

Acceptable with qualification. MS and MSD samples were prepared and analyzed with each batch of 20 or fewer samples of the same matrix. All percent recoveries were within the QC limits of 75 to 125 percent, and all RPDs between the MS and MSD values were less than the criterion of 25 percent, except as discussed in the following paragraphs.

Sample results affected by MS and MSD percent recoveries issues were qualified as estimated and either biased high (J+) if the recoveries were greater than 125 percent; or qualified as estimated and biased low (J-) if the recoveries were less than 75 percent. In at least one case, the MS was higher than 125 percent and the MSD was lower than 75 percent; the associated results were qualified as estimated (J) with no distinction for potential bias. All data qualified on the basis of MS and MSD recovery were also assigned the comment code “e.” Of all target analyte data, less than 1 percent was qualified as estimated and biased high (J+, e), while about 8 percent of the data were qualified as estimated and biased low (J-, e). Antimony and silver were the most frequently qualified sample results. Based on experience, antimony and silver soil recoveries are frequently low using the selected methods. Table 4 provides the results that were qualified based on MS/MSD results.

The precision between MS and MSD results were generally acceptable. If the RPD between MS and MSD results were greater than 25 percent, the data were already qualified based on exceedance of the acceptance window for recovery. Therefore, no additional qualification was required for MS/MSD precision.

No data were rejected on the basis of MS/MSD results. The relatively low occurrence of data qualification due to MS/MSD recoveries and RPDs contribute to the high technical quality of the data.

3.6 Serial Dilution Results

Acceptable with qualification. Serial dilutions were conducted and analyzed by Shealy at a frequency of 1 per batch of 20 samples. The serial dilution analysis can evaluate whether matrix interference exists and whether the accuracy of the analytical data is affected. For all target analyte data, less than 1 percent of the data was qualified as estimated and biased high (J+, j), while about 2 percent of the data were qualified as estimated and biased low (J-, j). Serial dilution results are used to determine whether characteristics of the digest matrix, such as viscosity or the presence of analytes at high concentrations, may interfere with the detected analytes. Qualifiers were applied to cases where interference was suspected. However, the low incidence of apparent matrix interference contributes to the high technical quality of the data. Table 5 provides the results that were qualified based on MS/MSD results.

3.7 ICP Interference Check Samples

Acceptable. ICP results for each ICS were evaluated. The ICS verifies the validity of the laboratory's inter-element and background correction factors. High levels of certain elements (including aluminum, calcium, iron, and magnesium) can affect sample results if the inter-element and background correction factors have not been optimized. Incorrect correction factors may result in false positives, false negatives, or biased results. All ICS recoveries were within QC limits of 80 to 120 percent, and no significant biases were observed due to potential spectral interference. No data were qualified or rejected because of ICS criteria violations. This fact contributes to the high technical quality of the data.

3.8 Target Analyte Identification and Quantitation

Acceptable Identification is determined by measuring the characteristic wavelength of energy emitted by the analyte (ICP) or absorbed by the analyte (CVAA). External calibration standards are used to quantify the analyte concentration in the sample digest. Sample digest concentrations are converted to soil units (milligrams per kilogram) and corrected for percent moisture. For 10 percent of the samples, results were recalculated to verify the accuracy of reporting. All results were correctly calculated by the laboratory, except for one mercury result, whose miscalculation was the result of an error in entering the dilution factor. Shealy immediately resolved this error and corrected reports were provided. Since the result was corrected, no qualification was required. No other reporting errors were observed.

For inorganic analyses, analytical instruments can make reliable qualitative identification of analytes at concentrations below the PQL. Detected results below the PQL are considered quantitatively uncertain. Sample results below the PQL were reported by the laboratory with a "J" qualifier. No additional qualification was required.

3.9 Quantitation Limit Verification

Acceptable. Reference laboratory quantitation limits were specified in the QAPP (Tetra Tech 2005). Circumstances that affected quantitation were limited and included dilution and percent moisture factors. Since the samples were prepared prior to submission to the reference laboratory, moisture content was very low and had little impact on quantitation limits. The laboratory did correct all quantitation limits for moisture content. Due to the presence of percent-level analytes in some samples, dilutions were required. However, the required PQLs for the reference laboratory were high enough that even with dilution and moisture content factors applied, the reporting limits did not exceed those of the XRF instruments. This allows for effective comparison of results between the reference laboratory and XRF instruments.

4.0 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY EVALUATION SUMMARY

All analytical data were reviewed for PARCC parameters to validate reference data. The following sections discuss the overall data quality, including the PARCC parameters, as determined by the data validation.

4.1 Precision

Precision is a measure of the reproducibility of an experimental value without considering a true or referenced value. The primary indicators of precision were the MS/MSD RPD and LCS/LCSD RPD between the duplicate results. Precision criteria of less than 20 percent RPD for LCS/LCSD and 25 percent for MS/MSD were generally met for all duplicate pairs. No data were qualified based on duplicate precision of MS/MSD or LCS/LCSD pairs that were not already qualified for other reasons. Such low occurrence of laboratory precision problems supports the validity, usability, and defensibility of the data.

4.2 Accuracy

Accuracy assesses the proximity of an experimental value to a true or referenced value. The primary accuracy indicators were the recoveries of MS and LCS spikes. Accuracy is expressed as percent recovery. Overall, about 8 percent of the data was qualified as estimated and no data were rejected because of accuracy problems. The low frequency of accuracy problems supports the validity, usability, and defensibility of the data.

4.3 Representativeness

Representativeness refers to how well sample data accurately reflect true environmental conditions. The QAPP was carefully designed to ensure that actual environmental samples be collected by choosing representative sites across the US from which sample material was collected. The blending and homogenization was executed according to the approved QAPP (Tetra Tech 2005).

4.4 Completeness

Completeness is defined as the percentage of measurements that are considered to be valid. The validity of sample results is evaluated through the data validation process. Sample results that are rejected and any missing analyses are considered incomplete. Data that are qualified as estimated (J) or undetected estimated (UJ) are considered valid and usable. Data qualified as rejected (R) are considered unusable for all purposes. Since no data were rejected in this data set, a completeness of 100 percent was achieved. A total of 4,238 target analyte results were evaluated. The completeness goal stated in the QAPP (Tetra Tech 2005) was 90 percent.

4.5 Comparability

Comparability is a qualitative parameter that expresses the confidence with which one data set may be compared to another. Widely-accepted SW-846 methods were used for this project. It is recognized that direct comparison of the reference laboratory data (using ICP-AES and CVAA techniques) to the XRF measurements may result in discrepancies due to differences in the preparation and measurement techniques; however, the reference laboratory data is expected to provide an acceptable basis for comparison to XRF measurement results in accordance with the project objectives.

Comparability of the data was also achieved by producing full data packages, by using a homogenous matrix, standard quantitation limits, standardized data validation procedures, and by evaluating the PARCC parameters uniformly. In addition, the use of specified and well-documented analyses, approved laboratories, and the standardized process of data review and validation have resulted in a high degree of comparability for the data.

5.0 CONCLUSIONS FOR DATA QUALITY AND DATA USABILITY

Although some qualifiers were added to the data, a final review of the data set with respect to the data quality parameters discussed in Section 4.0 indicates that the data are of overall good quality. No analytical data were rejected. The data quality is generally consistent with project objectives for producing data of suitable quality for comparison to XRF data. All supporting documentation and data are available upon request, including cursory review and full validation reports as well as the electronic database that contains sample results.

6.0 REFERENCES

Tetra Tech EM, Inc. (Tetra Tech). 2005. "Demonstration and Quality Assurance Project Plan, XRF Technologies for Measuring Trace Elements in Soil and Sediment." March.

U.S. Environmental Protection Agency (EPA). 1996. "Test Methods for Evaluating Solid Waste", Third Edition (SW-846). With promulgated revisions. December.

EPA. 2004. "USEPA Contract Laboratory Program National Functional Guidelines For Inorganic Data Review". October.

TABLES

TABLE 1: DATA VALIDATION QUALIFIERS AND COMMENT CODES

Qualifier	Definition
No Qualifier	Indicates that the data are acceptable both qualitatively and quantitatively.
U	Indicates compound was analyzed for but not detected above the concentration listed. The value listed is the sample quantitation limit.
J	Indicates an estimated concentration value. The result is considered qualitatively acceptable, but quantitatively unreliable.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	Indicates an estimated quantitation limit. The compound was analyzed for, but was considered non-detected.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.

Comment Code	Definition
a	Surrogate recovery exceeded (not applicable to this data set)
b	Laboratory method blank and common blank contamination
c	Calibration criteria exceeded
d	Duplicate precision criteria exceeded
e	Matrix spike or laboratory control sample recovery exceeded
f	Field blank contamination (not applicable to this data set)
g	Quantification below reporting limit
h	Holding time exceeded
i	Internal standard criteria exceeded (not applicable to this data set)
j	Other qualification (will be specified in report)

TABLE 2: QC CRITERIA

Parameter	Method	QC Check	Frequency	Criterion	Corrective Action
			Reference Method		
Target Metals (12 ICP metals and Hg)	3050B/6010B and 7471A	Method and instrument blanks	One per analytical batch of 20 or less	Less than the reporting limit	1. Check calculations 2. Assess and eliminate source of contamination 3. Reanalyze blank 4. Inform Tetra Tech project manager 5. Flag affected results
		MS/MSD	One per analytical batch of 20 or less	75 to 125 percent recovery RPD ≤ 25	1. Check calculations 2. Check LCS/LCSD and digest duplicate results to determine whether they meet criterion 3. Inform Tetra Tech project manager 4. Flag affected results
		LCS/LCSD	One per analytical batch of 20 or less	80 to 120 percent recovery RPD ≤ 20	1. Check calculations 2. Check instrument operating conditions and adjust as necessary 3. Check MS/MSD and digest duplicate results to determine whether they meet criterion 4. Inform Tetra Tech project manager 5. Redigest and reanalyze the entire batch of samples 6. Flag affected results
		Performance audit samples	One per analytical batch of 20 or less	Within acceptance limits	1. Evaluated by Tetra Tech QA chemist 2. Inform laboratory and recommend changes 3. Flag affected results
Percent moisture		Laboratory duplicates	One per analytical batch of 20 or less	RPD ≤ 20	1. Check calculations 2. Reanalyze sample batch 3. Inform Tetra Tech project manager 4. Flag affected results

TABLE 3: DATA QUALIFICATION: LABORATORY METHOD BLANK CONTAMINATION

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
AS-SO-04-XX	Selenium	6.2	mg/kg	U	b
AS-SO-06-XX	Antimony	2.4	mg/kg	UJ	b, e
AS-SO-10-XX	Selenium	1.1	mg/kg	U	b
AS-SO-11-XX	Selenium	1.1	mg/kg	U	b
AS-SO-13-XX	Antimony	2.4	mg/kg	UJ	b, e
BN-SO-18-XX	Silver	0.94	mg/kg	U	b
BN-SO-28-XX	Silver	0.77	mg/kg	U	b
BN-SO-31-XX	Silver	0.97	mg/kg	U	b
BN-SO-35-XX	Silver	0.85	mg/kg	U	b
KP-SE-01-XX	Mercury	0.053	mg/kg	U	b
KP-SE-11-XX	Mercury	0.079	mg/kg	U	b
KP-SE-12-XX	Mercury	0.06	mg/kg	U	b
KP-SE-14-XX	Mercury	0.065	mg/kg	U	b
KP-SE-17-XX	Mercury	0.082	mg/kg	U	b
KP-SE-19-XX	Mercury	0.044	mg/kg	U	b
KP-SE-25-XX	Mercury	0.096	mg/kg	U	b
KP-SE-25-XX	Selenium	0.26	mg/kg	U	b
KP-SE-28-XX	Mercury	0.056	mg/kg	U	b
KP-SE-30-XX	Mercury	0.1	mg/kg	U	b
KP-SE-30-XX	Selenium	0.24	mg/kg	U	b
KP-SO-02-XX	Mercury	0.043	mg/kg	U	b
KP-SO-02-XX	Selenium	0.42	mg/kg	U	b
KP-SO-03-XX	Cadmium	0.074	mg/kg	U	b
KP-SO-03-XX	Mercury	0.044	mg/kg	U	b
KP-SO-04-XX	Cadmium	0.046	mg/kg	U	b
KP-SO-04-XX	Mercury	0.018	mg/kg	U	b
KP-SO-04-XX	Selenium	0.28	mg/kg	U	b
KP-SO-05-XX	Cadmium	0.13	mg/kg	U	b
KP-SO-05-XX	Mercury	0.044	mg/kg	U	b
KP-SO-05-XX	Selenium	0.24	mg/kg	U	b
KP-SO-06-XX	Arsenic	0.73	mg/kg	J-	b
KP-SO-06-XX	Mercury	0.059	mg/kg	U	b
KP-SO-07-XX	Arsenic	2	mg/kg	J-	b
KP-SO-07-XX	Mercury	0.027	mg/kg	U	b
KP-SO-07-XX	Selenium	0.21	mg/kg	U	b
KP-SO-09-XX	Cadmium	0.094	mg/kg	U	b
KP-SO-09-XX	Mercury	0.046	mg/kg	U	b

TABLE 3: DATA QUALIFICATION: LABORATORY METHOD BLANK CONTAMINATION (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
KP-SO-10-XX	Arsenic	0.7	mg/kg	J-	b
KP-SO-10-XX	Mercury	0.028	mg/kg	U	b
KP-SO-10-XX	Selenium	0.22	mg/kg	U	b
KP-SO-13-XX	Arsenic	1.4	mg/kg	J-	b
KP-SO-13-XX	Cadmium	0.045	mg/kg	U	b
KP-SO-13-XX	Mercury	0.037	mg/kg	U	b
KP-SO-15-XX	Arsenic	0.76	mg/kg	J-	b
KP-SO-15-XX	Mercury	0.029	mg/kg	U	b
KP-SO-16-XX	Cadmium	0.063	mg/kg	U	b
KP-SO-16-XX	Mercury	0.016	mg/kg	U	b
KP-SO-18-XX	Arsenic	0.56	mg/kg	J-	b
KP-SO-18-XX	Mercury	0.016	mg/kg	U	b
KP-SO-20-XX	Arsenic	1.5	mg/kg	J-	b
KP-SO-20-XX	Mercury	0.03	mg/kg	U	b
KP-SO-21-XX	Cadmium	0.098	mg/kg	U	b
KP-SO-21-XX	Mercury	0.042	mg/kg	U	b
KP-SO-22-XX	Arsenic	0.7	mg/kg	J-	b
KP-SO-22-XX	Mercury	0.027	mg/kg	U	b
KP-SO-23-XX	Cadmium	0.048	mg/kg	U	b
KP-SO-23-XX	Mercury	0.017	mg/kg	U	b
KP-SO-24-XX	Arsenic	1.4	mg/kg	J-	b
KP-SO-24-XX	Mercury	0.017	mg/kg	U	b
KP-SO-26-XX	Cadmium	0.061	mg/kg	U	b
KP-SO-26-XX	Mercury	0.013	mg/kg	U	b
KP-SO-26-XX	Selenium	0.22	mg/kg	U	b
KP-SO-27-XX	Arsenic	1.3	mg/kg	J-	b
KP-SO-27-XX	Cadmium	0.05	mg/kg	U	b
KP-SO-27-XX	Mercury	0.021	mg/kg	U	b
KP-SO-29-XX	Arsenic	1.5	mg/kg	J-	b
KP-SO-29-XX	Mercury	0.013	mg/kg	U	b
KP-SO-31-XX	Mercury	0.017	mg/kg	U	b
KP-SO-32-XX	Arsenic	1.6	mg/kg	J-	b
KP-SO-32-XX	Cadmium	0.045	mg/kg	U	b
KP-SO-32-XX	Mercury	0.014	mg/kg	U	b
LV-SE-02-XX	Mercury	0.02	mg/kg	U	b
LV-SE-10-XX	Mercury	0.023	mg/kg	U	b
LV-SE-11-XX	Selenium	1.3	mg/kg	U	b

TABLE 3: DATA QUALIFICATION: LABORATORY METHOD BLANK CONTAMINATION (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
LV-SE-14-XX	Mercury	0.056	mg/kg	U	b
LV-SE-21-XX	Mercury	0.048	mg/kg	U	b
LV-SE-24-XX	Mercury	0.053	mg/kg	U	b
LV-SE-29-XX	Selenium	1.2	mg/kg	U	b
LV-SE-32-XX	Mercury	0.052	mg/kg	U	b
RF-SE-07-XX	Mercury	0.091	mg/kg	U	b
RF-SE-08-XX	Silver	0.39	mg/kg	U	b
RF-SE-10-XX	Silver	0.34	mg/kg	U	b
RF-SE-12-XX	Mercury	0.099	mg/kg	U	b
RF-SE-23-XX	Copper	0.2	mg/kg	U	b
RF-SE-23-XX	Zinc	0.6	mg/kg	U	b
RF-SE-33-XX	Silver	0.33	mg/kg	U	b
RF-SE-36-XX	Mercury	0.081	mg/kg	U	b
RF-SE-36-XX	Selenium	1	mg/kg	U	b
RF-SE-45-XX	Cadmium	0.52	mg/kg	U	b
RF-SE-53-XX	Cadmium	0.57	mg/kg	U	b
SB-SO-03-XX	Antimony	1.2	mg/kg	UJ	b, e
SB-SO-12-XX	Silver	2.1	mg/kg	UJ	b
SB-SO-13-XX	Silver	2.2	mg/kg	UJ	b
SB-SO-15-XX	Silver	1.6	mg/kg	UJ	b
SB-SO-17-XX	Silver	2.3	mg/kg	UJ	b, e
SB-SO-18-XX	Antimony	1.2	mg/kg	UJ	b, e
SB-SO-30-XX	Selenium	1.3	mg/kg	J+	b
SB-SO-32-XX	Silver	0.1	mg/kg	UJ	b, e
SB-SO-37-XX	Silver	2	mg/kg	UJ	b
SB-SO-46-XX	Silver	2.2	mg/kg	UJ	b, e
SB-SO-48-XX	Silver	0.1	mg/kg	UJ	b, e
SB-SO-53-XX	Antimony	1.2	mg/kg	UJ	b, e
TL-SE-01-XX	Mercury	0.074	mg/kg	U	b
TL-SE-03-XX	Mercury	0.32	mg/kg	J-	b
TL-SE-03-XX	Silver	0.94	mg/kg	U	b
TL-SE-04-XX	Mercury	0.26	mg/kg	J-	b
TL-SE-10-XX	Mercury	0.19	mg/kg	J-	b
TL-SE-11-XX	Mercury	0.021	mg/kg	U	b
TL-SE-12-XX	Mercury	0.22	mg/kg	J-	b
TL-SE-14-XX	Mercury	0.08	mg/kg	U	b
TL-SE-15-XX	Mercury	0.28	mg/kg	J-	b

TABLE 3: DATA QUALIFICATION: LABORATORY METHOD BLANK CONTAMINATION (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
TL-SE-15-XX	Silver	1	mg/kg	U	b
TL-SE-18-XX	Mercury	0.025	mg/kg	U	b
TL-SE-19-XX	Mercury	0.32	mg/kg	J-	b
TL-SE-19-XX	Silver	1.1	mg/kg	U	b
TL-SE-20-XX	Mercury	0.26	mg/kg	J-	b
TL-SE-22-XX	Mercury	0.082	mg/kg	U	b
TL-SE-23-XX	Mercury	0.41	mg/kg	J-	b
TL-SE-23-XX	Silver	1.3	mg/kg	U	b
TL-SE-24-XX	Mercury	0.26	mg/kg	J-	b
TL-SE-24-XX	Silver	1.3	mg/kg	U	b
TL-SE-25-XX	Mercury	0.44	mg/kg	J-	b
TL-SE-25-XX	Silver	0.94	mg/kg	U	b
TL-SE-26-XX	Mercury	0.24	mg/kg	J-	b
TL-SE-27-XX	Mercury	0.02	mg/kg	U	b
TL-SE-29-XX	Mercury	0.076	mg/kg	U	b
TL-SE-31-XX	Mercury	0.57	mg/kg	J-	b
TL-SE-31-XX	Silver	1.2	mg/kg	U	b
WS-SO-06-XX	Mercury	0.07	mg/kg	U	b
WS-SO-08-XX	Mercury	0.063	mg/kg	U	b
WS-SO-10-XX	Mercury	0.058	mg/kg	U	b
WS-SO-12-XX	Mercury	0.068	mg/kg	UJ	b, e
WS-SO-17-XX	Mercury	0.069	mg/kg	UJ	b, e
WS-SO-20-XX	Mercury	0.06	mg/kg	U	b
WS-SO-23-XX	Mercury	0.05	mg/kg	U	b
WS-SO-30-XX	Mercury	0.069	mg/kg	UJ	b, e
WS-SO-31-XX	Selenium	1.2	mg/kg	U	b
WS-SO-35-XX	Mercury	0.071	mg/kg	UJ	b, e

Notes:
mg/kg = Milligrams per kilogram
b = Data were qualified based on blank contamination
e = Data were additionally qualified based on matrix spike/matrix spike duplicate exceedances
J+ = Result is estimated and potentially biased high
J- = Result is estimated and potentially biased low
UJ = Result is undetected at estimated quantitation limits

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
AS-SO-01-XX	Antimony	3.8	mg/kg	J-	e
AS-SO-02-XX	Antimony	<2.6	mg/kg	UJ	e
AS-SO-03-XX	Mercury	3.7	mg/kg	J-	e
AS-SO-03-XX	Silver	480	mg/kg	J-	e
AS-SO-04-XX	Antimony	<6.4	mg/kg	UJ	e
AS-SO-05-XX	Mercury	2.5	mg/kg	J-	e
AS-SO-05-XX	Silver	330	mg/kg	J-	e
AS-SO-06-XX	Antimony	2.4	mg/kg	UJ	b, e
AS-SO-07-XX	Antimony	3.6	mg/kg	J-	e
AS-SO-08-XX	Mercury	2.5	mg/kg	J-	e
AS-SO-08-XX	Silver	280	mg/kg	J-	e
AS-SO-09-XX	Antimony	<2.6	mg/kg	UJ	e
AS-SO-10-XX	Antimony	1.9	mg/kg	J-	e
AS-SO-11-XX	Antimony	3.7	mg/kg	J-	e
AS-SO-12-XX	Antimony	<2.6	mg/kg	UJ	e
AS-SO-13-XX	Antimony	2.4	mg/kg	UJ	b, e
BN-SO-01-XX	Antimony	<1.3	mg/kg	UJ	e
BN-SO-01-XX	Silver	<1.3	mg/kg	UJ	e
BN-SO-05-XX	Antimony	160	mg/kg	J-	e
BN-SO-07-XX	Antimony	110	mg/kg	J-	e
BN-SO-07-XX	Silver	990	mg/kg	J+	e
BN-SO-09-XX	Antimony	750	mg/kg	J-	e
BN-SO-09-XX	Silver	100	mg/kg	J-	e
BN-SO-10-XX	Antimony	<1.3	mg/kg	UJ	e
BN-SO-10-XX	Silver	<1.3	mg/kg	UJ	e
BN-SO-11-XX	Antimony	4	mg/kg	J-	e
BN-SO-11-XX	Silver	140	mg/kg	J-	e
BN-SO-12-XX	Antimony	750	mg/kg	J-	e
BN-SO-12-XX	Silver	210	mg/kg	J-	e
BN-SO-14-XX	Antimony	3.5	mg/kg	J-	e
BN-SO-14-XX	Silver	140	mg/kg	J-	e
BN-SO-15-XX	Antimony	<1.3	mg/kg	UJ	e
BN-SO-15-XX	Silver	<1.3	mg/kg	UJ	e
BN-SO-16-XX	Antimony	120	mg/kg	J-	e
BN-SO-16-XX	Arsenic	1100	mg/kg	J+	e
BN-SO-19-XX	Antimony	150	mg/kg	J-	e
BN-SO-21-XX	Antimony	150	mg/kg	J-	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
BN-SO-21-XX	Arsenic	1300	mg/kg	J+	e
BN-SO-23-XX	Antimony	<1.2	mg/kg	UJ	e
BN-SO-23-XX	Silver	130	mg/kg	J-	e
BN-SO-24-XX	Antimony	810	mg/kg	J-	e
BN-SO-24-XX	Silver	140	mg/kg	J-	e
BN-SO-25-XX	Antimony	82	mg/kg	J-	e, j
BN-SO-25-XX	Arsenic	700	mg/kg	J	e, j
BN-SO-26-XX	Antimony	150	mg/kg	J-	e
BN-SO-29-XX	Antimony	150	mg/kg	J-	e
BN-SO-32-XX	Antimony	160	mg/kg	J-	e
BN-SO-33-XX	Antimony	100	mg/kg	J-	e
CN-SO-01-XX	Antimony	13	mg/kg	J-	e
CN-SO-02-XX	Mercury	270	mg/kg	J-	e
CN-SO-03-XX	Mercury	34	mg/kg	J-	e
CN-SO-04-XX	Antimony	13	mg/kg	J-	e
CN-SO-05-XX	Mercury	280	mg/kg	J-	e
CN-SO-06-XX	Mercury	40	mg/kg	J-	e
CN-SO-07-XX	Mercury	36	mg/kg	J-	e
CN-SO-08-XX	Antimony	15	mg/kg	J-	e
CN-SO-09-XX	Mercury	260	mg/kg	J-	e
CN-SO-10-XX	Antimony	13	mg/kg	J-	e
CN-SO-11-XX	Antimony	17	mg/kg	J-	e
KP-SE-01-XX	Lead	310	mg/kg	J-	e
KP-SE-01-XX	Silver	<0.26	mg/kg	UJ	e
KP-SE-08-XX	Lead	300	mg/kg	J-	e
KP-SE-08-XX	Silver	<0.27	mg/kg	UJ	e
KP-SE-11-XX	Lead	310	mg/kg	J-	e
KP-SE-11-XX	Silver	<0.27	mg/kg	UJ	e
KP-SE-12-XX	Lead	320	mg/kg	J-	e
KP-SE-12-XX	Silver	<0.26	mg/kg	UJ	e
KP-SE-14-XX	Lead	680	mg/kg	J-	e, j
KP-SE-14-XX	Silver	<0.26	mg/kg	UJ	e
KP-SE-17-XX	Lead	300	mg/kg	J-	e
KP-SE-17-XX	Silver	<0.27	mg/kg	UJ	e
KP-SE-25-XX	Lead	310	mg/kg	J-	e
KP-SE-25-XX	Silver	<0.27	mg/kg	UJ	e
KP-SE-30-XX	Lead	300	mg/kg	J-	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
KP-SE-30-XX	Silver	<0.27	mg/kg	UJ	e
KP-SO-04-XX	Antimony	94	mg/kg	J+	e
KP-SO-06-XX	Antimony	8.1	mg/kg	J+	e
KP-SO-07-XX	Antimony	17	mg/kg	J+	e
KP-SO-10-XX	Antimony	6.1	mg/kg	J+	e
KP-SO-13-XX	Antimony	16	mg/kg	J+	e
KP-SO-15-XX	Antimony	6.3	mg/kg	J+	e
KP-SO-16-XX	Antimony	93	mg/kg	J+	e
KP-SO-18-XX	Antimony	6.7	mg/kg	J+	e
KP-SO-20-XX	Antimony	19	mg/kg	J+	e
KP-SO-22-XX	Antimony	8.3	mg/kg	J+	e
KP-SO-23-XX	Antimony	86	mg/kg	J+	e
KP-SO-24-XX	Antimony	17	mg/kg	J+	e
KP-SO-26-XX	Antimony	90	mg/kg	J+	e
KP-SO-27-XX	Antimony	15	mg/kg	J+	e
KP-SO-29-XX	Antimony	18	mg/kg	J+	e
KP-SO-32-XX	Antimony	16	mg/kg	J+	e
LV-SE-01-XX	Antimony	<1.5	mg/kg	UJ	e
LV-SE-02-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-02-XX	Lead	20	mg/kg	J-	e
LV-SE-02-XX	Silver	<1.3	mg/kg	UJ	e
LV-SE-05-XX	Mercury	2.6	mg/kg	J-	e
LV-SE-06-XX	Mercury	610	mg/kg	J-	e
LV-SE-07-XX	Antimony	<6.7	mg/kg	UJ	e
LV-SE-08-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-09-XX	Lead	14	mg/kg	J-	e
LV-SE-10-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-10-XX	Lead	25	mg/kg	J-	e
LV-SE-10-XX	Silver	<1.3	mg/kg	UJ	e
LV-SE-11-XX	Antimony	<1.4	mg/kg	UJ	e
LV-SE-12-XX	Lead	19	mg/kg	J-	e
LV-SE-13-XX	Mercury	640	mg/kg	J-	e
LV-SE-14-XX	Antimony	<1.5	mg/kg	UJ	e
LV-SE-15-XX	Antimony	290	mg/kg	J+	e
LV-SE-15-XX	Silver	300	mg/kg	J-	e
LV-SE-16-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-17-XX	Antimony	280	mg/kg	J+	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
LV-SE-17-XX	Lead	17	mg/kg	J-	e
LV-SE-17-XX	Silver	200	mg/kg	J-	e
LV-SE-18-XX	Antimony	<6.7	mg/kg	UJ	e
LV-SE-19-XX	Lead	17	mg/kg	J-	e
LV-SE-20-XX	Antimony	140	mg/kg	J+	e
LV-SE-20-XX	Silver	75	mg/kg	J-	e
LV-SE-21-XX	Antimony	<1.5	mg/kg	UJ	e
LV-SE-22-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-22-XX	Lead	22	mg/kg	J-	e
LV-SE-22-XX	Silver	<1.3	mg/kg	UJ	e
LV-SE-23-XX	Antimony	<6.6	mg/kg	UJ	e
LV-SE-24-XX	Antimony	<1.5	mg/kg	UJ	e
LV-SE-25-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-25-XX	Lead	23	mg/kg	J-	e
LV-SE-25-XX	Silver	<1.3	mg/kg	UJ	e
LV-SE-26-XX	Lead	25	mg/kg	J-	e
LV-SE-27-XX	Lead	16	mg/kg	J-	e
LV-SE-28-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-29-XX	Antimony	<1.4	mg/kg	UJ	e
LV-SE-30-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-31-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-31-XX	Lead	49	mg/kg	J-	e
LV-SE-31-XX	Silver	<1.3	mg/kg	UJ	e
LV-SE-32-XX	Antimony	<1.4	mg/kg	UJ	e
LV-SE-33-XX	Lead	21	mg/kg	J-	e
LV-SE-35-XX	Antimony	<1.3	mg/kg	UJ	e
LV-SE-35-XX	Lead	22	mg/kg	J-	e
LV-SE-35-XX	Silver	<1.3	mg/kg	UJ	e
LV-SE-36-XX	Lead	21	mg/kg	J-	e
LV-SE-38-XX	Lead	15	mg/kg	J-	e
LV-SE-39-XX	Lead	22	mg/kg	J-	e
LV-SE-41-XX	Mercury	610	mg/kg	J-	e
LV-SE-42-XX	Lead	22	mg/kg	J-	e
LV-SE-43-XX	Antimony	160	mg/kg	J+	e
LV-SE-43-XX	Silver	60	mg/kg	J-	e
LV-SE-45-XX	Antimony	<6.7	mg/kg	UJ	e
LV-SE-47-XX	Antimony	<1.3	mg/kg	UJ	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
LV-SE-48-XX	Antimony	<6.6	mg/kg	UJ	e
LV-SE-50-XX	Lead	24	mg/kg	J-	e
LV-SE-51-XX	Antimony	210	mg/kg	J+	e
LV-SE-51-XX	Silver	250	mg/kg	J-	e
LV-SO-03-XX	Mercury	48	mg/kg	J-	e
LV-SO-03-XX	Silver	210	mg/kg	J-	e
LV-SO-04-XX	Mercury	130	mg/kg	J-	e
LV-SO-04-XX	Silver	<1.2	mg/kg	UJ	e
LV-SO-34-XX	Mercury	130	mg/kg	J-	e
LV-SO-34-XX	Silver	<1.2	mg/kg	UJ	e
LV-SO-37-XX	Mercury	130	mg/kg	J-	e
LV-SO-40-XX	Mercury	46	mg/kg	J-	e
LV-SO-40-XX	Silver	210	mg/kg	J-	e
LV-SO-49-XX	Mercury	52	mg/kg	J-	e
LV-SO-49-XX	Silver	220	mg/kg	J-	e
RF-SE-02-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-03-XX	Antimony	<1.2	mg/kg	UJ	e
RF-SE-04-XX	Antimony	3.2	mg/kg	J+	e
RF-SE-04-XX	Silver	12	mg/kg	J-	e
RF-SE-05-XX	Antimony	4.1	mg/kg	J+	e
RF-SE-05-XX	Silver	7.4	mg/kg	J-	e
RF-SE-06-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-13-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-14-XX	Antimony	4.4	mg/kg	J+	e
RF-SE-14-XX	Silver	13	mg/kg	J-	e
RF-SE-15-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-19-XX	Antimony	3.7	mg/kg	J+	e
RF-SE-19-XX	Silver	14	mg/kg	J-	e
RF-SE-22-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-24-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-25-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-26-XX	Antimony	2.2	mg/kg	J+	e
RF-SE-26-XX	Silver	7.2	mg/kg	J-	e
RF-SE-27-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-28-XX	Antimony	<1.2	mg/kg	UJ	e
RF-SE-30-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-31-XX	Antimony	<1.3	mg/kg	UJ	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
RF-SE-32-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-34-XX	Antimony	2.9	mg/kg	J+	e
RF-SE-34-XX	Silver	10	mg/kg	J-	e
RF-SE-38-XX	Antimony	<1.2	mg/kg	UJ	e
RF-SE-39-XX	Antimony	2.9	mg/kg	J+	e
RF-SE-39-XX	Silver	8.2	mg/kg	J-	e
RF-SE-42-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-43-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-44-XX	Antimony	2.7	mg/kg	J+	e
RF-SE-44-XX	Silver	7.2	mg/kg	J-	e
RF-SE-45-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-49-XX	Antimony	<1.2	mg/kg	UJ	e
RF-SE-52-XX	Antimony	3.4	mg/kg	J+	e
RF-SE-52-XX	Silver	11	mg/kg	J-	e
RF-SE-53-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-55-XX	Antimony	<1.2	mg/kg	UJ	e
RF-SE-56-XX	Antimony	3.5	mg/kg	J+	e
RF-SE-56-XX	Silver	8.3	mg/kg	J-	e
RF-SE-57-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-58-XX	Antimony	<1.3	mg/kg	UJ	e
RF-SE-59-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-01-XX	Antimony	180	mg/kg	J	e
SB-SO-02-XX	Antimony	44	mg/kg	J-	e, j
SB-SO-02-XX	Silver	<1.2	mg/kg	UJ	e
SB-SO-03-XX	Antimony	1.2	mg/kg	UJ	b, e
SB-SO-04-XX	Silver	<1.3	mg/kg	UJ	e
SB-SO-05-XX	Antimony	1.6	mg/kg	J-	e
SB-SO-06-XX	Antimony	1.7	mg/kg	J-	e
SB-SO-07-XX	Antimony	45	mg/kg	J	e
SB-SO-08-XX	Antimony	5.4	mg/kg	J-	e
SB-SO-09-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-09-XX	Silver	160	mg/kg	J-	e
SB-SO-10-XX	Antimony	62	mg/kg	J	e
SB-SO-11-XX	Antimony	5.7	mg/kg	J-	e
SB-SO-12-XX	Antimony	620	mg/kg	J	e
SB-SO-13-XX	Antimony	430	mg/kg	J	e
SB-SO-14-XX	Antimony	4.1	mg/kg	J-	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
SB-SO-15-XX	Antimony	600	mg/kg	J-	j, e
SB-SO-16-XX	Antimony	170	mg/kg	J	e
SB-SO-17-XX	Antimony	800	mg/kg	J+	e
SB-SO-17-XX	Silver	2.3	mg/kg	UJ	b, e
SB-SO-18-XX	Antimony	1.2	mg/kg	UJ	b, e
SB-SO-19-XX	Antimony	310	mg/kg	J	e
SB-SO-20-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-20-XX	Silver	140	mg/kg	J-	e
SB-SO-21-XX	Antimony	4.9	mg/kg	J	e
SB-SO-22-XX	Antimony	10	mg/kg	J	e, j
SB-SO-23-XX	Antimony	48	mg/kg	J-	e
SB-SO-23-XX	Silver	<0.26	mg/kg	UJ	e
SB-SO-24-XX	Antimony	180	mg/kg	J	e
SB-SO-25-XX	Antimony	6.8	mg/kg	J+	e
SB-SO-26-XX	Antimony	61	mg/kg	J	e
SB-SO-27-XX	Antimony	6.7	mg/kg	J+	e
SB-SO-28-XX	Antimony	42	mg/kg	J-	e
SB-SO-28-XX	Silver	<0.26	mg/kg	UJ	e
SB-SO-29-XX	Silver	<1.2	mg/kg	UJ	e
SB-SO-30-XX	Antimony	3.2	mg/kg	J-	e
SB-SO-31-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-31-XX	Silver	160	mg/kg	J-	e, j
SB-SO-32-XX	Antimony	46	mg/kg	J-	e
SB-SO-32-XX	Silver	0.1	mg/kg	UJ	b, e
SB-SO-33-XX	Antimony	350	mg/kg	J	e
SB-SO-33-XX	Silver	2	mg/kg	J	e
SB-SO-34-XX	Silver	<1.3	mg/kg	UJ	e
SB-SO-35-XX	Antimony	6	mg/kg	J+	e
SB-SO-36-XX	Silver	<1.2	mg/kg	UJ	e
SB-SO-37-XX	Antimony	340	mg/kg	J	e
SB-SO-38-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-39-XX	Antimony	4.7	mg/kg	J-	e
SB-SO-40-XX	Antimony	2.2	mg/kg	J-	e
SB-SO-41-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-42-XX	Antimony	4.6	mg/kg	J-	e
SB-SO-43-XX	Antimony	40	mg/kg	J-	e
SB-SO-43-XX	Silver	<0.26	mg/kg	UJ	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
SB-SO-44-XX	Antimony	6.8	mg/kg	J+	e
SB-SO-45-XX	Antimony	180	mg/kg	J	e
SB-SO-45-XX	Silver	2.1	mg/kg	J-	e
SB-SO-46-XX	Antimony	740	mg/kg	J+	e
SB-SO-46-XX	Silver	2.2	mg/kg	UJ	b, e
SB-SO-47-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-48-XX	Antimony	39	mg/kg	J-	e
SB-SO-48-XX	Silver	0.1	mg/kg	UJ	b, e
SB-SO-49-XX	Silver	<1.2	mg/kg	UJ	e
SB-SO-50-XX	Antimony	57	mg/kg	J	e
SB-SO-51-XX	Antimony	<1.3	mg/kg	UJ	e
SB-SO-52-XX	Antimony	150	mg/kg	J	e
SB-SO-53-XX	Antimony	1.2	mg/kg	UJ	b, e
SB-SO-54-XX	Lead	5.2	mg/kg	J-	e
SB-SO-54-XX	Silver	<0.5	mg/kg	UJ	e
SB-SO-55-XX	Antimony	340	mg/kg	J	e
SB-SO-55-XX	Silver	2.2	mg/kg	J	e
SB-SO-56-XX	Silver	<1.2	mg/kg	UJ	e
TL-SE-01-XX	Antimony	<1.2	mg/kg	UJ	e
TL-SE-01-XX	Lead	48	mg/kg	J-	e
TL-SE-01-XX	Silver	5.7	mg/kg	J-	e
TL-SE-05-XX	Antimony	100	mg/kg	J+	e
TL-SE-05-XX	Silver	180	mg/kg	J-	e
TL-SE-09-XX	Antimony	100	mg/kg	J+	e
TL-SE-09-XX	Silver	170	mg/kg	J-	e
TL-SE-11-XX	Antimony	<1.2	mg/kg	UJ	e
TL-SE-11-XX	Lead	54	mg/kg	J-	e
TL-SE-11-XX	Silver	5.5	mg/kg	J-	e
TL-SE-13-XX	Antimony	95	mg/kg	J+	j, e
TL-SE-13-XX	Silver	160	mg/kg	J	j, e
TL-SE-14-XX	Antimony	<1.2	mg/kg	UJ	e
TL-SE-14-XX	Lead	50	mg/kg	J-	e
TL-SE-14-XX	Silver	5.7	mg/kg	J-	e
TL-SE-18-XX	Antimony	<1.2	mg/kg	UJ	e
TL-SE-18-XX	Lead	46	mg/kg	J-	e
TL-SE-18-XX	Silver	6.3	mg/kg	J-	e
TL-SE-22-XX	Antimony	<1.2	mg/kg	UJ	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
TL-SE-22-XX	Lead	54	mg/kg	J-	e
TL-SE-22-XX	Silver	6.5	mg/kg	J-	e
TL-SE-27-XX	Antimony	<1.2	mg/kg	UJ	e
TL-SE-27-XX	Lead	51	mg/kg	J-	e
TL-SE-27-XX	Silver	7.8	mg/kg	J-	e
TL-SE-29-XX	Antimony	<1.2	mg/kg	UJ	e
TL-SE-29-XX	Lead	51	mg/kg	J-	e
TL-SE-29-XX	Silver	5.9	mg/kg	J-	e
WS-SO-01-XX	Antimony	41	mg/kg	J-	e
WS-SO-01-XX	Mercury	5.8	mg/kg	J	e, j
WS-SO-01-XX	Silver	69	mg/kg	J-	e
WS-SO-02-XX	Antimony	130	mg/kg	J-	e
WS-SO-02-XX	Silver	150	mg/kg	J-	e
WS-SO-03-XX	Antimony	8.9	mg/kg	J-	e
WS-SO-03-XX	Mercury	0.86	mg/kg	J-	e
WS-SO-04-XX	Antimony	45	mg/kg	J-	e
WS-SO-04-XX	Silver	76	mg/kg	J-	e
WS-SO-05-XX	Antimony	8.6	mg/kg	J-	e
WS-SO-05-XX	Silver	0.76	mg/kg	J-	e
WS-SO-07-XX	Silver	400	mg/kg	J-	e
WS-SO-09-XX	Antimony	7.1	mg/kg	J-	e
WS-SO-09-XX	Mercury	0.89	mg/kg	J-	e
WS-SO-10-XX	Silver	<1.3	mg/kg	UJ	e
WS-SO-11-XX	Silver	340	mg/kg	J-	e
WS-SO-12-XX	Antimony	<1.3	mg/kg	UJ	e
WS-SO-12-XX	Mercury	0.068	mg/kg	UJ	b, e
WS-SO-13-XX	Antimony	200	mg/kg	J-	e
WS-SO-13-XX	Silver	170	mg/kg	J-	e
WS-SO-14-XX	Antimony	8.4	mg/kg	J-	e
WS-SO-14-XX	Mercury	0.74	mg/kg	J-	e
WS-SO-15-XX	Antimony	48	mg/kg	J-	e
WS-SO-15-XX	Silver	90	mg/kg	J-	e
WS-SO-16-XX	Antimony	110	mg/kg	J-	e
WS-SO-16-XX	Silver	150	mg/kg	J-	e
WS-SO-17-XX	Antimony	<1.3	mg/kg	UJ	e
WS-SO-17-XX	Mercury	0.069	mg/kg	UJ	b, e
WS-SO-18-XX	Antimony	130	mg/kg	J-	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Validation Code
WS-SO-18-XX	Silver	140	mg/kg	J-	e
WS-SO-19-XX	Antimony	150	mg/kg	J-	e
WS-SO-19-XX	Silver	160	mg/kg	J-	e
WS-SO-20-XX	Silver	<1.3	mg/kg	UJ	e
WS-SO-21-XX	Antimony	120	mg/kg	J-	e
WS-SO-21-XX	Silver	150	mg/kg	J-	e
WS-SO-22-XX	Antimony	41	mg/kg	J-	e
WS-SO-22-XX	Silver	72	mg/kg	J-	e
WS-SO-23-XX	Silver	<1.3	mg/kg	UJ	e
WS-SO-24-XX	Antimony	97	mg/kg	J-	e
WS-SO-24-XX	Silver	140	mg/kg	J-	e
WS-SO-25-XX	Silver	450	mg/kg	J-	e
WS-SO-26-XX	Antimony	7.6	mg/kg	J-	e
WS-SO-26-XX	Mercury	0.83	mg/kg	J-	e
WS-SO-27-XX	Antimony	<1.3	mg/kg	UJ	e
WS-SO-27-XX	Mercury	0.11	mg/kg	J-	e
WS-SO-28-XX	Antimony	120	mg/kg	J-	e
WS-SO-28-XX	Silver	130	mg/kg	J-	e
WS-SO-29-XX	Antimony	120	mg/kg	J-	e
WS-SO-29-XX	Silver	140	mg/kg	J-	e
WS-SO-30-XX	Antimony	1.2	mg/kg	J-	e
WS-SO-30-XX	Mercury	0.069	mg/kg	UJ	b, e
WS-SO-31-XX	Antimony	7.2	mg/kg	J-	e
WS-SO-31-XX	Mercury	0.85	mg/kg	J-	e
WS-SO-32-XX	Antimony	190	mg/kg	J-	e
WS-SO-32-XX	Silver	190	mg/kg	J-	e
WS-SO-33-XX	Antimony	6.9	mg/kg	J-	e
WS-SO-33-XX	Mercury	0.87	mg/kg	J-	e
WS-SO-34-XX	Antimony	45	mg/kg	J-	e
WS-SO-34-XX	Silver	78	mg/kg	J-	e
WS-SO-35-XX	Antimony	<1.3	mg/kg	UJ	e
WS-SO-35-XX	Mercury	0.071	mg/kg	UJ	b, e
WS-SO-36-XX	Antimony	120	mg/kg	J-	e
WS-SO-36-XX	Silver	120	mg/kg	J-	e
WS-SO-37-XX	Antimony	120	mg/kg	J-	e
WS-SO-37-XX	Silver	140	mg/kg	J-	e

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES (Continued)

Notes:
< = Less than
mg/kg = Milligram per kilogram
b = Data were qualified based on blank contamination
e = Data were additionally qualified based on matrix spike/matrix spike duplicate exceedances
j = Data were additionally qualified based on serial dilution exceedances
J = Result is estimated and biased could not be determined
J+ = Result is estimated and potentially biased high
J- = Result is estimated and potentially biased low
UJ = Result is undetected at estimated quantitation limit

TABLE 5: DATA QUALIFICATION: SERIAL DILUTION EXCEEDANCES

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
AS-SO-09-XX	Arsenic	25	mg/kg	J-	j
AS-SO-09-XX	Cadmium	100	mg/kg	J-	j
AS-SO-09-XX	Chromium	390	mg/kg	J-	j
AS-SO-09-XX	Copper	250	mg/kg	J-	j
AS-SO-09-XX	Iron	94000	mg/kg	J-	j
AS-SO-09-XX	Lead	3200	mg/kg	J-	j
AS-SO-09-XX	Nickel	170	mg/kg	J-	j
AS-SO-09-XX	Silver	9.6	mg/kg	J-	j
AS-SO-09-XX	Vanadium	65	mg/kg	J-	j
AS-SO-09-XX	Zinc	6800	mg/kg	J-	j
BN-SO-11-XX	Mercury	24	mg/kg	J-	j
BN-SO-25-XX	Antimony	82	mg/kg	J-	e, j
BN-SO-25-XX	Arsenic	700	mg/kg	J	e, j
BN-SO-25-XX	Cadmium	370	mg/kg	J-	j
BN-SO-25-XX	Chromium	64	mg/kg	J-	j
BN-SO-25-XX	Copper	930	mg/kg	J-	j
BN-SO-25-XX	Iron	16000	mg/kg	J-	j
BN-SO-25-XX	Lead	5400	mg/kg	J-	j
BN-SO-25-XX	Nickel	88	mg/kg	J-	j
BN-SO-25-XX	Selenium	19	mg/kg	J-	j
BN-SO-25-XX	Silver	48	mg/kg	J-	j
BN-SO-25-XX	Vanadium	28	mg/kg	J-	j
BN-SO-25-XX	Zinc	2900	mg/kg	J-	j
KP-SE-14-XX	Antimony	11	mg/kg	J-	j
KP-SE-14-XX	Chromium	46	mg/kg	J-	j
KP-SE-14-XX	Copper	2.7	mg/kg	J+	j
KP-SE-14-XX	Iron	520	mg/kg	J-	j
KP-SE-14-XX	Lead	680	mg/kg	J-	e, j
KP-SE-14-XX	Nickel	23	mg/kg	J-	j
LV-SE-29-XX	Lead	7.2	mg/kg	J+	j
LV-SE-29-XX	Mercury	1.5	mg/kg	J-	j
LV-SE-35-XX	Arsenic	31	mg/kg	J-	j
LV-SE-35-XX	Chromium	74	mg/kg	J-	j
LV-SE-35-XX	Iron	24000	mg/kg	J-	j
LV-SE-35-XX	Nickel	170	mg/kg	J-	j
LV-SE-35-XX	Vanadium	55	mg/kg	J-	j
LV-SE-35-XX	Zinc	67	mg/kg	J-	j
LV-SO-34-XX	Antimony	870	mg/kg	J-	j

TABLE 5: DATA QUALIFICATIONS: SERIAL DILUTION EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
LV-SO-34-XX	Arsenic	110	mg/kg	J-	j
LV-SO-34-XX	Cadmium	2300	mg/kg	J-	j
LV-SO-34-XX	Chromium	2200	mg/kg	J-	j
LV-SO-34-XX	Iron	20000	mg/kg	J-	j
LV-SO-34-XX	Lead	3700	mg/kg	J-	j
LV-SO-34-XX	Nickel	1900	mg/kg	J-	j
LV-SO-34-XX	Selenium	220	mg/kg	J-	j
LV-SO-34-XX	Vanadium	230	mg/kg	J-	j
LV-SO-34-XX	Zinc	48	mg/kg	J-	j
RF-SE-16-XX	Antimony	85	mg/kg	J-	j
RF-SE-16-XX	Arsenic	72	mg/kg	J-	j
RF-SE-16-XX	Cadmium	310	mg/kg	J-	j
RF-SE-16-XX	Chromium	820	mg/kg	J-	j
RF-SE-16-XX	Copper	73	mg/kg	J-	j
RF-SE-16-XX	Iron	16000	mg/kg	J-	j
RF-SE-16-XX	Lead	24	mg/kg	J-	j
RF-SE-16-XX	Nickel	1700	mg/kg	J-	j
RF-SE-16-XX	Silver	130	mg/kg	J-	j
RF-SE-16-XX	Vanadium	32	mg/kg	J-	j
RF-SE-16-XX	Zinc	760	mg/kg	J-	j
RF-SE-24-XX	Arsenic	130	mg/kg	J+	j
RF-SE-24-XX	Cadmium	6.5	mg/kg	J+	j
RF-SE-24-XX	Chromium	74	mg/kg	J+	j
RF-SE-24-XX	Copper	860	mg/kg	J+	j
RF-SE-24-XX	Iron	24000	mg/kg	J+	j
RF-SE-24-XX	Lead	410	mg/kg	J+	j
RF-SE-24-XX	Nickel	170	mg/kg	J+	j
RF-SE-24-XX	Silver	3.8	mg/kg	J+	j
RF-SE-24-XX	Vanadium	46	mg/kg	J+	j
RF-SE-24-XX	Zinc	1400	mg/kg	J-	j
SB-SO-02-XX	Antimony	44	mg/kg	J-	e, j
SB-SO-02-XX	Arsenic	23	mg/kg	J-	j
SB-SO-02-XX	Lead	22	mg/kg	J-	j
SB-SO-02-XX	Mercury	130	mg/kg	J+	j
SB-SO-15-XX	Antimony	600	mg/kg	J-	j, e
SB-SO-15-XX	Arsenic	170	mg/kg	J-	j
SB-SO-15-XX	Chromium	91	mg/kg	J-	j
SB-SO-15-XX	Copper	30	mg/kg	J-	j
SB-SO-15-XX	Iron	51000	mg/kg	J-	j

TABLE 5: DATA QUALIFICATIONS: SERIAL DILUTION EXCEEDANCES (Continued)

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
SB-SO-15-XX	Lead	40	mg/kg	J-	j
SB-SO-15-XX	Nickel	100	mg/kg	J-	j
SB-SO-15-XX	Vanadium	52	mg/kg	J-	j
SB-SO-15-XX	Zinc	36	mg/kg	J-	j
SB-SO-22-XX	Antimony	10	mg/kg	J	e, j
SB-SO-22-XX	Zinc	64	mg/kg	J-	j
SB-SO-31-XX	Arsenic	8	mg/kg	J-	j
SB-SO-31-XX	Nickel	3200	mg/kg	J-	j
SB-SO-31-XX	Selenium	28	mg/kg	J-	j
SB-SO-31-XX	Silver	160	mg/kg	J-	e, j
SB-SO-31-XX	Zinc	3900	mg/kg	J-	j
TL-SE-13-XX	Antimony	95	mg/kg	J+	j, e
TL-SE-13-XX	Chromium	36	mg/kg	J+	j
TL-SE-13-XX	Copper	4400	mg/kg	J+	j
TL-SE-13-XX	Iron	22000	mg/kg	J+	j
TL-SE-13-XX	Lead	1100	mg/kg	J+	j
TL-SE-13-XX	Silver	160	mg/kg	J	j, e
TL-SE-13-XX	Vanadium	59	mg/kg	J+	j
WS-SO-01-XX	Mercury	5.8	mg/kg	J	e, j
WS-SO-33-XX	Arsenic	450	mg/kg	J-	j
WS-SO-33-XX	Cadmium	11	mg/kg	J-	j
WS-SO-33-XX	Chromium	120	mg/kg	J-	j
WS-SO-33-XX	Copper	150	mg/kg	J-	j
WS-SO-33-XX	Iron	28000	mg/kg	J-	j
WS-SO-33-XX	Lead	3700	mg/kg	J-	j
WS-SO-33-XX	Nickel	65	mg/kg	J-	j
WS-SO-33-XX	Silver	13	mg/kg	J-	j
WS-SO-33-XX	Vanadium	53	mg/kg	J-	j
WS-SO-33-XX	Zinc	830	mg/kg	J-	j

Notes:
mg/kg = Milligram per kilogram
e = Data were additionally qualified based on matrix spike/matrix spike duplicate exceedances
j = Data were qualified based on serial dilution exceedances
J = Result is estimated and biased could not be determined
J+ = Result is estimated and potentially biased high
J- = Result is estimated and potentially biased low

APPENDIX D

DEVELOPER AND REFERENCE LABORATORY DATA

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
1	KP-SO-06-XX	Reference Laboratory	8.1 J+	1 J-	0.1 U	290	26	1,400	620
1	KP-SO-10-XX	Reference Laboratory	6.1 J+	1 J-	0.1 U	300	26	1,600	560
1	KP-SO-15-XX	Reference Laboratory	6.3 J+	1 J-	0.1 U	340	26	1,600	510
1	KP-SO-18-XX	Reference Laboratory	6.7 J+	1 J-	0.1 U	250	24	1,200	500
1	KP-SO-22-XX	Reference Laboratory	8.3 J+	1 J-	0.1 U	260	29	1,300	650
1	KP-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	521	34	5,161	640
1	KP-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	418	29	5,013	553
1	KP-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	425	24	4,986	542
1	KP-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	419	25	4,905	500
1	KP-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	393	27	4,902	568
2	KP-SO-07-XX	Reference Laboratory	17 J+	2 J-	0.1 U	170	48	990	1,200
2	KP-SO-13-XX	Reference Laboratory	16 J+	1 J-	0.045 U	180	52	980	1,200
2	KP-SO-20-XX	Reference Laboratory	19 J+	2 J-	0.1 U	160	46	910	1,300
2	KP-SO-24-XX	Reference Laboratory	17 J+	1 J-	0.1 U	160	49	900	1,100
2	KP-SO-27-XX	Reference Laboratory	15 J+	1 J-	0.05 U	170	45	970	1,200
2	KP-SO-29-XX	Reference Laboratory	18 J+	2 J-	0.1 U	150	42	870	1,200
2	KP-SO-32-XX	Reference Laboratory	16 J+	2 J-	0.045 U	180	50	970	1,200
2	KP-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	188	62	4,531	1,360
2	KP-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	255	52	4,590	1,366
2	KP-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	206	28	4,396	1,208
2	KP-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	113	48	4,363	1,183
2	KP-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	160	51	4,398	1,246
2	KP-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	215	62	4,460	1,347
2	KP-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	174	39	4,364	1,203
3	KP-SO-04-XX	Reference Laboratory	94 J+	3	0.046 U	180	200	1,300	5,800
3	KP-SO-16-XX	Reference Laboratory	93 J+	3	0.063 U	200	230	1,400	6,100
3	KP-SO-23-XX	Reference Laboratory	86 J+	3	0.048 U	180	190	1,300	5,300
3	KP-SO-26-XX	Reference Laboratory	90 J+	4	0.061 U	210	230	1,500	6,500
3	KP-SO-31-XX	Reference Laboratory	88	28	0.1 U	140	200	1,100	5,700
3	KP-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	11	0	0	202	246	3,753	5,302
3	KP-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	44	0	0	67	217	3,702	4,793
3	KP-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	15	0	8	227	287	3,790	6,042
3	KP-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	19	0	0	175	261	3,699	5,613
3	KP-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	22	0	0	152	251	3,699	5,666

Appendix D. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
1	KP-SO-06-XX	Reference Laboratory	0.06 U	140	0.25 U	0.25 U	2 J	11
1	KP-SO-10-XX	Reference Laboratory	0.03 U	150	0.22 U	0.25 U	2 J	12
1	KP-SO-15-XX	Reference Laboratory	0.03 U	170	0.25 U	0.25 U	2 J	15
1	KP-SO-18-XX	Reference Laboratory	0.02 U	120	0.25 U	0.25 U	2 J	11
1	KP-SO-22-XX	Reference Laboratory	0.03 U	130	0.25 U	0.25 U	2 J	11
1	KP-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	211	3	0	30	0
1	KP-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	152	0	2	0	0
1	KP-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	159	2	0	0	0
1	KP-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	0	149	0	0	0	0
1	KP-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	0	156	5	0	0	0
2	KP-SO-07-XX	Reference Laboratory	0.03 U	87	0.21 U	0.25 U	1 J	26
2	KP-SO-13-XX	Reference Laboratory	0.04 U	90	0.25 U	0.25 U	1 J	24
2	KP-SO-20-XX	Reference Laboratory	0.03 U	79	0.25 U	0.25 U	1 J	25
2	KP-SO-24-XX	Reference Laboratory	0.02 U	78	0.25 U	0.25 U	1 J	22
2	KP-SO-27-XX	Reference Laboratory	0.02 U	87	0.25 U	0.25 U	1 J	24
2	KP-SO-29-XX	Reference Laboratory	0.01 U	73	0.25 U	0.25 U	1 J	22
2	KP-SO-32-XX	Reference Laboratory	0.01 U	88	0.51	0.25 U	1 J	24
2	KP-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	0	104	5	0	0	0
2	KP-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	97	3	0	0	0
2	KP-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	0	85	1	0	16	0
2	KP-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	0	65	6	19	0	0
2	KP-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	0	86	4	0	22	0
2	KP-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	0	97	6	0	35	0
2	KP-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	0	58	5	0	0	0
3	KP-SO-04-XX	Reference Laboratory	0.02 U	93	0.28 U	0.16 J	1 J	45
3	KP-SO-16-XX	Reference Laboratory	0.02 U	100	0.25 U	0.16 J	1 J	47
3	KP-SO-23-XX	Reference Laboratory	0.02 U	91	0.25 U	0.13 J	1 J	41
3	KP-SO-26-XX	Reference Laboratory	0.01 U	110	0.22 U	0.17 J	1 J	52
3	KP-SO-31-XX	Reference Laboratory	0.02 U	68	0.25 U	0.4	2 J	38
3	KP-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	6	29	16	0	0	13
3	KP-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	6	14	10	0	0	22
3	KP-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	21	62	14	0	0	21
3	KP-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	24	57	13	2	0	24
3	KP-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	14	33	14	0	0	29

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
4	KP-SO-02-XX	Reference Laboratory	410	10	0.1	6	780	1,700	18,000
4	KP-SO-03-XX	Reference Laboratory	360	9	0.074 U	5	670	1,600	19,000
4	KP-SO-05-XX	Reference Laboratory	410	12	0.13 U	6	780	2,000	24,000
4	KP-SO-09-XX	Reference Laboratory	420	11	0.094 U	5	780	1,800	22,000
4	KP-SO-21-XX	Reference Laboratory	370	10	0.098 U	5	700	1,700	19,000
4	KP-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	387	379	88	0	1,413	0	25,388
4	KP-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	324	0	49	0	1,029	578	21,341
4	KP-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	349	450	51	0	1,408	218	25,094
4	KP-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	451	439	64	0	1,373	0	25,824
4	KP-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	423	165	50	0	1,243	204	23,675
5	WS-SO-06-XX	Reference Laboratory	1.3 U	48	1.9	120	50	28,000	110
5	WS-SO-08-XX	Reference Laboratory	1.3	45	2	120	47	26,000	71
5	WS-SO-12-XX	Reference Laboratory	1.3 UJ	43	1.8	110	45	25,000	65
5	WS-SO-17-XX	Reference Laboratory	1.3 UJ	47	1.9	120	49	28,000	70
5	WS-SO-27-XX	Reference Laboratory	1.3 UJ	49	2	120	51	28,000	72
5	WS-SO-30-XX	Reference Laboratory	1.2 J-	51	2	130	53	29,000	81
5	WS-SO-35-XX	Reference Laboratory	1.3 UJ	49	2	130	51	28,000	74
5	WS-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	50	0	120	35	21,064	114
5	WS-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	54	0	111	41	21,383	105
5	WS-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	65	0	120	64	21,715	165
5	WS-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	0	49	2	123	54	21,606	133
5	WS-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX	0	50	0	82	56	21,577	122
5	WS-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	0	51	0	124	52	21,242	112
5	WS-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX	0	53	10	128	54	20,973	112
6	WS-SO-03-XX	Reference Laboratory	8.9 J-	500	12	140	170	32,000	4,300
6	WS-SO-05-XX	Reference Laboratory	8.6 J-	440	12	140	160	31,000	4,000
6	WS-SO-09-XX	Reference Laboratory	7.1 J-	480	12	130	160	30,000	4,000
6	WS-SO-14-XX	Reference Laboratory	8.4 J-	430	11	120	150	28,000	3,700
6	WS-SO-26-XX	Reference Laboratory	7.6 J-	520	12	140	160	30,000	4,000
6	WS-SO-31-XX	Reference Laboratory	7.2 J-	520	12	140	170	32,000	4,200
6	WS-SO-33-XX	Reference Laboratory	6.9 J-	450 J-	11 J-	120 J-	150 J-	28,000 J-	3,700 J-
6	WS-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	36	223	25	68	157	22,689	4,078
6	WS-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	13	225	17	49	199	23,745	4,319
6	WS-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX	14	252	13	87	163	23,510	4,108
6	WS-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	0	278	0	171	194	24,077	4,134
6	WS-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX	22	222	17	61	189	22,656	3,964
6	WS-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	5	274	19	178	199	23,510	4,211
6	WS-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX	3	240	20	115	174	23,772	4,119

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
4	KP-SO-02-XX	Reference Laboratory	0.04 U	4	0.42 U	0.82	0 J	100
4	KP-SO-03-XX	Reference Laboratory	0.04 U	3	0.25 U	0.73	0 J	92
4	KP-SO-05-XX	Reference Laboratory	0.04 U	4	0.24 U	0.82	0 J	110
4	KP-SO-09-XX	Reference Laboratory	0.05 U	3	0.25 U	0.84	0 J	110
4	KP-SO-21-XX	Reference Laboratory	0.04 U	4	0.25 U	0.76	0 J	100
4	KP-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	241	0	48	8	140	226
4	KP-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	193	0	41	10	120	183
4	KP-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	197	0	51	1	149	208
4	KP-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	187	0	38	13	123	226
4	KP-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	185	0	49	0	165	217
5	WS-SO-06-XX	Reference Laboratory	0.07 U	61	1.3 U	0.93 J	56	230
5	WS-SO-08-XX	Reference Laboratory	0.06 U	58	1.3 U	0.86 J	52	220
5	WS-SO-12-XX	Reference Laboratory	0.07 UJ	55	1.3 U	0.94 J	49	210
5	WS-SO-17-XX	Reference Laboratory	0.07 UJ	59	1.3 U	0.89 J	56	230
5	WS-SO-27-XX	Reference Laboratory	0.11 J-	61	1.3 U	0.9 J	57	230
5	WS-SO-30-XX	Reference Laboratory	0.07 UJ	65	1.3 U	1 J	58	240
5	WS-SO-35-XX	Reference Laboratory	0.07 UJ	62	1.3 U	1 J	57	240
5	WS-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	0	1	184
5	WS-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	14	40	182
5	WS-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	0	52	191
5	WS-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	7	0	0	164
5	WS-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	8	112	180
5	WS-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	10	42	178
5	WS-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	3	17	70	184
6	WS-SO-03-XX	Reference Laboratory	0.86 J-	75	1.6	15	58	930
6	WS-SO-05-XX	Reference Laboratory	0.76 J-	71	1.3 U	15	57	900
6	WS-SO-09-XX	Reference Laboratory	0.89 J-	70	1.3 U	14	56	870
6	WS-SO-14-XX	Reference Laboratory	0.74 J-	64	1.3 U	13	50	820
6	WS-SO-26-XX	Reference Laboratory	0.83 J-	70	1.3 U	14	56	900
6	WS-SO-31-XX	Reference Laboratory	0.85 J-	72	1.2 U	15	60	950
6	WS-SO-33-XX	Reference Laboratory	0.87 J-	65 J-	1.3 U	13 J-	53 J-	830 J-
6	WS-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	3	32	0	705
6	WS-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	29	22	778
6	WS-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	25	91	839
6	WS-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	0	53	707
6	WS-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	25	93	738
6	WS-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	21	75	721
6	WS-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	11	0	785

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
7	WS-SO-01-XX	Reference Laboratory	41 J-	1900	47	100	590	32,000	18,000
7	WS-SO-04-XX	Reference Laboratory	45 J-	2000	50	94	640	34,000	20,000
7	WS-SO-15-XX	Reference Laboratory	48 J-	2300	56	82	720	37,000	24,000
7	WS-SO-22-XX	Reference Laboratory	41 J-	1900	47	84	620	33,000	17,000
7	WS-SO-34-XX	Reference Laboratory	45 J-	2000	50	91	660	36,000	22,000
7	WS-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	179	2,291	81	69	880	32,450	23,108
7	WS-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	199	2,356	133	124	1,004	32,211	24,076
7	WS-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX	163	2,292	98	260	927	32,329	22,930
7	WS-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	209	2,347	112	197	999	31,903	24,179
7	WS-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX	247	2,326	120	142	853	31,437	22,127
8	WS-SO-02-XX	Reference Laboratory	130 J-	4200	98	49	1300	44,000	35,000
8	WS-SO-16-XX	Reference Laboratory	110 J-	3900	91	59	1300	42,000	24,000
8	WS-SO-18-XX	Reference Laboratory	130 J-	4100	95	63	1300	44,000	37,000
8	WS-SO-21-XX	Reference Laboratory	120 J-	3900	90	43	1200	40,000	43,000
8	WS-SO-24-XX	Reference Laboratory	97 J-	3600	81	54	1100	38,000	27,000
8	WS-SO-29-XX	Reference Laboratory	120 J-	3800	90	51	1200	40,000	42,000
8	WS-SO-37-XX	Reference Laboratory	120 J-	4100	95	63	1300	42,000	26,000
8	WS-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	424	5,305	235	144	1,738	45,478	42,996
8	WS-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	584	5,245	210	55	1,684	44,691	42,525
8	WS-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	331	5,471	245	177	1,804	45,692	44,692
8	WS-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX	581	5,315	208	46	1,543	45,058	40,743
8	WS-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	381	5,219	236	0	1,571	45,091	41,419
8	WS-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	389	5,370	274	66	1,867	45,346	44,808
8	WS-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX	378	5,273	274	255	1,764	44,082	43,896
9	WS-SO-13-XX	Reference Laboratory	200 J-	5800	150	53	1800	47,000	45,000
9	WS-SO-19-XX	Reference Laboratory	150 J-	5000	130	66	1500	39,000	24,000
9	WS-SO-28-XX	Reference Laboratory	120 J-	4200	100	54	1200	33,000	30,000
9	WS-SO-32-XX	Reference Laboratory	190 J-	5500	140	54	1700	44,000	30,000
9	WS-SO-36-XX	Reference Laboratory	120 J-	3800	92	51	1100	30,000	45,000
9	WS-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	438	6,288	303	517	2,089	48,386	51,242
9	WS-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	405	6,288	332	344	2,290	48,914	53,646
9	WS-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	497	6,238	327	427	2,120	45,551	50,967
9	WS-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	329	6,262	347	264	2,201	47,062	52,720
9	WS-SO-36-MX	Oxford Instrument Analytical X-Met 3000TX	546	6,380	316	473	2,000	50,451	51,099

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
7	WS-SO-01-XX	Reference Laboratory	5.8 J	66	1.3 U	69 J-	42	3,000
7	WS-SO-04-XX	Reference Laboratory	6.5	62	1.3 U	76 J-	44	3,100
7	WS-SO-15-XX	Reference Laboratory	5.8	58	1.3 U	90 J-	52	3,400
7	WS-SO-22-XX	Reference Laboratory	4.8	57	1.3 U	72 J-	44	3,000
7	WS-SO-34-XX	Reference Laboratory	5.4	60	1.3 U	78 J-	47	3,200
7	WS-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	88	143	4,672
7	WS-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	111	0	4,356
7	WS-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	80	108	4,210
7	WS-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	40	0	11	93	241	4,263
7	WS-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX	19	0	0	97	289	4,560
8	WS-SO-02-XX	Reference Laboratory	17	57	1.3 U	150 J-	36	6,000
8	WS-SO-16-XX	Reference Laboratory	15	60	1.1 J	150 J-	35	5,700
8	WS-SO-18-XX	Reference Laboratory	17	62	1.9	140 J-	36	5,900
8	WS-SO-21-XX	Reference Laboratory	14	51	1.6	150 J-	33	5,500
8	WS-SO-24-XX	Reference Laboratory	16	54	2.1	140 J-	30	5,200
8	WS-SO-29-XX	Reference Laboratory	15	55	1.7	140 J-	33	5,500
8	WS-SO-37-XX	Reference Laboratory	14	63	3	140 J-	34	5,800
8	WS-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	196	401	8,347
8	WS-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	190	87	8,598
8	WS-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	204	235	8,242
8	WS-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX	51	0	0	228	210	9,927
8	WS-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	59	0	0	194	352	9,077
8	WS-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	25	0	0	185	252	7,958
8	WS-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX	123	0	2	229	211	7,813
9	WS-SO-13-XX	Reference Laboratory	11	75	3.7	170 J-	24	9,000
9	WS-SO-19-XX	Reference Laboratory	12	74	3.7	160 J-	20	7,700
9	WS-SO-28-XX	Reference Laboratory	11	59	2.3	130 J-	16	6,100
9	WS-SO-32-XX	Reference Laboratory	11	73	3.7	190 J-	23	8,500
9	WS-SO-36-XX	Reference Laboratory	13	55	1.7	120 J-	15	5,700
9	WS-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	237	419	11,361
9	WS-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	213	369	9,697
9	WS-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	45	0	0	259	396	11,142
9	WS-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	15	0	11	215	113	10,527
9	WS-SO-36-MX	Oxford Instrument Analytical X-Met 3000TX	70	0	29	259	495	12,534

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
10	BN-SO-01-XX	Reference Laboratory	1.3 UJ	38	0.94	120	32	24,000	63
10	BN-SO-10-XX	Reference Laboratory	1.3 UJ	50	1.2	110	35	24,000	140
10	BN-SO-15-XX	Reference Laboratory	1.3 UJ	34	0.82	110	29	22,000	56
10	BN-SO-18-XX	Reference Laboratory	1.3 U	37	0.89	110	29	22,000	59
10	BN-SO-28-XX	Reference Laboratory	1.5	35	0.87	100	28	22,000	58
10	BN-SO-31-XX	Reference Laboratory	1.3	41	1	140	33	26,000	65
10	BN-SO-35-XX	Reference Laboratory	1.4	37	0.98	120	30	23,000	60
10	BN-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	0	46	0	248	29	20,962	89
10	BN-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	40	0	149	28	18,973	88
10	BN-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	13	50	0	140	38	20,096	83
10	BN-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	0	50	0	152	30	19,717	77
10	BN-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	1	40	5	92	28	20,644	98
10	BN-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX	15	45	0	152	29	19,222	84
10	BN-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX	0	44	0	114	30	20,255	85
11	BN-SO-02-XX	Reference Laboratory	11	140	50	90	170	28,000	840
11	BN-SO-04-XX	Reference Laboratory	9.1	120	42	79	140	24,000	700
11	BN-SO-17-XX	Reference Laboratory	9.3	110	39	79	140	23,000	680
11	BN-SO-22-XX	Reference Laboratory	7.3	98	34	65	110	20,000	590
11	BN-SO-27-XX	Reference Laboratory	9.6	110	39	78	130	24,000	660
11	BN-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	24	114	26	81	166	20,458	802
11	BN-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	23	108	42	182	179	20,212	753
11	BN-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX	59	112	26	68	153	19,726	758
11	BN-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX	42	106	36	122	145	19,970	710
11	BN-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	4	109	45	89	160	20,662	760
12	BN-SO-03-XX	Reference Laboratory	65	620	290	120	840	25,000	4,700
12	BN-SO-06-XX	Reference Laboratory	60	600	280	94	810	24,000	4,500
12	BN-SO-08-XX	Reference Laboratory	57	570	270	100	750	22,000	4,300
12	BN-SO-13-XX	Reference Laboratory	65	320	150	98	410	17,000	2,400
12	BN-SO-20-XX	Reference Laboratory	57	540	260	88	730	22,000	4,100
12	BN-SO-30-XX	Reference Laboratory	64	630	300	100	860	26,000	4,800
12	BN-SO-34-XX	Reference Laboratory	68	630	290	110	830	25,000	4,700
12	BN-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	243	554	273	153	985	21,784	4,602
12	BN-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	213	574	294	155	1,011	22,303	4,799
12	BN-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	188	525	268	221	952	21,742	4,638
12	BN-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX	298	479	267	144	855	20,882	4,395
12	BN-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX	216	547	283	143	1,004	22,183	4,747
12	BN-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX	195	527	296	256	1,014	22,202	4,785
12	BN-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX	196	525	295	270	936	21,362	4,591

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
10	BN-SO-01-XX	Reference Laboratory	0.13	63	1.3 U	1.3 UJ	55	92
10	BN-SO-10-XX	Reference Laboratory	0.14	54	1.2 J	1.3 UJ	55	110
10	BN-SO-15-XX	Reference Laboratory	0.15	58	1.3 U	1.3 UJ	49	89
10	BN-SO-18-XX	Reference Laboratory	0.13	59	1.3	0.94 U	46	88
10	BN-SO-28-XX	Reference Laboratory	0.16	54	1.3 U	0.77 U	48	81
10	BN-SO-31-XX	Reference Laboratory	0.14	71	1.3 U	0.97 U	54	94
10	BN-SO-35-XX	Reference Laboratory	0.15	63	1.2 J	0.85 U	50	87
10	BN-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	46	1	0	90
10	BN-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	40	0	0	75
10	BN-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	50	20	65	149
10	BN-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	50	0	5	89
10	BN-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	40	4	62	75
10	BN-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	45	26	54	75
10	BN-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	44	12	30	96
11	BN-SO-02-XX	Reference Laboratory	0.37	54	4.3	7.6	60	470
11	BN-SO-04-XX	Reference Laboratory	0.36	48	2.9	6.5	50	400
11	BN-SO-17-XX	Reference Laboratory	0.39	47	2.7	6.3	49	390
11	BN-SO-22-XX	Reference Laboratory	0.37	40	2.8	5.4	43	330
11	BN-SO-27-XX	Reference Laboratory	0.38	46	3.7	6.1	52	380
11	BN-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	114	25	122	526
11	BN-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	108	11	47	416
11	BN-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	112	19	59	469
11	BN-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	106	14	101	426
11	BN-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	109	20	43	453
12	BN-SO-03-XX	Reference Laboratory	1.6	100	17	42	48	2,300
12	BN-SO-06-XX	Reference Laboratory	2	92	15	41	48	2,300
12	BN-SO-08-XX	Reference Laboratory	2	94	14	38	39	2,200
12	BN-SO-13-XX	Reference Laboratory	1.6	71	9.2	21	37	1,200
12	BN-SO-20-XX	Reference Laboratory	1.6	84	14	37	44	2,100
12	BN-SO-30-XX	Reference Laboratory	1.6	99	17	44	50	2,400
12	BN-SO-34-XX	Reference Laboratory	2	100	17	42	49	2,300
12	BN-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	554	54	27	3,068
12	BN-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	574	44	36	3,016
12	BN-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	525	40	13	2,833
12	BN-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	479	77	33	2,927
12	BN-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	547	42	0	3,005
12	BN-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	527	63	137	3,072
12	BN-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	525	63	22	2,854

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
13	BN-SO-07-XX	Reference Laboratory	110 J-	990 J+	520	82	1,400	23,000	6,900
13	BN-SO-16-XX	Reference Laboratory	120 J-	1,100 J+	570	86	1,500	25,000	8,100
13	BN-SO-21-XX	Reference Laboratory	150 J-	1,300 J+	660	110	1,700	30,000	8,900
13	BN-SO-25-XX	Reference Laboratory	82 J-	700 J	370 J-	64 J-	930 J-	16,000 J-	5,400 J-
13	BN-SO-33-XX	Reference Laboratory	100 J-	1,100	640	100	1,600	27,000	8,000
13	BN-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	511	937	530	203	1,662	22,144	7,949
13	BN-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	431	925	522	163	1,713	21,925	7,982
13	BN-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	450	984	541	244	1,734	22,094	7,915
13	BN-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	551	899	531	278	1,683	21,398	7,766
13	BN-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	414	958	516	157	1,774	22,555	8,118
14	BN-SO-05-XX	Reference Laboratory	160 J-	1,600	850	86	2,200	26,000	12,000
14	BN-SO-19-XX	Reference Laboratory	150 J-	1,600	860	79	2,200	26,000	12,000
14	BN-SO-26-XX	Reference Laboratory	150 J-	1,700	900	82	2,400	27,000	12,000
14	BN-SO-29-XX	Reference Laboratory	150 J-	1,600	880	86	2,300	26,000	12,000
14	BN-SO-32-XX	Reference Laboratory	160 J-	1,600	860	84	2,300	26,000	12,000
14	BN-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	838	1,612	724	289	2,863	23,960	12,710
14	BN-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	789	1,547	707	262	2,696	23,316	12,398
14	BN-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	962	1,731	693	315	2,728	25,370	12,796
14	BN-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	732	1,563	708	249	2,720	23,507	12,561
14	BN-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	819	1,559	698	322	2,728	23,635	12,529
15	CN-SO-01-XX	Reference Laboratory	13 J-	13	21	190	700	38,000	1,200
15	CN-SO-04-XX	Reference Laboratory	13 J-	11	21	200	680	37,000	1,200
15	CN-SO-08-XX	Reference Laboratory	15 J-	15	25	210	740	43,000	1,300
15	CN-SO-10-XX	Reference Laboratory	13 J-	13	22	200	760	39,000	1,200
15	CN-SO-11-XX	Reference Laboratory	17 J-	16	30	240	860	47,000	1,600
15	CN-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	52	0	3	375	800	28,081	1,181
15	CN-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	66	0	20	453	751	28,694	1,198
15	CN-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	28	0	9	436	755	28,839	1,191
15	CN-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	23	0	16	497	793	30,504	1,333
15	CN-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	16	0	8	442	682	27,696	1,177

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
13	BN-SO-07-XX	Reference Laboratory	3.4	120	26	70	41	4,000
13	BN-SO-16-XX	Reference Laboratory	3.4	130	29	77	44	4,400
13	BN-SO-21-XX	Reference Laboratory	3.6	160	35	88	52	5,100
13	BN-SO-25-XX	Reference Laboratory	3.8	88 J-	19 J-	48 J-	28 J-	2,900 J-
13	BN-SO-33-XX	Reference Laboratory	4	150	34	81	48	5,100
13	BN-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	937	103	172	5,801
13	BN-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	27	925	88	177	5,610
13	BN-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	3	0	984	98	116	5,641
13	BN-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	5	21	899	122	27	6,083
13	BN-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	958	89	102	5,790
14	BN-SO-05-XX	Reference Laboratory	5	160	48	110	39	6,700
14	BN-SO-19-XX	Reference Laboratory	5	160	48	120	39	6,700
14	BN-SO-26-XX	Reference Laboratory	5.4	160	49	120	40	7,000
14	BN-SO-29-XX	Reference Laboratory	5.4	160	48	120	41	6,800
14	BN-SO-32-XX	Reference Laboratory	5.4	160	48	120	39	6,700
14	BN-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	9	45	1,612	143	178	9,570
14	BN-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	36	54	1,547	122	0	9,290
14	BN-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	31	1,731	130	26	10,950
14	BN-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	0	40	1,563	145	120	9,228
14	BN-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	0	1	1,559	157	29	9,695
15	CN-SO-01-XX	Reference Laboratory	0.13	240	2.2	12	21	3,100
15	CN-SO-04-XX	Reference Laboratory	0.14	240	1.5	12	22	2,900
15	CN-SO-08-XX	Reference Laboratory	0.16	280	1.3 U	15	26	3,200
15	CN-SO-10-XX	Reference Laboratory	0.12	240	1.9	14	22	3,000
15	CN-SO-11-XX	Reference Laboratory	0.15	320	1.3 U	16	27	3,500
15	CN-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	6	14	12	3	12	3,438
15	CN-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	37	12	18	57	3,596
15	CN-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	11	71	9	1	148	3,538
15	CN-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	11	58	10	18	106	3,514
15	CN-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	14	18	13	0	12	3,532

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
16	AS-SO-02-XX	Reference Laboratory	2.6 UJ	18	50	180	140	48,000	1,600
16	AS-SO-06-XX	Reference Laboratory	2.4 UJ	19	52	190	130	52,000	1,600
16	AS-SO-10-XX	Reference Laboratory	1.9 J-	18	48	180	110	45,000	1,400
16	AS-SO-11-XX	Reference Laboratory	3.7 J-	22	63	230	150	52,000	2,100
16	AS-SO-13-XX	Reference Laboratory	2.4 UJ	20	57	200	150	52,000	1,700
16	AS-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	23	0	45	285	100	30,067	1,491
16	AS-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	13	0	51	236	110	29,714	1,467
16	AS-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	26	0	64	204	130	31,098	1,549
16	AS-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	43	182	115	31,107	1,566
16	AS-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	26	0	56	186	109	32,106	1,583
17	AS-SO-01-XX	Reference Laboratory	3.8 J-	26	100	420	250	100,000	3,200
17	AS-SO-04-XX	Reference Laboratory	6.4 UJ	22	110	480	260	110,000	3,300
17	AS-SO-07-XX	Reference Laboratory	3.6 J-	21	97	380	240	88,000	2,900
17	AS-SO-09-XX	Reference Laboratory	2.6 UJ	25 J-	100 J-	390 J-	250 J-	94,000 J-	3,200 J-
17	AS-SO-12-XX	Reference Laboratory	2.6 UJ	29	120	440	270	93,000	3,300
17	AS-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	46	0	98	835	216	65,292	3,052
17	AS-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	102	0	100	724	206	73,303	2,915
17	AS-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	137	0	85	731	187	71,145	2,829
17	AS-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	27	6	96	604	192	73,503	2,883
17	AS-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	101	0	92	592	191	71,234	2,816
18	SB-SO-03-XX	Reference Laboratory	1.2 UJ	9	0.51 U	150	48	38,000	18
18	SB-SO-06-XX	Reference Laboratory	1.7 J-	8	0.51 U	140	44	35,000	16
18	SB-SO-14-XX	Reference Laboratory	4.1 J-	9	0.51 U	150	46	37,000	17
18	SB-SO-38-XX	Reference Laboratory	1.3 UJ	10	0.51 U	150	57	37,000	18
18	SB-SO-41-XX	Reference Laboratory	1.3 UJ	9	0.51 U	160	58	40,000	19
18	SB-SO-47-XX	Reference Laboratory	1.3 UJ	8	0.51 U	140	44	34,000	16
18	SB-SO-51-XX	Reference Laboratory	1.3 UJ	9	0.51 U	160	50	40,000	18
18	SB-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	1	17	0	208	44	29,386	31
18	SB-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	1	18	0	151	34	29,263	35
18	SB-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX	0	8	0	120	43	29,441	47
18	SB-SO-38-MX	Oxford Instrument Analytical X-Met 3000TX	0	16	0	183	41	29,592	33
18	SB-SO-41-MX	Oxford Instrument Analytical X-Met 3000TX	0	12	0	245	43	29,977	41
18	SB-SO-47-MX	Oxford Instrument Analytical X-Met 3000TX	0	15	12	203	50	29,818	45
18	SB-SO-51-MX	Oxford Instrument Analytical X-Met 3000TX	19	14	3	281	46	29,260	42

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
16	AS-SO-02-XX	Reference Laboratory	0.76	91	2.6 U	4.5	42	3,300
16	AS-SO-06-XX	Reference Laboratory	0.74	93	2.6 U	4.8	44	3,500
16	AS-SO-10-XX	Reference Laboratory	0.78	84	1.1 U	4.4	42	3,000
16	AS-SO-11-XX	Reference Laboratory	0.72	120	1.1 U	5.6	54	3,800
16	AS-SO-13-XX	Reference Laboratory	0.79	100	3	5.2	50	3,800
16	AS-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	15	0	3,817
16	AS-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	8	19	0	3,978
16	AS-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	5	20	0	4,101
16	AS-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	8	7	0	3,751
16	AS-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	5	13	0	4,196
17	AS-SO-01-XX	Reference Laboratory	1.4	180	2.6 U	9.3	66	6,900
17	AS-SO-04-XX	Reference Laboratory	1.3	200	6.2 U	12	72	7,400
17	AS-SO-07-XX	Reference Laboratory	1.4	160	2.7	8.9	63	6,300
17	AS-SO-09-XX	Reference Laboratory	1.4	170 J-	2.6 U	9.6 J-	65 J-	6,800 J-
17	AS-SO-12-XX	Reference Laboratory	1.4	190	2.6 U	3.2	73	7,500
17	AS-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	10	54	0	13,231
17	AS-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	7	41	0	12,907
17	AS-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	8	37	0	12,719
17	AS-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	6	33	0	13,130
17	AS-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	19	0	13,016
18	SB-SO-03-XX	Reference Laboratory	62	210	1.3 U	1.3 U	67	90
18	SB-SO-06-XX	Reference Laboratory	55	200	1.3 U	1.3 U	63	82
18	SB-SO-14-XX	Reference Laboratory	55	210	1.3 U	1.3 U	66	95
18	SB-SO-38-XX	Reference Laboratory	56	210	1.3 U	1.3 U	68	91
18	SB-SO-41-XX	Reference Laboratory	54	230	1.3 U	1.3 U	71	96
18	SB-SO-47-XX	Reference Laboratory	58	200	1.3 U	1.3 U	62	82
18	SB-SO-51-XX	Reference Laboratory	54	230	1.3 U	1.3 U	74	93
18	SB-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	31	0	3	19	0	81
18	SB-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	35	13	4	1	0	72
18	SB-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX	47	15	1	6	47	76
18	SB-SO-38-MX	Oxford Instrument Analytical X-Met 3000TX	33	25	4	0	0	92
18	SB-SO-41-MX	Oxford Instrument Analytical X-Met 3000TX	41	19	5	0	0	80
18	SB-SO-47-MX	Oxford Instrument Analytical X-Met 3000TX	45	70	4	9	0	76
18	SB-SO-51-MX	Oxford Instrument Analytical X-Met 3000TX	42	46	3	18	87	75

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
19	SB-SO-05-XX	Reference Laboratory	1.6 J-	9	0.51 U	140	46	35,000	16
19	SB-SO-18-XX	Reference Laboratory	1.2 UJ	10	0.51 U	150	46	38,000	17
19	SB-SO-30-XX	Reference Laboratory	3.2 J-	7	0.51 U	94	27	22,000	10
19	SB-SO-40-XX	Reference Laboratory	2.2 J-	9	0.51 U	120	40	33,000	15
19	SB-SO-53-XX	Reference Laboratory	1.2 UJ	10	0.51 U	140	44	37,000	17
19	SB-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	5	16	0	184	33	28,684	39
19	SB-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	15	0	158	43	29,072	40
19	SB-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	21	21	0	181	30	28,982	28
19	SB-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	21	0	165	29	27,155	31
19	SB-SO-45-MX	Oxford Instrument Analytical X-Met 3000TX	0	7	0	146	46	28,205	46
20	SB-SO-08-XX	Reference Laboratory	5.4 J-	13	0.51 U	120	39	32,000	17
20	SB-SO-11-XX	Reference Laboratory	5.7 J-	13	0.51 U	140	46	36,000	20
20	SB-SO-21-XX	Reference Laboratory	4.9 J	13	0.51 U	130	43	34,000	18
20	SB-SO-39-XX	Reference Laboratory	4.7 J-	13	0.51 U	140	46	34,000	19
20	SB-SO-42-XX	Reference Laboratory	4.6 J-	13	0.51 U	140	45	35,000	18
20	SB-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	0	19	0	126	51	25,923	39
20	SB-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX	10	15	0	237	40	26,786	49
20	SB-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	25	0	99	33	26,755	31
20	SB-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX	9	21	0	137	41	26,269	43
20	SB-SO-53-MX	Oxford Instrument Analytical X-Met 3000TX	23	27	0	145	38	25,942	33
21	SB-SO-22-XX	Reference Laboratory	10 J	18	0.51 U	120	37	29,000	22
21	SB-SO-25-XX	Reference Laboratory	6.8 J+	18	0.51 U	120	37	29,000	22
21	SB-SO-27-XX	Reference Laboratory	6.7 J+	18	0.51 U	120	37	29,000	22
21	SB-SO-35-XX	Reference Laboratory	6 J+	17	0.51 U	110	35	28,000	21
21	SB-SO-44-XX	Reference Laboratory	6.8 J+	18	0.51 U	120	37	29,000	22
21	SB-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	27	0	171	44	23,136	49
21	SB-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	14	23	0	169	32	22,857	49
21	SB-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	0	29	1	243	39	22,176	51
21	SB-SO-40-MX	Oxford Instrument Analytical X-Met 3000TX	23	28	0	173	33	22,605	48
21	SB-SO-55-MX	Oxford Instrument Analytical X-Met 3000TX	14	21	0	289	36	22,320	60

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se		Ag		V	Zn	
19	SB-SO-05-XX	Reference Laboratory	540	200	1.3	U	1.3	U	61	80	
19	SB-SO-18-XX	Reference Laboratory	280	210	1.3	U	1.3	U	70	84	
19	SB-SO-30-XX	Reference Laboratory	290	120	1.3	J+	1.3	U	43	50	
19	SB-SO-40-XX	Reference Laboratory	280	180	1.3	U	1.3	U	58	74	
19	SB-SO-53-XX	Reference Laboratory	270	200	1.3	U	1.3	U	64	81	
19	SB-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	39	0	3		0		0	72	
19	SB-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	40	0	3		0		22	68	
19	SB-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	28	4	3		7		62	77	
19	SB-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX	31	0	5		16		0	62	
19	SB-SO-45-MX	Oxford Instrument Analytical X-Met 3000TX	46	34	2		0		0	57	
20	SB-SO-08-XX	Reference Laboratory	730	180	1.3	U	1.3	U	57	70	
20	SB-SO-11-XX	Reference Laboratory	810	200	1.3	U	1.3	U	66	84	
20	SB-SO-21-XX	Reference Laboratory	740	190	1.3	U	1.3	U	58	75	
20	SB-SO-39-XX	Reference Laboratory	790	200	1.3	U	1.3	U	62	77	
20	SB-SO-42-XX	Reference Laboratory	740	200	1.3	U	1.3	U	65	78	
20	SB-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	39	0	5		0		0	57	
20	SB-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX	49	12	4		3		90	56	
20	SB-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	31	9	4		16		0	60	
20	SB-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX	43	41	3		4		41	56	
20	SB-SO-53-MX	Oxford Instrument Analytical X-Met 3000TX	33	29	3		22		0	62	
21	SB-SO-22-XX	Reference Laboratory	3300	160	1.3	U	1.3	U	52	64	J-
21	SB-SO-25-XX	Reference Laboratory	3000	160	1.3	U	1.3	U	54	63	
21	SB-SO-27-XX	Reference Laboratory	3100	170	1.3	U	1.3	U	54	65	
21	SB-SO-35-XX	Reference Laboratory	3100	160	1.3	U	1.3	U	50	62	
21	SB-SO-44-XX	Reference Laboratory	3000	170	1.3	U	1.3	U	53	64	
21	SB-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	49	6	7		3		0	47	
21	SB-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	49	26	9		9		0	43	
21	SB-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	51	33	9		4		0	28	
21	SB-SO-40-MX	Oxford Instrument Analytical X-Met 3000TX	48	41	7		15		0	44	
21	SB-SO-55-MX	Oxford Instrument Analytical X-Met 3000TX	60	12	9		0		36	37	

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
22	SB-SO-23-XX	Reference Laboratory	48 J-	37	0.1 U	21	7	4,500	36
22	SB-SO-28-XX	Reference Laboratory	42 J-	36	0.1 U	21	7	4,400	36
22	SB-SO-32-XX	Reference Laboratory	46 J-	40	0.1 U	23	7.6	4,900	40
22	SB-SO-43-XX	Reference Laboratory	40 J-	35	0.1 U	20	6.7	4,200	34
22	SB-SO-48-XX	Reference Laboratory	39 J-	36	0.1 U	21	6.9	4,500	36
22	SB-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX	35	50	20	138	22	6,628	104
22	SB-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	23	52	16	100	33	6,725	107
22	SB-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	0	41	9	172	34	6,678	102
22	SB-SO-43-MX	Oxford Instrument Analytical X-Met 3000TX	36	51	16	113	32	6,898	92
22	SB-SO-48-MX	Oxford Instrument Analytical X-Met 3000TX	27	48	0	160	21	6,753	99
23	SB-SO-02-XX	Reference Laboratory	44 J-	23 J-	0.5 U	130	43	35,000	22 J-
23	SB-SO-07-XX	Reference Laboratory	45 J	22	0.5 U	120	38	35,000	23
23	SB-SO-10-XX	Reference Laboratory	62 J	26	0.5 U	140	44	41,000	27
23	SB-SO-26-XX	Reference Laboratory	61 J	30	0.5 U	160	50	46,000	31
23	SB-SO-50-XX	Reference Laboratory	57 J	27	0.5 U	140	46	42,000	28
23	SB-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	148	41	0	124	51	30,478	40
23	SB-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	130	26	0	191	43	31,011	55
23	SB-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	143	35	0	182	23	30,677	42
23	SB-SO-39-MX	Oxford Instrument Analytical X-Met 3000TX	113	28	0	136	54	31,722	59
23	SB-SO-44-MX	Oxford Instrument Analytical X-Met 3000TX	122	39	0	172	39	31,016	47
24	SB-SO-01-XX	Reference Laboratory	180 J	65	0.5 U	140	46	47,000	30
24	SB-SO-16-XX	Reference Laboratory	170 J	64	0.5 U	140	45	47,000	30
24	SB-SO-24-XX	Reference Laboratory	180 J	66	0.5 U	150	49	49,000	32
24	SB-SO-45-XX	Reference Laboratory	180 J	63	0.5 U	140	45	47,000	30
24	SB-SO-52-XX	Reference Laboratory	150 J	62	0.5 U	140	47	46,000	29
24	SB-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	487	86	0	211	43	34,469	31
24	SB-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX	387	75	0	161	42	33,723	52
24	SB-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX	446	71	0	184	55	33,301	37
24	SB-SO-37-MX	Oxford Instrument Analytical X-Met 3000TX	430	79	0	208	36	33,768	37
24	SB-SO-49-MX	Oxford Instrument Analytical X-Met 3000TX	466	81	0	193	32	32,926	34

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
22	SB-SO-23-XX	Reference Laboratory	8500	26	0.22 J	0.26 UJ	13	8
22	SB-SO-28-XX	Reference Laboratory	8800	26	0.26 U	0.26 UJ	13	8
22	SB-SO-32-XX	Reference Laboratory	8900	28	0.36	0.1 UJ	14	9
22	SB-SO-43-XX	Reference Laboratory	7600	24	0.26 U	0.26 UJ	13	8
22	SB-SO-48-XX	Reference Laboratory	8200	25	0.26 U	0.1 UJ	13	8
22	SB-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX	104	8	23	24	253	0
22	SB-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	107	8	24	24	257	0
22	SB-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	102	9	25	31	265	0
22	SB-SO-43-MX	Oxford Instrument Analytical X-Met 3000TX	92	15	20	16	196	0
22	SB-SO-48-MX	Oxford Instrument Analytical X-Met 3000TX	99	19	22	23	129	0
23	SB-SO-02-XX	Reference Laboratory	130 J+	180	1.2 U	1.2 UJ	59	88
23	SB-SO-07-XX	Reference Laboratory	270	170	1.4	1.6	53	86
23	SB-SO-10-XX	Reference Laboratory	220	200	2.8	1.8	59	100
23	SB-SO-26-XX	Reference Laboratory	260	220	3.4	1.8	68	110
23	SB-SO-50-XX	Reference Laboratory	200	200	2.9	1.8	61	100
23	SB-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	40	0	5	16	0	91
23	SB-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	55	0	2	0	0	90
23	SB-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	42	0	4	0	0	83
23	SB-SO-39-MX	Oxford Instrument Analytical X-Met 3000TX	59	5	0	0	0	95
23	SB-SO-44-MX	Oxford Instrument Analytical X-Met 3000TX	47	0	1	0	0	80
24	SB-SO-01-XX	Reference Laboratory	400	190	1.8	2.3	65	95
24	SB-SO-16-XX	Reference Laboratory	480	190	1.9	2.2	65	97
24	SB-SO-24-XX	Reference Laboratory	420	200	2.5	2.3	67	95
24	SB-SO-45-XX	Reference Laboratory	450	190	2.8	2.1 J-	63	93
24	SB-SO-52-XX	Reference Laboratory	430	190	1.8	2.2	64	90
24	SB-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	31	0	1	0	0	89
24	SB-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX	52	0	0	0	0	77
24	SB-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX	37	0	0	1	0	70
24	SB-SO-37-MX	Oxford Instrument Analytical X-Met 3000TX	37	0	2	0	0	78
24	SB-SO-49-MX	Oxford Instrument Analytical X-Met 3000TX	34	0	2	0	0	80

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
25	SB-SO-13-XX	Reference Laboratory	430 J	160	1 U	140	46	61,000	36
25	SB-SO-19-XX	Reference Laboratory	310 J	100	0.5 U	100	32	42,000	25
25	SB-SO-33-XX	Reference Laboratory	350 J	110	0.5 U	100	33	45,000	28
25	SB-SO-37-XX	Reference Laboratory	340 J	130	1 U	120	39	51,000	31
25	SB-SO-55-XX	Reference Laboratory	340 J	120	0.5 U	120	37	49,000	29
25	SB-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	852	151	0	163	38	39,533	41
25	SB-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	904	157	0	188	41	39,774	25
25	SB-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	1,260	152	0	150	33	40,721	29
25	SB-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX	819	147	4	306	31	39,789	40
25	SB-SO-50-MX	Oxford Instrument Analytical X-Met 3000TX	955	147	0	157	31	39,510	35
26	SB-SO-12-XX	Reference Laboratory	620 J	190	1 U	100	33	55,000	43
26	SB-SO-15-XX	Reference Laboratory	600 J-	170 J-	1 U	91 J-	30 J-	51,000 J-	40 J-
26	SB-SO-17-XX	Reference Laboratory	800 J+	210	1 U	110	37	61,000	48
26	SB-SO-46-XX	Reference Laboratory	740 J+	190	1 U	120	35	57,000	47
26	SB-SO-54-XX	Reference Laboratory	280	31	0.2 U	25	5.8	8,600	5 J-
26	SB-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	2,018	224	1	122	38	46,257	37
26	SB-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	1,938	228	0	186	30	47,037	36
26	SB-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	1,538	217	0	126	29	46,032	42
26	SB-SO-46-MX	Oxford Instrument Analytical X-Met 3000TX	2,097	227	2	123	41	47,338	40
26	SB-SO-54-MX	Oxford Instrument Analytical X-Met 3000TX	1,729	218	0	100	31	46,197	42
27	KP-SE-08-XX	Reference Laboratory	6.2	3	0.11 U	88	3.8	840	300 J-
27	KP-SE-11-XX	Reference Laboratory	5.6	3	0.11 U	96	4.1	940	310 J-
27	KP-SE-17-XX	Reference Laboratory	4.9	3	0.11 U	98	4.1	940	300 J-
27	KP-SE-25-XX	Reference Laboratory	6	3	0.11 U	99	4.3	960	310 J-
27	KP-SE-30-XX	Reference Laboratory	5.7	3	0.11 U	83	3.6	830	300 J-
27	KP-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	77	0	4,603	466
27	KP-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	65	0	4,616	462
27	KP-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	72	0	4,711	473
27	KP-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	70	0	4,644	466
27	KP-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	23	0	4,582	453

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
25	SB-SO-13-XX	Reference Laboratory	850	180	4.4	2.2 UJ	74	70
25	SB-SO-19-XX	Reference Laboratory	740	120	2.5	1.8	51	51
25	SB-SO-33-XX	Reference Laboratory	870	130	3	2 J	52	56
25	SB-SO-37-XX	Reference Laboratory	790	150	2.5 U	2 UJ	63	58
25	SB-SO-55-XX	Reference Laboratory	900	140	2.5	2.2 J	61	60
25	SB-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	41	0	3	0	0	60
25	SB-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	25	0	0	0	0	56
25	SB-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	29	0	0	0	0	65
25	SB-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX	40	0	1	1	0	41
25	SB-SO-50-MX	Oxford Instrument Analytical X-Met 3000TX	35	0	3	0	0	53
26	SB-SO-12-XX	Reference Laboratory	1,400	110	2.5 U	2.1 UJ	59	42
26	SB-SO-15-XX	Reference Laboratory	1,100	100 J-	3.4	1.6 UJ	52 J-	36 J-
26	SB-SO-17-XX	Reference Laboratory	1,200	120	2.8	2.3 UJ	60	42
26	SB-SO-46-XX	Reference Laboratory	670	120	2.6	2.2 UJ	57	41
26	SB-SO-54-XX	Reference Laboratory	560	20	0.5 U	0.5 UJ	11	6
26	SB-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	37	0	1	5	0	42
26	SB-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	36	0	0	0	0	64
26	SB-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	42	0	0	0	0	42
26	SB-SO-46-MX	Oxford Instrument Analytical X-Met 3000TX	40	0	0	20	0	55
26	SB-SO-54-MX	Oxford Instrument Analytical X-Met 3000TX	42	0	0	0	0	35
27	KP-SE-08-XX	Reference Laboratory	0.09 U	42	0.27 U	0.27 UJ	4	5
27	KP-SE-11-XX	Reference Laboratory	0.08 U	46	0.43	0.27 UJ	4	6
27	KP-SE-17-XX	Reference Laboratory	0.08 U	47	0.27 U	0.27 UJ	4	5
27	KP-SE-25-XX	Reference Laboratory	0.1 U	47	0.26 U	0.27 UJ	4	5
27	KP-SE-30-XX	Reference Laboratory	0.1 U	39	0.24 U	0.27 UJ	4	5
27	KP-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	28	5	1	5	0
27	KP-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	36	3	18	20	0
27	KP-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	33	5	2	0	0
27	KP-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	0	32	5	9	0	0
27	KP-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	21	3	14	21	0

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
28	KP-SE-01-XX	Reference Laboratory	3.2	2	0.1 U	34	2.2	480	310 J-
28	KP-SE-12-XX	Reference Laboratory	3.1	2	0.1 U	42	2.5	510	320 J-
28	KP-SE-14-XX	Reference Laboratory	11 J-	2	0.1 U	46 J-	2.7 J+	520 J-	680 J-
28	KP-SE-19-XX	Reference Laboratory	3	2	0.1 U	44	2.3	510	330
28	KP-SE-28-XX	Reference Laboratory	3.3	2	0.1 U	45	2.3	520	320
28	KP-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	5	0	79	0	4,385	460
28	KP-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	0	9	0	0	0	4,402	569
28	KP-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	9	0	4,183	349
28	KP-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	0	0	4,376	570
28	KP-SE-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	0	0	4,232	424
29	TL-SE-04-XX	Reference Laboratory	1.2 U	10	0.5 U	62	1,900	42,000	32
29	TL-SE-10-XX	Reference Laboratory	1.2 U	10	0.5 U	64	2,000	43,000	35
29	TL-SE-12-XX	Reference Laboratory	1.2 U	10	0.5 U	66	2,100	44,000	34
29	TL-SE-15-XX	Reference Laboratory	1.2 U	9	0.5 U	54	1,800	36,000	28
29	TL-SE-20-XX	Reference Laboratory	1.2 U	10	0.5 U	64	2,000	42,000	32
29	TL-SE-24-XX	Reference Laboratory	1.2 U	11	0.5 U	67	2,100	43,000	37
29	TL-SE-26-XX	Reference Laboratory	1.2 U	10	0.5 U	62	2,000	40,000	34
29	TL-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	6	0	44	2,101	37,922	75
29	TL-SE-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	8	0	5	2,044	37,001	74
29	TL-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	12	5	55	2,136	37,361	69
29	TL-SE-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	15	0	112	2,044	37,251	67
29	TL-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	12	2	96	2,058	37,366	76
29	TL-SE-24-MX	Oxford Instrument Analytical X-Met 3000TX	0	20	0	143	1,988	37,417	55
29	TL-SE-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	10	5	87	2,146	37,687	73
30	TL-SE-03-XX	Reference Laboratory	2.5 U	9	1 U	91	1,600	63,000	12
30	TL-SE-19-XX	Reference Laboratory	2.5 U	10	1 U	96	1,700	66,000	13
30	TL-SE-23-XX	Reference Laboratory	2.5 U	9	1 U	92	1,600	64,000	12
30	TL-SE-25-XX	Reference Laboratory	2.5 U	10	1 U	91	1,600	62,000	11
30	TL-SE-31-XX	Reference Laboratory	2.5 U	10	1 U	110	1,800	74,000	13
30	TL-SE-03-MX	Oxford Instrument Analytical X-Met 3000TX	0	5	6	34	1,574	56,405	53
30	TL-SE-19-MX	Oxford Instrument Analytical X-Met 3000TX	0	1	0	200	1,585	57,862	63
30	TL-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	13	56	1,672	59,115	66
30	TL-SE-25-MX	Oxford Instrument Analytical X-Met 3000TX	0	7	16	65	1,586	57,187	46
30	TL-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	9	9	47	1,650	58,877	47

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
28	KP-SE-01-XX	Reference Laboratory	0.05 U	16	0.26 U	0.26 UJ	2 J	6
28	KP-SE-12-XX	Reference Laboratory	0.06 U	20	0.26 U	0.26 UJ	2 J	8
28	KP-SE-14-XX	Reference Laboratory	0.07 U	23 J-	0.26 U	0.26 UJ	3 J	7
28	KP-SE-19-XX	Reference Laboratory	0.04 U	22	0.26 U	0.26 U	2 J	7
28	KP-SE-28-XX	Reference Laboratory	0.06 U	22	0.26 U	0.26 U	2 J	6
28	KP-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	15	0	0
28	KP-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	15	0	0
28	KP-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	12	36	0
28	KP-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	3	0	0	0
28	KP-SE-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	2	0	0
29	TL-SE-04-XX	Reference Laboratory	0.26 J-	71	1.2 U	1.3	95	160
29	TL-SE-10-XX	Reference Laboratory	0.19 J-	72	1.2 U	1.2 U	95	160
29	TL-SE-12-XX	Reference Laboratory	0.22 J-	75	1.2 U	1.2 U	100	170
29	TL-SE-15-XX	Reference Laboratory	0.28 J-	63	1.2 U	1 U	84	140
29	TL-SE-20-XX	Reference Laboratory	0.26 J-	74	1.2 U	1.2 U	100	160
29	TL-SE-24-XX	Reference Laboratory	0.26 J-	77	1.2 U	1.3 U	100	170
29	TL-SE-26-XX	Reference Laboratory	0.24 J-	70	1.2 U	1.2 U	96	160
29	TL-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	0	0	222
29	TL-SE-10-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	9	0	236
29	TL-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	7	0	212
29	TL-SE-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	0	0	229
29	TL-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	3	14	0	230
29	TL-SE-24-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	3	0	0	234
29	TL-SE-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	12	0	222
30	TL-SE-03-XX	Reference Laboratory	0.32 J-	110	2.5 U	0.94 U	140	200
30	TL-SE-19-XX	Reference Laboratory	0.32 J-	120	2.5 U	1.1 U	150	210
30	TL-SE-23-XX	Reference Laboratory	0.41 J-	110	2.5 U	1.3 U	150	200
30	TL-SE-25-XX	Reference Laboratory	0.44 J-	110	2.5 U	0.94 U	150	200
30	TL-SE-31-XX	Reference Laboratory	0.57 J-	130	2.5 U	1.2 U	170	230
30	TL-SE-03-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	10	0	266
30	TL-SE-19-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	3	0	0	276
30	TL-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	7	0	254
30	TL-SE-25-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	22	0	282
30	TL-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	12	0	257

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
31	TL-SE-01-XX	Reference Laboratory	1.2 UJ	9	0.5 U	110	1,400	19,000	48 J-
31	TL-SE-11-XX	Reference Laboratory	1.2 UJ	15	0.5 U	140	1,600	28,000	54 J-
31	TL-SE-14-XX	Reference Laboratory	1.2 UJ	10	0.27 J	110	1,500	18,000	50 J-
31	TL-SE-18-XX	Reference Laboratory	1.2 UJ	10	0.5 U	150	1,300	24,000	46 J-
31	TL-SE-22-XX	Reference Laboratory	1.2 UJ	11	0.5 U	150	1,700	26,000	54 J-
31	TL-SE-27-XX	Reference Laboratory	1.2 UJ	10	0.28 J	130	1,500	19,000	51 J-
31	TL-SE-29-XX	Reference Laboratory	1.2 UJ	11	0.22 J	140	1,600	23,000	51 J-
31	TL-SE-05-MX	Oxford Instrument Analytical X-Met 3000TX	72	13	0	305	1,447	36,889	80
31	TL-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	3	21	0	277	1,485	39,468	83
31	TL-SE-13-MX	Oxford Instrument Analytical X-Met 3000TX	46	33	0	444	1,568	39,499	85
31	TL-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	60	29	0	193	1,414	38,817	60
31	TL-SE-21-MX	Oxford Instrument Analytical X-Met 3000TX	49	14	2	377	1,528	35,487	74
31	TL-SE-28-MX	Oxford Instrument Analytical X-Met 3000TX	93	18	3	233	1,495	40,405	73
31	TL-SE-30-MX	Oxford Instrument Analytical X-Met 3000TX	68	11	0	290	1,486	37,586	86
32	LV-SE-02-XX	Reference Laboratory	1.3 UJ	28	0.51 U	72	33	23,000	20 J-
32	LV-SE-10-XX	Reference Laboratory	1.3 UJ	34	0.51 U	84	42	28,000	25 J-
32	LV-SE-22-XX	Reference Laboratory	1.3 UJ	30	0.51 U	69	33	23,000	22 J-
32	LV-SE-25-XX	Reference Laboratory	1.3 UJ	31	0.51 U	74	36	25,000	23 J-
32	LV-SE-31-XX	Reference Laboratory	1.3 UJ	32	0.51 U	78	36	25,000	49 J-
32	LV-SE-35-XX	Reference Laboratory	1.3 UJ	31 J-	0.51 U	74 J-	35	24,000 J-	22 J-
32	LV-SE-50-XX	Reference Laboratory	2.5 U	29	1 U	74	34	24,000	24 J-
32	LV-SE-02-MX	Oxford Instrument Analytical X-Met 3000TX	0	30	10	183	53	23,036	78
32	LV-SE-10-MX	Oxford Instrument Analytical X-Met 3000TX	13	35	0	125	39	22,055	53
32	LV-SE-22-MX	Oxford Instrument Analytical X-Met 3000TX	0	38	0	16	43	22,993	62
32	LV-SE-25-MX	Oxford Instrument Analytical X-Met 3000TX	0	36	0	55	46	22,734	74
32	LV-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	38	0	43	52	23,006	71
32	LV-SE-35-MX	Oxford Instrument Analytical X-Met 3000TX	18	38	4	106	40	21,722	55
32	LV-SE-50-MX	Oxford Instrument Analytical X-Met 3000TX	30	41	0	114	49	22,115	57
33	LV-SE-12-XX	Reference Laboratory	2.6 U	190	1 U	55	34	72,000	19 J-
33	LV-SE-26-XX	Reference Laboratory	2.6 U	220	1 U	64	39	83,000	25 J-
33	LV-SE-33-XX	Reference Laboratory	2.6 U	170	1 U	52	31	66,000	21 J-
33	LV-SE-39-XX	Reference Laboratory	2.6 U	190	1 U	58	35	74,000	22 J-
33	LV-SE-42-XX	Reference Laboratory	2.7 U	170	1.1 U	50	30	65,000	22 J-
33	LV-SE-01-MX	Oxford Instrument Analytical X-Met 3000TX	28	205	0	89	48	61,940	43
33	LV-SE-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	190	0	62	45	61,266	45
33	LV-SE-17-MX	Oxford Instrument Analytical X-Met 3000TX	31	197	4	41	34	62,835	46
33	LV-SE-37-MX	Oxford Instrument Analytical X-Met 3000TX	43	192	1	28	31	61,369	47
33	LV-SE-49-MX	Oxford Instrument Analytical X-Met 3000TX	44	191	0	65	39	64,126	57

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
31	TL-SE-01-XX	Reference Laboratory	0.07 U	180	1.2 U	5.7 J-	75	130
31	TL-SE-11-XX	Reference Laboratory	0.02 U	210	1.2 U	5.5 J-	85	140
31	TL-SE-14-XX	Reference Laboratory	0.08 U	180	1.2 U	5.7 J-	73	140
31	TL-SE-18-XX	Reference Laboratory	0.03 U	190	1.2 U	6.3 J-	70	120
31	TL-SE-22-XX	Reference Laboratory	0.08 U	210	1.2 U	6.5 J-	80	150
31	TL-SE-27-XX	Reference Laboratory	0.02 U	200	1.2 U	7.8 J-	67	140
31	TL-SE-29-XX	Reference Laboratory	0.08 U	200	1.2 U	5.9 J-	80	140
31	TL-SE-05-MX	Oxford Instrument Analytical X-Met 3000TX	40	0	2	12	91	252
31	TL-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	57	0	7	0	0	247
31	TL-SE-13-MX	Oxford Instrument Analytical X-Met 3000TX	85	16	4	10	0	281
31	TL-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	46	0	7	21	0	239
31	TL-SE-21-MX	Oxford Instrument Analytical X-Met 3000TX	44	0	8	16	0	230
31	TL-SE-28-MX	Oxford Instrument Analytical X-Met 3000TX	61	0	4	20	0	261
31	TL-SE-30-MX	Oxford Instrument Analytical X-Met 3000TX	47	0	9	30	0	254
32	LV-SE-02-XX	Reference Laboratory	0.02 U	160	3.8	1.3 UJ	53	65
32	LV-SE-10-XX	Reference Laboratory	0.02 U	200	4.7	1.3 UJ	66	77
32	LV-SE-22-XX	Reference Laboratory	1.1	170	5.2	1.3 UJ	51	66
32	LV-SE-25-XX	Reference Laboratory	1	170	5.1	1.3 UJ	56	70
32	LV-SE-31-XX	Reference Laboratory	1	180	5.1	1.3 UJ	58	70
32	LV-SE-35-XX	Reference Laboratory	1.4	170 J-	5	1.3 UJ	55 J-	67 J-
32	LV-SE-50-XX	Reference Laboratory	1.2	170	3.3	2.5 U	57	65
32	LV-SE-02-MX	Oxford Instrument Analytical X-Met 3000TX	56	56	12	9	0	83
32	LV-SE-10-MX	Oxford Instrument Analytical X-Met 3000TX	44	44	8	24	136	84
32	LV-SE-22-MX	Oxford Instrument Analytical X-Met 3000TX	37	37	9	4	123	93
32	LV-SE-25-MX	Oxford Instrument Analytical X-Met 3000TX	61	61	12	0	110	85
32	LV-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	45	45	11	0	164	72
32	LV-SE-35-MX	Oxford Instrument Analytical X-Met 3000TX	49	49	10	32	83	78
32	LV-SE-50-MX	Oxford Instrument Analytical X-Met 3000TX	49	49	9	0	201	68
33	LV-SE-12-XX	Reference Laboratory	5.6	71	3	2.6 U	72	66
33	LV-SE-26-XX	Reference Laboratory	6	83	6.1	2.6 U	86	75
33	LV-SE-33-XX	Reference Laboratory	6.8	66	2.8	2.6 U	67	59
33	LV-SE-39-XX	Reference Laboratory	8	74	5.1	2.6 U	74	66
33	LV-SE-42-XX	Reference Laboratory	4.3	67	3.4	2.7 U	64	57
33	LV-SE-01-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	6	0	0	78
33	LV-SE-06-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	0	0	82
33	LV-SE-17-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	16	0	94
33	LV-SE-37-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	12	0	102
33	LV-SE-49-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	2	12	0	100

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
34	LV-SE-09-XX	Reference Laboratory	6.7 U	450	2.7 U	48	34	150,000	14 J-
34	LV-SE-19-XX	Reference Laboratory	6.7 U	500	2.7 U	55	37	160,000	17 J-
34	LV-SE-27-XX	Reference Laboratory	6.7 U	530	2.7 U	56	39	180,000	16 J-
34	LV-SE-36-XX	Reference Laboratory	6.7 U	550	2.7 U	60	40	180,000	21 J-
34	LV-SE-38-XX	Reference Laboratory	6.7 U	480	2.7 U	52	36	160,000	15 J-
34	LV-SE-03-MX	Oxford Instrument Analytical X-Met 3000TX	46	431	3	0	33	147,883	63
34	LV-SE-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	428	8	0	27	149,431	60
34	LV-SE-24-MX	Oxford Instrument Analytical X-Met 3000TX	88	428	7	0	27	146,845	57
34	LV-SE-32-MX	Oxford Instrument Analytical X-Met 3000TX	87	407	22	0	9	143,583	66
34	LV-SE-42-MX	Oxford Instrument Analytical X-Met 3000TX	65	425	9	0	19	144,894	50
35	LV-SE-07-XX	Reference Laboratory	6.7 UJ	780	2.7 U	57	48	200,000	11
35	LV-SE-18-XX	Reference Laboratory	6.7 UJ	800	2.7 U	61	49	210,000	11
35	LV-SE-23-XX	Reference Laboratory	6.6 UJ	660	2.6 U	53	40	170,000	8
35	LV-SE-45-XX	Reference Laboratory	6.7 UJ	650	2.7 U	50	40	170,000	8
35	LV-SE-48-XX	Reference Laboratory	6.6 UJ	680	2.6 U	52	42	180,000	9
35	LV-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	96	639	0	0	24	202,788	104
35	LV-SE-18-MX	Oxford Instrument Analytical X-Met 3000TX	68	651	0	0	28	201,208	63
35	LV-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	156	672	20	0	13	203,000	82
35	LV-SE-45-MX	Oxford Instrument Analytical X-Met 3000TX	126	673	19	0	34	203,687	70
35	LV-SE-48-MX	Oxford Instrument Analytical X-Met 3000TX	100	655	0	0	19	199,835	71
36	LV-SE-01-XX	Reference Laboratory	1.5 UJ	6	0.76	4	18	1,100	17
36	LV-SE-14-XX	Reference Laboratory	1.5 UJ	5	0.74	4	16	980	14
36	LV-SE-21-XX	Reference Laboratory	1.5 UJ	7	0.84	4	19	970	18
36	LV-SE-24-XX	Reference Laboratory	1.5 UJ	5	0.68	4	15	840	14
36	LV-SE-32-XX	Reference Laboratory	1.4 UJ	6	0.87	4	16	860	14
36	LV-SE-05-MX	Oxford Instrument Analytical X-Met 3000TX	0	11	17	0	0	4,299	24
36	LV-SE-19-MX	Oxford Instrument Analytical X-Met 3000TX	0	14	9	0	0	4,281	15
36	LV-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	0	9	0	0	0	4,269	25
36	LV-SE-39-MX	Oxford Instrument Analytical X-Met 3000TX	0	6	13	0	0	4,254	22
36	LV-SE-51-MX	Oxford Instrument Analytical X-Met 3000TX	0	9	3	0	0	4,331	24

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
34	LV-SE-09-XX	Reference Laboratory	6	55	6.7 U	6.7 U	100	51 J
34	LV-SE-19-XX	Reference Laboratory	7.2	65	5.9 J	6.7 U	110	55 J
34	LV-SE-27-XX	Reference Laboratory	11	64	6.7 U	6.7 U	120	58 J
34	LV-SE-36-XX	Reference Laboratory	8.5	70	11	6.7 U	120	60 J
34	LV-SE-38-XX	Reference Laboratory	7.9	75	6.7 U	6.7 U	100	54 J
34	LV-SE-03-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	3	0	146
34	LV-SE-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	4	2	0	141
34	LV-SE-24-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	24	0	127
34	LV-SE-32-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	4	0	115
34	LV-SE-42-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	9	0	132
35	LV-SE-07-XX	Reference Laboratory	5.5	58	10	6.7 U	130	24 J
35	LV-SE-18-XX	Reference Laboratory	5.4	60	12	6.7 U	140	52 J
35	LV-SE-23-XX	Reference Laboratory	5	50 J	9.6	6.6 U	120	18 J
35	LV-SE-45-XX	Reference Laboratory	5.6	50 J	8.2	6.7 U	120	19 J
35	LV-SE-48-XX	Reference Laboratory	7.3	50 J	7.6	6.6 U	120	30 J
35	LV-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	0	0	134
35	LV-SE-18-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	25	0	135
35	LV-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	39	0	142
35	LV-SE-45-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	16	0	130
35	LV-SE-48-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	6	0	122
36	LV-SE-01-XX	Reference Laboratory	0.1 U	49	1.5 U	1.5 U	2 J	14 J
36	LV-SE-14-XX	Reference Laboratory	0.06 U	46	1.5 U	1.5 U	1 J	12 J
36	LV-SE-21-XX	Reference Laboratory	0.05 U	49	1.5 U	1.5 U	2 J	14 J
36	LV-SE-24-XX	Reference Laboratory	0.05 U	44	1.5 U	1.5 U	1 J	12 J
36	LV-SE-32-XX	Reference Laboratory	0.05 U	47	1.4 U	1.4 U	1 J	19
36	LV-SE-05-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	25	0	0
36	LV-SE-19-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	18	0	0
36	LV-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	8	0	0
36	LV-SE-39-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	11	0	0
36	LV-SE-51-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	15	0	0

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
37	LV-SE-08-XX	Reference Laboratory	1.3 UJ	30	0.52 U	54	23	23,000	55
37	LV-SE-16-XX	Reference Laboratory	1.3 UJ	29	0.52 U	53	22	22,000	53
37	LV-SE-28-XX	Reference Laboratory	1.3 UJ	31	0.52 U	59	25	25,000	59
37	LV-SE-30-XX	Reference Laboratory	1.3 UJ	30	0.52 U	58	25	24,000	58
37	LV-SE-47-XX	Reference Laboratory	1.3 UJ	31	0.52 U	56	23	23,000	57
37	LV-SE-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	41	7	37	37	23,676	139
37	LV-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	0	40	0	89	33	22,810	125
37	LV-SE-28-MX	Oxford Instrument Analytical X-Met 3000TX	0	39	0	65	32	23,322	128
37	LV-SE-30-MX	Oxford Instrument Analytical X-Met 3000TX	0	42	1	167	42	23,489	118
37	LV-SE-47-MX	Oxford Instrument Analytical X-Met 3000TX	0	41	0	152	41	23,625	107
38	LV-SE-11-XX	Reference Laboratory	1.4 UJ	150	6.6	120	270	42,000	7
38	LV-SE-29-XX	Reference Laboratory	1.4 UJ	150	6.3	120	260	42,000	7 J+
38	LV-SE-44-XX	Reference Laboratory	1.4 U	140	6.1	120	250	40,000	8
38	LV-SE-46-XX	Reference Laboratory	0.88 U	110	5	92	200	32,000	6
38	LV-SE-52-XX	Reference Laboratory	1.4 U	160	6.8	130	280	44,000	8
38	LV-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	156	10	186	309	35,709	48
38	LV-SE-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	160	0	52	272	34,790	27
38	LV-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	148	0	110	299	34,532	41
38	LV-SE-34-MX	Oxford Instrument Analytical X-Met 3000TX	0	170	8	8	296	34,985	34
38	LV-SE-43-MX	Oxford Instrument Analytical X-Met 3000TX	9	169	0	148	271	34,858	17
39	RF-SE-07-XX	Reference Laboratory	1.3 U	12	0.5 U	92	81	17,000	24
39	RF-SE-12-XX	Reference Laboratory	1.2 U	14	0.5 U	100	110	20,000	25
39	RF-SE-23-XX	Reference Laboratory	0.25 U	0 U	0.1 U	0 U	0.2 U	4 J	0 U
39	RF-SE-36-XX	Reference Laboratory	1.2 U	12	0.5 U	91	82	17,000	22
39	RF-SE-42-XX	Reference Laboratory	1.3 UJ	14	0.56	110	95	19,000	28
39	RF-SE-45-XX	Reference Laboratory	1.3 UJ	15	0.52 U	110	100	21,000	33
39	RF-SE-53-XX	Reference Laboratory	1.3 UJ	14	0.57 U	110	95	19,000	28
39	RF-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	22	0	126	98	18,491	58
39	RF-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	14	0	182	126	21,245	74
39	RF-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	0	26	0	93	125	19,811	62
39	RF-SE-36-MX	Oxford Instrument Analytical X-Met 3000TX	5	22	0	114	111	19,369	64
39	RF-SE-42-MX	Oxford Instrument Analytical X-Met 3000TX	0	26	0	163	145	21,255	70
39	RF-SE-45-MX	Oxford Instrument Analytical X-Met 3000TX	0	16	0	157	138	20,825	90
39	RF-SE-53-MX	Oxford Instrument Analytical X-Met 3000TX	0	22	0	117	127	19,603	70

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
37	LV-SE-08-XX	Reference Laboratory	5.2	110	4.8	1.3 U	44	61
37	LV-SE-16-XX	Reference Laboratory	5.4	110	5	1.3 U	42	59
37	LV-SE-28-XX	Reference Laboratory	5.4	120	5.8	1.3 U	48	65
37	LV-SE-30-XX	Reference Laboratory	6.3	120	5.6	1.3 U	48	66
37	LV-SE-47-XX	Reference Laboratory	4.9	120	4.2	1.3 U	45	65
37	LV-SE-08-MX	Oxford Instrument Analytical X-Met 3000TX	65	65	14	7	106	60
37	LV-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	40	40	10	0	197	82
37	LV-SE-28-MX	Oxford Instrument Analytical X-Met 3000TX	58	58	13	0	191	72
37	LV-SE-30-MX	Oxford Instrument Analytical X-Met 3000TX	52	52	11	13	156	72
37	LV-SE-47-MX	Oxford Instrument Analytical X-Met 3000TX	49	49	11	6	16	66
38	LV-SE-11-XX	Reference Laboratory	2.8	870	1.3 U	1.4 U	35	200
38	LV-SE-29-XX	Reference Laboratory	1.5 J-	860	1.2 U	1.4 U	35	200
38	LV-SE-44-XX	Reference Laboratory	1.5	830	1.4 U	1.4 U	34	190
38	LV-SE-46-XX	Reference Laboratory	1.4	660	0.88 U	0.88 U	27	150
38	LV-SE-52-XX	Reference Laboratory	21	910	1.4 U	1.4 U	38	210
38	LV-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	13	0	178
38	LV-SE-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	0	0	200
38	LV-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1	0	0	179
38	LV-SE-34-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	0	0	186
38	LV-SE-43-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	5	0	205
39	RF-SE-07-XX	Reference Laboratory	0.09 U	180	1.3 U	1.3 U	34	130
39	RF-SE-12-XX	Reference Laboratory	0.1 U	210	1.2 U	1.2 U	38	140
39	RF-SE-23-XX	Reference Laboratory	2.4	2 U	0.25 U	0.37	3 U	1 U
39	RF-SE-36-XX	Reference Laboratory	0.08 U	180	1 U	1.2 U	34	120
39	RF-SE-42-XX	Reference Laboratory	0.08 U	210	1.3 U	1.3 U	40	140
39	RF-SE-45-XX	Reference Laboratory	0.08 U	220	1.3 U	1.3 U	43	150
39	RF-SE-53-XX	Reference Laboratory	0.08 U	210	1.3 U	1.3 U	40	140
39	RF-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX	86	83	9	0	0	144
39	RF-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	105	118	10	0	0	162
39	RF-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX	97	80	8	0	71	168
39	RF-SE-36-MX	Oxford Instrument Analytical X-Met 3000TX	69	51	8	2	0	164
39	RF-SE-42-MX	Oxford Instrument Analytical X-Met 3000TX	81	90	9	0	17	180
39	RF-SE-45-MX	Oxford Instrument Analytical X-Met 3000TX	105	82	10	0	0	186
39	RF-SE-53-MX	Oxford Instrument Analytical X-Met 3000TX	80	74	11	0	75	171

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
40	RF-SE-03-XX	Reference Laboratory	1.2 UJ	27	1.3	93	200	17,000	88
40	RF-SE-28-XX	Reference Laboratory	1.2 UJ	31	1.5	100	220	18,000	99
40	RF-SE-38-XX	Reference Laboratory	1.2 UJ	27	1.2	90	190	16,000	83
40	RF-SE-49-XX	Reference Laboratory	1.2 UJ	31	1.5	100	220	18,000	97
40	RF-SE-55-XX	Reference Laboratory	1.2 UJ	24	1.1	91	180	15,000	75
40	RF-SE-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	28	0	145	266	18,405	146
40	RF-SE-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	39	0	32	274	18,550	127
40	RF-SE-32-MX	Oxford Instrument Analytical X-Met 3000TX	1	42	0	184	351	21,528	167
40	RF-SE-44-MX	Oxford Instrument Analytical X-Met 3000TX	0	43	0	136	309	19,374	148
40	RF-SE-51-MX	Oxford Instrument Analytical X-Met 3000TX	20	43	0	107	270	18,586	131
41	RF-SE-06-XX	Reference Laboratory	1.3 UJ	70	3.6	90	490	20,000	230
41	RF-SE-13-XX	Reference Laboratory	1.3 UJ	76	3.7	92	530	21,000	230
41	RF-SE-27-XX	Reference Laboratory	1.3 UJ	64	3.1	78	440	18,000	200
41	RF-SE-31-XX	Reference Laboratory	1.3 UJ	39	1.8	63	250	12,000	120
41	RF-SE-58-XX	Reference Laboratory	1.3 UJ	71	3.6	89	500	21,000	230
41	RF-SE-02-MX	Oxford Instrument Analytical X-Met 3000TX	0	87	0	202	605	19,874	267
41	RF-SE-18-MX	Oxford Instrument Analytical X-Met 3000TX	4	80	2	161	631	20,702	285
41	RF-SE-22-MX	Oxford Instrument Analytical X-Met 3000TX	19	79	0	34	598	20,043	281
41	RF-SE-38-MX	Oxford Instrument Analytical X-Met 3000TX	30	78	0	132	662	20,576	313
41	RF-SE-48-MX	Oxford Instrument Analytical X-Met 3000TX	1	83	0	184	672	20,721	304
42	RF-SE-02-XX	Reference Laboratory	1.3 UJ	110	5.4	93	740	24,000	330
42	RF-SE-22-XX	Reference Laboratory	1.3 UJ	99	4.7	84	670	22,000	300
42	RF-SE-25-XX	Reference Laboratory	1.3 UJ	88	4	78	580	19,000	270
42	RF-SE-30-XX	Reference Laboratory	1.3 UJ	89	4.3	78	610	21,000	290
42	RF-SE-57-XX	Reference Laboratory	1.3 UJ	89	4.5	79	610	21,000	300
42	RF-SE-09-MX	Oxford Instrument Analytical X-Met 3000TX	0	103	25	116	832	21,242	363
42	RF-SE-17-MX	Oxford Instrument Analytical X-Met 3000TX	0	117	0	173	978	22,768	404
42	RF-SE-28-MX	Oxford Instrument Analytical X-Met 3000TX	0	99	0	258	785	20,257	344
42	RF-SE-40-MX	Oxford Instrument Analytical X-Met 3000TX	10	105	0	169	817	21,438	381
42	RF-SE-50-MX	Oxford Instrument Analytical X-Met 3000TX	1	122	0	132	880	21,891	368

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
40	RF-SE-03-XX	Reference Laboratory	0.48	150	1.2 U	1.2 U	40	300
40	RF-SE-28-XX	Reference Laboratory	0.57	160	1.2 U	1.2 U	44	320
40	RF-SE-38-XX	Reference Laboratory	0.41	140	1.2 U	1.2 U	39	300
40	RF-SE-49-XX	Reference Laboratory	0.43	170	1.2 U	1.2 U	43	330
40	RF-SE-55-XX	Reference Laboratory	0.42	140	1.2 U	1.2 U	35	280
40	RF-SE-08-MX	Oxford Instrument Analytical X-Met 3000TX	57	53	8	0	0	325
40	RF-SE-15-MX	Oxford Instrument Analytical X-Met 3000TX	41	58	9	2	37	346
40	RF-SE-32-MX	Oxford Instrument Analytical X-Met 3000TX	72	59	9	3	101	425
40	RF-SE-44-MX	Oxford Instrument Analytical X-Met 3000TX	54	51	7	4	34	397
40	RF-SE-51-MX	Oxford Instrument Analytical X-Met 3000TX	40	36	10	21	0	358
41	RF-SE-06-XX	Reference Laboratory	1.1	150	1.3 U	1.3 U	44	740
41	RF-SE-13-XX	Reference Laboratory	1.2	160	1.3 U	1.3	45	790
41	RF-SE-27-XX	Reference Laboratory	1.2	130	1.3 U	1.3 U	39	670
41	RF-SE-31-XX	Reference Laboratory	1.1	86	1.3 U	1.3 U	28	420
41	RF-SE-58-XX	Reference Laboratory	1.2	150	1.3 U	1.3 U	46	770
41	RF-SE-02-MX	Oxford Instrument Analytical X-Met 3000TX	37	23	4	8	0	926
41	RF-SE-18-MX	Oxford Instrument Analytical X-Met 3000TX	72	51	6	0	0	875
41	RF-SE-22-MX	Oxford Instrument Analytical X-Met 3000TX	47	48	5	18	33	923
41	RF-SE-38-MX	Oxford Instrument Analytical X-Met 3000TX	45	10	8	11	19	892
41	RF-SE-48-MX	Oxford Instrument Analytical X-Met 3000TX	51	43	5	12	53	872
42	RF-SE-02-XX	Reference Laboratory	1.6	180	1.3 U	2.7	50	1,100
42	RF-SE-22-XX	Reference Laboratory	1.7	160	1.3 U	2.3	44	990
42	RF-SE-25-XX	Reference Laboratory	1.5	140	1.5	1.7	40	890
42	RF-SE-30-XX	Reference Laboratory	1.5	150	1.3 U	1.9	44	960
42	RF-SE-57-XX	Reference Laboratory	1.5	150	2	2.2	44	1,000
42	RF-SE-09-MX	Oxford Instrument Analytical X-Met 3000TX	78	68	10	3	29	1,091
42	RF-SE-17-MX	Oxford Instrument Analytical X-Met 3000TX	75	67	12	0	0	1,068
42	RF-SE-28-MX	Oxford Instrument Analytical X-Met 3000TX	51	51	6	1	59	992
42	RF-SE-40-MX	Oxford Instrument Analytical X-Met 3000TX	56	37	6	17	16	1,280
42	RF-SE-50-MX	Oxford Instrument Analytical X-Met 3000TX	38	23	6	0	114	1,223

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
43	RF-SE-15-XX	Reference Laboratory	1.3 UJ	120	6.2	72	820	23,000	390
43	RF-SE-24-XX	Reference Laboratory	1.3 UJ	130 J+	6.5 J+	74 J+	860 J+	24,000 J+	410 J+
43	RF-SE-32-XX	Reference Laboratory	1.3 UJ	120	5.1	64	770	20,000	330
43	RF-SE-43-XX	Reference Laboratory	1.3 UJ	130	5.7	68	840	22,000	350
43	RF-SE-59-XX	Reference Laboratory	1.3 UJ	140	5.9	73	890	23,000	380
43	RF-SE-03-MX	Oxford Instrument Analytical X-Met 3000TX	0	111	7	67	967	19,970	459
43	RF-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	11	150	0	31	1,089	21,571	455
43	RF-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	10	154	0	140	1,174	22,611	494
43	RF-SE-35-MX	Oxford Instrument Analytical X-Met 3000TX	8	139	0	73	1,120	21,674	478
43	RF-SE-54-MX	Oxford Instrument Analytical X-Met 3000TX	0	148	0	95	1,047	21,776	481
44	RF-SE-05-XX	Reference Laboratory	4.1 J+	160	9.1	69	1,000	26,000	450
44	RF-SE-26-XX	Reference Laboratory	2.2 J+	140	8.4	64	990	23,000	440
44	RF-SE-39-XX	Reference Laboratory	2.9 J+	160	9.3	73	1,100	26,000	490
44	RF-SE-44-XX	Reference Laboratory	2.7 J+	140	8.2	64	970	24,000	420
44	RF-SE-56-XX	Reference Laboratory	3.5 J+	180	9.6	75	1200	27,000	490
44	RF-SE-01-MX	Oxford Instrument Analytical X-Met 3000TX	25	178	15	132	1,408	23,616	523
44	RF-SE-11-MX	Oxford Instrument Analytical X-Met 3000TX	0	166	0	59	1,295	23,253	497
44	RF-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	0	183	7	97	1,419	23,937	518
44	RF-SE-33-MX	Oxford Instrument Analytical X-Met 3000TX	2	159	0	42	1,405	23,221	533
44	RF-SE-59-MX	Oxford Instrument Analytical X-Met 3000TX	0	196	5	269	1,533	24,762	508
45	RF-SE-04-XX	Reference Laboratory	3.2 J+	230	12	42	1,500	27,000	730
45	RF-SE-14-XX	Reference Laboratory	4.4 J+	260	12	47	1,700	30,000	800
45	RF-SE-19-XX	Reference Laboratory	3.7 J+	250	13	48	1,700	30,000	800
45	RF-SE-34-XX	Reference Laboratory	2.9 J+	210	10	39	1,400	24,000	660
45	RF-SE-52-XX	Reference Laboratory	3.4 J+	220	11	42	1,500	26,000	720
45	RF-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	12	219	0	34	1,749	23,188	776
45	RF-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	19	220	0	81	1,838	23,503	746
45	RF-SE-19-MX	Oxford Instrument Analytical X-Met 3000TX	13	250	18	23	1,830	23,160	729
45	RF-SE-34-MX	Oxford Instrument Analytical X-Met 3000TX	0	258	14	56	2,077	25,592	873
45	RF-SE-52-MX	Oxford Instrument Analytical X-Met 3000TX	2	226	1	0	1,746	23,189	778

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
43	RF-SE-15-XX	Reference Laboratory	2.6	160	1.4	3.6	45	1,300
43	RF-SE-24-XX	Reference Laboratory	2.3	170 J+	1.3 U	3.8 J+	46 J+	1,400 J-
43	RF-SE-32-XX	Reference Laboratory	2.8	140	1.3 U	4.2	36	1,100
43	RF-SE-43-XX	Reference Laboratory	2.7	150	1.3 U	4	40	1,200
43	RF-SE-59-XX	Reference Laboratory	0.09 U	160	1.3 U	4.5	42	1,300
43	RF-SE-03-MX	Oxford Instrument Analytical X-Met 3000TX	55	9	8	14	0	1,335
43	RF-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX	57	69	8	6	53	1,527
43	RF-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	61	54	12	6	0	1,679
43	RF-SE-35-MX	Oxford Instrument Analytical X-Met 3000TX	54	26	7	0	0	1,451
43	RF-SE-54-MX	Oxford Instrument Analytical X-Met 3000TX	46	39	9	0	3	1,578
44	RF-SE-05-XX	Reference Laboratory	2.6	150	3.1	7.4 J-	48	1,800
44	RF-SE-26-XX	Reference Laboratory	2.5	140	2.8	7.2 J-	42	1,700
44	RF-SE-39-XX	Reference Laboratory	2.2	150	2.6	8.2 J-	49	1,900
44	RF-SE-44-XX	Reference Laboratory	2.3	140	2.4	7.2 J-	44	1,600
44	RF-SE-56-XX	Reference Laboratory	2.2	160	1.8	8.3 J-	51	1,900
44	RF-SE-01-MX	Oxford Instrument Analytical X-Met 3000TX	65	37	7	12	53	1,996
44	RF-SE-11-MX	Oxford Instrument Analytical X-Met 3000TX	49	0	6	17	0	2,056
44	RF-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX	53	36	8	13	0	2,196
44	RF-SE-33-MX	Oxford Instrument Analytical X-Met 3000TX	36	0	7	6	0	2,175
44	RF-SE-59-MX	Oxford Instrument Analytical X-Met 3000TX	32	1	10	0	2	2,145
45	RF-SE-04-XX	Reference Laboratory	4.2	130	2.8	12 J-	46	2,400
45	RF-SE-14-XX	Reference Laboratory	4.7	140	3	13 J-	51	2,600
45	RF-SE-19-XX	Reference Laboratory	3.9	140	4.1	14 J-	52	2,700
45	RF-SE-34-XX	Reference Laboratory	4.5	120	1.9	10 J-	42	2,200
45	RF-SE-52-XX	Reference Laboratory	4.1	130	2	11 J-	47	2,300
45	RF-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX	50	0	9	23	0	2,583
45	RF-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	34	0	9	8	0	2,510
45	RF-SE-19-MX	Oxford Instrument Analytical X-Met 3000TX	37	13	8	16	0	2,518
45	RF-SE-34-MX	Oxford Instrument Analytical X-Met 3000TX	44	1	9	13	0	2,785
45	RF-SE-52-MX	Oxford Instrument Analytical X-Met 3000TX	36	0	8	13	1	2,419

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
46	BN-SO-11-XX	Reference Laboratory	4 J-	2,900	720	820	120	23,000	56
46	BN-SO-14-XX	Reference Laboratory	3.5 J-	2,800	690	800	120	22,000	51
46	BN-SO-23-XX	Reference Laboratory	1.2 UJ	2,800	700	800	120	23,000	52
46	BN-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	3,427	744	1,214	251	21,211	0
46	BN-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	3,411	743	1,157	260	21,389	0
46	BN-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	0	3,438	716	1,106	248	21,495	0
47	BN-SO-09-XX	Reference Laboratory	750 J-	97	2,700	2,900	100	22,000	4,700
47	BN-SO-12-XX	Reference Laboratory	750 J-	89	2,600	2,800	96	21,000	4,500
47	BN-SO-24-XX	Reference Laboratory	810 J-	97	2,900	3,000	100	23,000	4,900
47	BN-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	1,853	0	2,690	3,107	177	19,109	4,583
47	BN-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	1,851	0	2,569	3,007	192	19,329	4,529
47	BN-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX	2,079	0	2,438	2,829	162	20,102	4,309
48	SB-SO-09-XX	Reference Laboratory	1.3 UJ	9	0.51 U	130	120	35,000	19
48	SB-SO-20-XX	Reference Laboratory	1.3 UJ	11	0.51 U	170	150	44,000	24
48	SB-SO-31-XX	Reference Laboratory	1.3 UJ	8 J-	0.51 U	140	130	38,000	21
48	SB-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	0	16	0	223	238	29,821	38
48	SB-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	0	17	0	206	207	29,660	45
48	SB-SO-56-MX	Oxford Instrument Analytical X-Met 3000TX	0	20	4	210	232	29,268	37
49	SB-SO-29-XX	Reference Laboratory	1.2 U	9	0.5 U	140	130	41,000	19
49	SB-SO-36-XX	Reference Laboratory	1.2 U	8	0.5 U	120	100	33,000	15
49	SB-SO-56-XX	Reference Laboratory	1.2 U	10	0.5 U	150	140	42,000	20
49	SB-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	27	27	2	238	130	28,105	19
49	SB-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX	28	20	0	180	141	29,100	33
49	SB-SO-42-MX	Oxford Instrument Analytical X-Met 3000TX	0	24	0	244	152	29,607	36
50	SB-SO-04-XX	Reference Laboratory	940	13	2,800	2,800	100	38,000	21
50	SB-SO-34-XX	Reference Laboratory	980	12	2,500	2,500	91	34,000	18
50	SB-SO-49-XX	Reference Laboratory	700	12	2,500	2,400	89	33,000	18
50	SB-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	1,690	30	2,992	3,375	216	27,821	17
50	SB-SO-36-MX	Oxford Instrument Analytical X-Met 3000TX	1,451	21	3,007	3,422	216	27,464	36
50	SB-SO-52-MX	Oxford Instrument Analytical X-Met 3000TX	1,422	30	3,054	3,337	242	27,479	25

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
46	BN-SO-11-XX	Reference Laboratory	24 J-	2,900	140	140 J-	150	3,900
46	BN-SO-14-XX	Reference Laboratory	26	2,800	130	140 J-	150	3,800
46	BN-SO-23-XX	Reference Laboratory	31	2,800	130	130 J-	150	3,800
46	BN-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	3,469	3,427	174	84	4,933
46	BN-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	3,557	3,411	179	245	4,820
46	BN-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	0	3,608	3,438	172	294	5,306
47	BN-SO-09-XX	Reference Laboratory	0.39	1,500	290	100 J-	340	81
47	BN-SO-12-XX	Reference Laboratory	0.34	1,400	290	210 J-	310	74
47	BN-SO-24-XX	Reference Laboratory	0.37	1,600	300	140 J-	350	81
47	BN-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	27	1,574	0	426	565	105
47	BN-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	29	1,406	0	417	426	94
47	BN-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX	31	1,449	0	404	344	107
48	SB-SO-09-XX	Reference Laboratory	30	2900	26	160 J-	120	3,600
48	SB-SO-20-XX	Reference Laboratory	10	3700	30	140 J-	160	4,500
48	SB-SO-31-XX	Reference Laboratory	32	3200 J-	28 J-	160 J-	140	3,900 J-
48	SB-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	38	3,414	28	378	29	4,308
48	SB-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	45	3,251	25	392	32	3,961
48	SB-SO-56-MX	Oxford Instrument Analytical X-Met 3000TX	37	3,399	29	384	0	3,995
49	SB-SO-29-XX	Reference Laboratory	7.9 J	200	160	1.2 UJ	400	3,900
49	SB-SO-36-XX	Reference Laboratory	36	160	130	1.2 UJ	320	3,200
49	SB-SO-56-XX	Reference Laboratory	9	210	160	1.2 UJ	410	4,100
49	SB-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	19	0	140	24	178	4,535
49	SB-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX	33	0	146	3	249	4,293
49	SB-SO-42-MX	Oxford Instrument Analytical X-Met 3000TX	36	26	143	19	317	4,206
50	SB-SO-04-XX	Reference Laboratory	40	3,300	390	1.3 UJ	58	86
50	SB-SO-34-XX	Reference Laboratory	36	3,000	360	1.3 UJ	52	77
50	SB-SO-49-XX	Reference Laboratory	36	2,800	330	1.2 UJ	52	72
50	SB-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	17	3,438	347	36	210	70
50	SB-SO-36-MX	Oxford Instrument Analytical X-Met 3000TX	36	3,394	347	46	130	60
50	SB-SO-52-MX	Oxford Instrument Analytical X-Met 3000TX	25	3,453	358	37	77	60

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
51	WS-SO-07-XX	Reference Laboratory	3.8	53	1.9	640	4,400	25,000	1,700
51	WS-SO-11-XX	Reference Laboratory	1.2 U	46	1.4	570	3,900	19,000	1,500
51	WS-SO-25-XX	Reference Laboratory	1.2 U	59	3.1	730	4,900	24,000	1,900
51	WS-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	1,036	6,252	22,956	2,252
51	WS-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	858	6,283	23,021	2,236
51	WS-SO-37-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	0	843	7,112	21,418	2,058
52	WS-SO-10-XX	Reference Laboratory	1.3 U	83	1.8	67	76	19,000	1,900
52	WS-SO-20-XX	Reference Laboratory	1.3 U	100	1.9	81	90	23,000	2,300
52	WS-SO-23-XX	Reference Laboratory	1.3 U	110	2.1	82	96	23,000	2,500
52	WS-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	0	44	0	154	140	22,105	3,025
52	WS-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	1	38	0	32	136	21,516	2,842
52	WS-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	0	43	3	134	137	21,990	2,965
53	AS-SO-03-XX	Reference Laboratory	1.2 U	14	1,300	33	6,200	15,000	160
53	AS-SO-05-XX	Reference Laboratory	1.2 U	9	900	23	4,500	11,000	110
53	AS-SO-08-XX	Reference Laboratory	1.2 U	10	930	24	4,600	11,000	120
53	AS-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	15	1,245	242	6,312	14,659	192
53	AS-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	19	1,189	141	5,895	14,557	187
53	AS-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	17	1,213	150	5,725	14,346	179
54	LV-SO-03-XX	Reference Laboratory	1.6	42	590	600	130	24,000	94
54	LV-SO-40-XX	Reference Laboratory	2.7	42	580	590	130	24,000	92
54	LV-SO-49-XX	Reference Laboratory	7.4	43	600	610	130	25,000	98
54	LV-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	0	63	617	1,070	267	27,579	147
54	LV-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	0	68	602	911	222	27,115	142
54	LV-SO-40-MX	Oxford Instrument Analytical X-Met 3000TX	0	71	655	1,003	263	28,728	154
55	LV-SO-04-XX	Reference Laboratory	860	120	2,400	2,300	98	22,000	4,000
55	LV-SO-34-XX	Reference Laboratory	870 J-	110 J-	2,300 J-	2,200 J-	87	20,000 J-	3,700 J-
55	LV-SO-37-XX	Reference Laboratory	590	84	1,700	1,600	66	16,000	2,800
55	LV-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	1,144	84	2,645	3,900	267	28,617	5,488
55	LV-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	1,626	39	2,663	3,524	253	26,489	4,984
55	LV-SO-46-MX	Oxford Instrument Analytical X-Met 3000TX	1,823	13	2,646	3,358	246	26,479	5,059

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
51	WS-SO-07-XX	Reference Laboratory	0.26	260	1.2 U	400 J-	48	180
51	WS-SO-11-XX	Reference Laboratory	0.27	240	1.2 U	340 J-	43	160
51	WS-SO-25-XX	Reference Laboratory	0.25	300	1.2 U	450 J-	54	200
51	WS-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	182	5	482	20	331
51	WS-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	0	208	3	460	149	311
51	WS-SO-37-MX	Oxford Instrument Analytical X-Met 3000TX	0	218	3	504	124	315
52	WS-SO-10-XX	Reference Laboratory	0.06 U	290	280	1.3 UJ	260	1,900
52	WS-SO-20-XX	Reference Laboratory	0.06 U	350	340	1.3 UJ	320	2,300
52	WS-SO-23-XX	Reference Laboratory	0.05 U	380	360	1.3 UJ	330	2,500
52	WS-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	0	251	351	5	200	2,835
52	WS-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	0	292	341	0	299	2,789
52	WS-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	0	336	344	7	249	2,681
53	AS-SO-03-XX	Reference Laboratory	3.7 J-	520	200	480 J-	29	350
53	AS-SO-05-XX	Reference Laboratory	2.5 J-	370	140	330 J-	23	250
53	AS-SO-08-XX	Reference Laboratory	2.5 J-	380	140	280 J-	23	260
53	AS-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	157	508	137	440
53	AS-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	158	585	158	431
53	AS-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	161	487	136	434
54	LV-SO-03-XX	Reference Laboratory	48 J-	2,000	120	210 J-	120	3,700
54	LV-SO-40-XX	Reference Laboratory	46 J-	1,900	120	210 J-	120	3,700
54	LV-SO-49-XX	Reference Laboratory	52 J-	2,000	120	220 J-	120	3,800
54	LV-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	104	104	131	234	183	4,746
54	LV-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	94	94	130	258	205	4,892
54	LV-SO-40-MX	Oxford Instrument Analytical X-Met 3000TX	118	118	133	261	177	5,077
55	LV-SO-04-XX	Reference Laboratory	130 J-	2,000	230	1.2 UJ	260	53
55	LV-SO-34-XX	Reference Laboratory	130 J-	1,900 J-	220 J-	1.2 UJ	230 J-	48 J-
55	LV-SO-37-XX	Reference Laboratory	130 J-	1,400	170	1.2 U	180	37
55	LV-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	351	351	280	8	615	117
55	LV-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	314	314	275	24	545	113
55	LV-SO-46-MX	Oxford Instrument Analytical X-Met 3000TX	341	341	270	21	691	133

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
56	CN-SO-03-XX	Reference Laboratory	22	87	63	17	72	15,000	130
56	CN-SO-06-XX	Reference Laboratory	20	91	64	18	74	16,000	130
56	CN-SO-07-XX	Reference Laboratory	20	90	63	19	72	17,000	130
56	CN-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	44	124	53	131	112	17,975	212
56	CN-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	60	144	54	53	125	19,272	244
56	CN-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	47	151	87	77	142	18,828	263
57	CN-SO-02-XX	Reference Laboratory	230	19	820	290	140	22,000	490
57	CN-SO-05-XX	Reference Laboratory	130	6	630	26	160	23,000	25
57	CN-SO-09-XX	Reference Laboratory	120	6	580	21	140	19,000	23
57	CN-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	238	8	568	66	210	18,493	74
57	CN-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	267	7	597	218	199	21,571	80
57	CN-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	254	17	635	57	199	19,325	64
58	LV-SE-06-XX	Reference Laboratory	30	23	160	540	30	18,000	1,600
58	LV-SE-13-XX	Reference Laboratory	31	24	160	540	30	18,000	1,600
58	LV-SE-41-XX	Reference Laboratory	30	21	150	480	26	16,000	1,500
58	LV-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	111	0	166	769	66	21,971	2,167
58	LV-SE-36-MX	Oxford Instrument Analytical X-Met 3000TX	85	0	167	726	79	22,692	2,158
58	LV-SE-52-MX	Oxford Instrument Analytical X-Met 3000TX	97	0	181	784	71	22,163	2,140
59	LV-SE-05-XX	Reference Laboratory	92	20	440	840	39	16,000	14
59	LV-SE-20-XX	Reference Laboratory	140 J+	31	680	1,400	60	22,000	21
59	LV-SE-43-XX	Reference Laboratory	160 J+	24	550	1,100	47	19,000	17
59	LV-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	305	56	628	1,750	88	22,908	35
59	LV-SE-33-MX	Oxford Instrument Analytical X-Met 3000TX	252	42	624	1,456	109	23,356	58
59	LV-SE-38-MX	Oxford Instrument Analytical X-Met 3000TX	316	41	624	1,356	86	23,228	49
60	LV-SE-15-XX	Reference Laboratory	290 J+	32	1,300	83	2,300	22,000	18
60	LV-SE-17-XX	Reference Laboratory	280 J+	31	1,300	79	2,200	21,000	17 J-
60	LV-SE-51-XX	Reference Laboratory	210 J+	26	1,100	72	2,000	19,000	15
60	LV-SE-29-MX	Oxford Instrument Analytical X-Met 3000TX	367	41	1,179	140	2,456	22,587	56
60	LV-SE-41-MX	Oxford Instrument Analytical X-Met 3000TX	364	34	1,186	228	2,402	22,134	60
60	LV-SE-44-MX	Oxford Instrument Analytical X-Met 3000TX	472	46	1,199	227	2,451	22,633	58
61	TL-SE-05-XX	Reference Laboratory	100 J+	34	0.34 J	40	4,900	24,000	1,200
61	TL-SE-09-XX	Reference Laboratory	100 J+	33	0.24 J	39	4,800	23,000	1,200
61	TL-SE-13-XX	Reference Laboratory	95 J+	31	0.45 J	36 J+	4,400 J+	22,000 J+	1,100 J+
61	TL-SE-01-MX	Oxford Instrument Analytical X-Met 3000TX	523	0	0	28	4,288	25,677	1,155
61	TL-SE-11-MX	Oxford Instrument Analytical X-Met 3000TX	548	0	0	151	4,222	25,153	1,185
61	TL-SE-29-MX	Oxford Instrument Analytical X-Met 3000TX	642	0	0	80	4,051	24,487	1,135

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
56	CN-SO-03-XX	Reference Laboratory	34 J-	74	36	90	30	58
56	CN-SO-06-XX	Reference Laboratory	40 J-	76	38	94	32	59
56	CN-SO-07-XX	Reference Laboratory	36 J-	75	37	91	33	58
56	CN-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	48	0	45	113	0	57
56	CN-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	41	0	49	105	52	78
56	CN-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	49	0	55	132	0	91
57	CN-SO-02-XX	Reference Laboratory	270 J-	530	190	68	160	1,900
57	CN-SO-05-XX	Reference Laboratory	280 J-	360	190	78	160	2,200
57	CN-SO-09-XX	Reference Laboratory	260 J-	330	170	74	140	2,100
57	CN-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	369	341	171	65	297	2,669
57	CN-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	367	288	183	79	345	2,577
57	CN-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	361	295	168	80	86	2,502
58	LV-SE-06-XX	Reference Laboratory	610 J-	360	160	110	480	52
58	LV-SE-13-XX	Reference Laboratory	640 J-	360	160	110	470	51
58	LV-SE-41-XX	Reference Laboratory	610 J-	320	150	99	420	46
58	LV-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX	967	967	195	141	696	103
58	LV-SE-36-MX	Oxford Instrument Analytical X-Met 3000TX	949	949	184	154	716	113
58	LV-SE-52-MX	Oxford Instrument Analytical X-Met 3000TX	964	964	186	153	632	107
59	LV-SE-05-XX	Reference Laboratory	2.6 J-	400	340	49	340	1,800
59	LV-SE-20-XX	Reference Laboratory	2.8	660	500	75 J-	530	2,800
59	LV-SE-43-XX	Reference Laboratory	2.8	530	420	60 J-	430	2,300
59	LV-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	43	43	445	72	572	2,958
59	LV-SE-33-MX	Oxford Instrument Analytical X-Met 3000TX	39	39	440	85	675	2,827
59	LV-SE-38-MX	Oxford Instrument Analytical X-Met 3000TX	44	44	403	106	581	2,741
60	LV-SE-15-XX	Reference Laboratory	500	230	92	300 J-	180	62
60	LV-SE-17-XX	Reference Laboratory	490	220	89	200 J-	170	58
60	LV-SE-51-XX	Reference Laboratory	470	200	76	250 J-	160	54
60	LV-SE-29-MX	Oxford Instrument Analytical X-Met 3000TX	754	754	83	479	281	140
60	LV-SE-41-MX	Oxford Instrument Analytical X-Met 3000TX	721	721	82	441	237	141
60	LV-SE-44-MX	Oxford Instrument Analytical X-Met 3000TX	754	754	81	486	253	142
61	TL-SE-05-XX	Reference Laboratory	980	54	130	180 J-	66	100
61	TL-SE-09-XX	Reference Laboratory	820	53	130	170 J-	63	100
61	TL-SE-13-XX	Reference Laboratory	990	49	120	160 J	59 J+	96
61	TL-SE-01-MX	Oxford Instrument Analytical X-Met 3000TX	1,130	0	119	161	58	167
61	TL-SE-11-MX	Oxford Instrument Analytical X-Met 3000TX	1,113	0	123	163	0	180
61	TL-SE-29-MX	Oxford Instrument Analytical X-Met 3000TX	1,063	0	111	164	0	170

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
62	TL-SE-06-XX	Reference Laboratory	1.2 U	86	350	34	2000	22,000	1,700
62	TL-SE-17-XX	Reference Laboratory	1.2 U	85	340	33	2100	21,000	1,700
62	TL-SE-28-XX	Reference Laboratory	1.2 U	89	360	34	2100	22,000	1,700
62	TL-SE-02-MX	Oxford Instrument Analytical X-Met 3000TX	0	18	388	60	2,230	24,242	1,903
62	TL-SE-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	21	348	12	2,186	23,792	1,890
62	TL-SE-22-MX	Oxford Instrument Analytical X-Met 3000TX	0	29	365	38	2,308	23,816	1,898
63	TL-SE-07-XX	Reference Laboratory	30	11	48	66	2200	37,000	13
63	TL-SE-21-XX	Reference Laboratory	33	13	51	73	2300	44,000	15
63	TL-SE-30-XX	Reference Laboratory	31	11	47	64	2200	36,000	14
63	TL-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	257	18	55	77	2,318	52,447	36
63	TL-SE-18-MX	Oxford Instrument Analytical X-Met 3000TX	258	16	56	34	2,304	53,463	37
63	TL-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	237	17	60	77	2,417	53,394	41
64	TL-SE-02-XX	Reference Laboratory	77	15	160	64	3,100	32,000	12
64	TL-SE-08-XX	Reference Laboratory	66	10	180	74	3,200	45,000	11
64	TL-SE-16-XX	Reference Laboratory	73	15	170	69	3,100	38,000	13
64	TL-SE-06-MX	Oxford Instrument Analytical X-Met 3000TX	622	13	166	189	3,285	55,723	43
64	TL-SE-09-MX	Oxford Instrument Analytical X-Met 3000TX	709	23	157	19	3,165	52,435	28
64	TL-SE-17-MX	Oxford Instrument Analytical X-Met 3000TX	688	27	150	150	3,151	53,620	27
65	RF-SE-01-XX	Reference Laboratory	12	230	40	280	63	14,000	22
65	RF-SE-09-XX	Reference Laboratory	10	260	45	310	71	16,000	26
65	RF-SE-11-XX	Reference Laboratory	11	240	43	300	72	15,000	25
65	RF-SE-17-XX	Reference Laboratory	11	250	43	300	67	15,000	26
65	RF-SE-29-XX	Reference Laboratory	13	280	49	330	75	17,000	26
65	RF-SE-37-XX	Reference Laboratory	11	260	45	320	72	16,000	27
65	RF-SE-50-XX	Reference Laboratory	8.9	230	40	280	65	14,000	23
65	RF-SE-05-MX	Oxford Instrument Analytical X-Met 3000TX	0	335	40	414	112	16,003	36
65	RF-SE-21-MX	Oxford Instrument Analytical X-Met 3000TX	40	385	41	425	114	17,785	29
65	RF-SE-25-MX	Oxford Instrument Analytical X-Met 3000TX	18	338	41	340	90	16,147	28
65	RF-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	0	332	42	341	111	16,312	53
65	RF-SE-41-MX	Oxford Instrument Analytical X-Met 3000TX	27	398	53	511	110	17,419	24
65	RF-SE-47-MX	Oxford Instrument Analytical X-Met 3000TX	25	345	36	449	106	16,427	36
65	RF-SE-57-MX	Oxford Instrument Analytical X-Met 3000TX	0	356	34	510	112	17,628	50

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
62	TL-SE-06-XX	Reference Laboratory	2.2	44	45	56	78	83
62	TL-SE-17-XX	Reference Laboratory	2.6	43	44	56	78	81
62	TL-SE-28-XX	Reference Laboratory	2.8	44	45	57	81	83
62	TL-SE-02-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	48	86	70	119
62	TL-SE-08-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	44	69	6	117
62	TL-SE-22-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	48	68	28	130
63	TL-SE-07-XX	Reference Laboratory	40	94	120	63	110	160
63	TL-SE-21-XX	Reference Laboratory	120	100	140	67	120	170
63	TL-SE-30-XX	Reference Laboratory	100	93	120	62	100	160
63	TL-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX	44	0	141	91	0	247
63	TL-SE-18-MX	Oxford Instrument Analytical X-Met 3000TX	24	0	146	101	0	256
63	TL-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX	52	0	148	111	0	232
64	TL-SE-02-XX	Reference Laboratory	400	99	44	120	110	160
64	TL-SE-08-XX	Reference Laboratory	350	100	39	130	120	170
64	TL-SE-16-XX	Reference Laboratory	420	100	44	120	110	160
64	TL-SE-06-MX	Oxford Instrument Analytical X-Met 3000TX	210	0	55	157	0	300
64	TL-SE-09-MX	Oxford Instrument Analytical X-Met 3000TX	185	0	53	180	0	297
64	TL-SE-17-MX	Oxford Instrument Analytical X-Met 3000TX	201	0	48	153	0	293
65	RF-SE-01-XX	Reference Laboratory	47	200	21	37	29	1,700
65	RF-SE-09-XX	Reference Laboratory	45	220	23	42	32	1,900
65	RF-SE-11-XX	Reference Laboratory	52	210	20	40	29	1,800
65	RF-SE-17-XX	Reference Laboratory	20	210	22	40	30	1,800
65	RF-SE-29-XX	Reference Laboratory	20	240	26	44	35	2,100
65	RF-SE-37-XX	Reference Laboratory	22	220	23	44	32	1,900
65	RF-SE-50-XX	Reference Laboratory	19	200	20	38	29	1,700
65	RF-SE-05-MX	Oxford Instrument Analytical X-Met 3000TX	99	158	25	41	0	2,084
65	RF-SE-21-MX	Oxford Instrument Analytical X-Met 3000TX	109	227	30	66	84	2,675
65	RF-SE-25-MX	Oxford Instrument Analytical X-Met 3000TX	76	169	22	50	8	2,195
65	RF-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX	88	165	28	30	0	2,139
65	RF-SE-41-MX	Oxford Instrument Analytical X-Met 3000TX	103	189	28	44	49	2,635
65	RF-SE-47-MX	Oxford Instrument Analytical X-Met 3000TX	79	148	26	48	19	2,363
65	RF-SE-57-MX	Oxford Instrument Analytical X-Met 3000TX	82	213	25	19	0	2,557

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
66	RF-SE-08-XX	Reference Laboratory	14	460	67	510	1,800	18,000	580
66	RF-SE-10-XX	Reference Laboratory	12	400	58	440	1,500	16,000	510
66	RF-SE-33-XX	Reference Laboratory	13	440	64	490	1,700	18,000	570
66	RF-SE-13-MX	Oxford Instrument Analytical X-Met 3000TX	10	571	70	615	2,423	17,744	631
66	RF-SE-29-MX	Oxford Instrument Analytical X-Met 3000TX	35	594	60	663	2,359	18,132	644
66	RF-SE-56-MX	Oxford Instrument Analytical X-Met 3000TX	26	619	89	676	2,471	18,277	663
67	RF-SE-16-XX	Reference Laboratory	85 J-	72 J-	310 J-	820 J-	73 J-	16,000 J-	24 J-
67	RF-SE-41-XX	Reference Laboratory	100	82	360	950	85	18,000	25
67	RF-SE-48-XX	Reference Laboratory	100	87	380	1,000	90	19,000	27
67	RF-SE-06-MX	Oxford Instrument Analytical X-Met 3000TX	117	97	380	1,180	151	17,128	69
67	RF-SE-26-MX	Oxford Instrument Analytical X-Met 3000TX	200	112	332	1,111	156	17,296	48
67	RF-SE-55-MX	Oxford Instrument Analytical X-Met 3000TX	129	94	342	1,216	143	17,441	64
68	RF-SE-18-XX	Reference Laboratory	320	810	770	950	78	16,000	860
68	RF-SE-35-XX	Reference Laboratory	300	740	700	860	70	15,000	780
68	RF-SE-54-XX	Reference Laboratory	320	880	840	1,000	86	18,000	920
68	RF-SE-24-MX	Oxford Instrument Analytical X-Met 3000TX	550	987	790	1,184	108	17,176	935
68	RF-SE-39-MX	Oxford Instrument Analytical X-Met 3000TX	538	1,093	812	1,270	132	17,363	973
68	RF-SE-46-MX	Oxford Instrument Analytical X-Met 3000TX	614	1,001	809	1,142	111	16,316	919
69	RF-SE-20-XX	Reference Laboratory	550	1300	540	94	93	20,000	28
69	RF-SE-46-XX	Reference Laboratory	270	590	240	44	40	8,900	13
69	RF-SE-51-XX	Reference Laboratory	480	1100	450	77	77	17,000	23
69	RF-SE-10-MX	Oxford Instrument Analytical X-Met 3000TX	743	1,455	518	98	139	18,056	0
69	RF-SE-37-MX	Oxford Instrument Analytical X-Met 3000TX	741	1,383	492	124	125	17,337	0
69	RF-SE-49-MX	Oxford Instrument Analytical X-Met 3000TX	680	1,490	519	221	156	18,192	0
70	RF-SE-21-XX	Reference Laboratory	1.3 U	62	1,700	76	1,000	16,000	2,100
70	RF-SE-40-XX	Reference Laboratory	1.3 U	70	1,900	85	1,100	18,000	2,400
70	RF-SE-47-XX	Reference Laboratory	1.3 U	72	1,900	90	1,200	19,000	2,400
70	RF-SE-30-MX	Oxford Instrument Analytical X-Met 3000TX	0	40	1,896	187	1,382	18,077	2,701
70	RF-SE-43-MX	Oxford Instrument Analytical X-Met 3000TX	0	32	1,941	175	1,513	18,326	2,839
70	RF-SE-58-MX	Oxford Instrument Analytical X-Met 3000TX	0	0	1,877	110	1,310	17,734	2,657

Appendix D1. Analytical Data Summary, Oxford X-Met 3000TX Original Data Set (Submitted January 28, 2005) and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
66	RF-SE-08-XX	Reference Laboratory	29	250	42	0.39 U	120	120
66	RF-SE-10-XX	Reference Laboratory	27	220	39	0.34 U	100	110
66	RF-SE-33-XX	Reference Laboratory	28	240	41	0.33 U	120	130
66	RF-SE-13-MX	Oxford Instrument Analytical X-Met 3000TX	143	179	45	0	186	199
66	RF-SE-29-MX	Oxford Instrument Analytical X-Met 3000TX	152	185	44	10	91	229
66	RF-SE-56-MX	Oxford Instrument Analytical X-Met 3000TX	146	253	41	7	135	219
67	RF-SE-16-XX	Reference Laboratory	260	1,700 J-	1.2 U	130 J-	32 J-	760 J-
67	RF-SE-41-XX	Reference Laboratory	230	1,900	1.2 U	140	39	830
67	RF-SE-48-XX	Reference Laboratory	250	2,000	2.2	150	40	880
67	RF-SE-06-MX	Oxford Instrument Analytical X-Met 3000TX	457	2,138	9	169	76	854
67	RF-SE-26-MX	Oxford Instrument Analytical X-Met 3000TX	484	2,116	7	186	16	990
67	RF-SE-55-MX	Oxford Instrument Analytical X-Met 3000TX	487	2,149	11	150	0	933
68	RF-SE-18-XX	Reference Laboratory	600	390	140	140	390	120
68	RF-SE-35-XX	Reference Laboratory	650	350	140	150	340	110
68	RF-SE-54-XX	Reference Laboratory	670	420	160	180	410	120
68	RF-SE-24-MX	Oxford Instrument Analytical X-Met 3000TX	1,160	385	148	255	336	190
68	RF-SE-39-MX	Oxford Instrument Analytical X-Met 3000TX	1,290	431	164	244	394	190
68	RF-SE-46-MX	Oxford Instrument Analytical X-Met 3000TX	1,183	368	150	243	450	185
69	RF-SE-20-XX	Reference Laboratory	0.48	1,400	380	59	36	1,400
69	RF-SE-46-XX	Reference Laboratory	0.45	650	170	26	16	650
69	RF-SE-51-XX	Reference Laboratory	0.48	1,200	320	48	30	1,200
69	RF-SE-10-MX	Oxford Instrument Analytical X-Met 3000TX	9	1,489	333	89	31	1,473
69	RF-SE-37-MX	Oxford Instrument Analytical X-Met 3000TX	0	1,390	319	77	0	1,401
69	RF-SE-49-MX	Oxford Instrument Analytical X-Met 3000TX	15	1,495	339	58	66	1,463
70	RF-SE-21-XX	Reference Laboratory	320	220	440	120	130	100
70	RF-SE-40-XX	Reference Laboratory	280	250	480	100	150	120
70	RF-SE-47-XX	Reference Laboratory	320	250	510	120	150	120
70	RF-SE-30-MX	Oxford Instrument Analytical X-Met 3000TX	699	184	514	384	293	231
70	RF-SE-43-MX	Oxford Instrument Analytical X-Met 3000TX	736	164	533	406	272	207
70	RF-SE-58-MX	Oxford Instrument Analytical X-Met 3000TX	701	163	510	388	224	220

Notes:
All concentrations reported in milligrams per kilogram (mg/kg), or parts per million (ppm)
J Estimated concentration
J+ Concentration is considered estimated and biased high
J- Concentration is considered estimated and biased low
U Analyte is not detected; the associated concentration value is the sample reporting limit

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
10	BN-SO-01-XX	Reference Laboratory			1.3 U
10	BN-SO-10-XX	Reference Laboratory			1.2 J
10	BN-SO-15-XX	Reference Laboratory			1.3 U
10	BN-SO-18-XX	Reference Laboratory			1.3
10	BN-SO-28-XX	Reference Laboratory			1.3 U
10	BN-SO-31-XX	Reference Laboratory			1.3 U
10	BN-SO-35-XX	Reference Laboratory			1.2 J
10	BN-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX			0
10	BN-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX			1
10	BN-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX			1
10	BN-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX			3
10	BN-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX			3
10	BN-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX			2
10	BN-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX			0
11	BN-SO-02-XX	Reference Laboratory			4.3
11	BN-SO-04-XX	Reference Laboratory			2.9
11	BN-SO-17-XX	Reference Laboratory			2.7
11	BN-SO-22-XX	Reference Laboratory			2.8
11	BN-SO-27-XX	Reference Laboratory			3.7
11	BN-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX			3
11	BN-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX			2
11	BN-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX			3
11	BN-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX			5
11	BN-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX			4
12	BN-SO-03-XX	Reference Laboratory			17
12	BN-SO-06-XX	Reference Laboratory			15
12	BN-SO-08-XX	Reference Laboratory			14
12	BN-SO-13-XX	Reference Laboratory			9.2
12	BN-SO-20-XX	Reference Laboratory			14
12	BN-SO-30-XX	Reference Laboratory			17
12	BN-SO-34-XX	Reference Laboratory			17
12	BN-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX			14
12	BN-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX			23
12	BN-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX			20
12	BN-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX			17
12	BN-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX			23
12	BN-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX			21
12	BN-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX			22

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
13	BN-SO-07-XX	Reference Laboratory			26
13	BN-SO-16-XX	Reference Laboratory			29
13	BN-SO-21-XX	Reference Laboratory			35
13	BN-SO-25-XX	Reference Laboratory			19 J-
13	BN-SO-33-XX	Reference Laboratory			34
13	BN-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX			36
13	BN-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX			31
13	BN-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX			31
13	BN-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX			38
13	BN-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX			32
14	BN-SO-05-XX	Reference Laboratory			48
14	BN-SO-19-XX	Reference Laboratory			48
14	BN-SO-26-XX	Reference Laboratory			49
14	BN-SO-29-XX	Reference Laboratory			48
14	BN-SO-32-XX	Reference Laboratory			48
14	BN-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX			49
14	BN-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX			46
14	BN-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX			52
14	BN-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX			53
14	BN-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX			56
18	SB-SO-03-XX	Reference Laboratory	62		
18	SB-SO-06-XX	Reference Laboratory	55		
18	SB-SO-14-XX	Reference Laboratory	55		
18	SB-SO-38-XX	Reference Laboratory	56		
18	SB-SO-41-XX	Reference Laboratory	54		
18	SB-SO-47-XX	Reference Laboratory	58		
18	SB-SO-51-XX	Reference Laboratory	54		
18	SB-SO-03-MX	Oxford Instrument Analytical X-Met 3000TX	51		
18	SB-SO-06-MX	Oxford Instrument Analytical X-Met 3000TX	65		
18	SB-SO-14-MX	Oxford Instrument Analytical X-Met 3000TX	60		
18	SB-SO-38-MX	Oxford Instrument Analytical X-Met 3000TX	69		
18	SB-SO-41-MX	Oxford Instrument Analytical X-Met 3000TX	65		
18	SB-SO-47-MX	Oxford Instrument Analytical X-Met 3000TX	67		
18	SB-SO-51-MX	Oxford Instrument Analytical X-Met 3000TX	62		

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
19	SB-SO-05-XX	Reference Laboratory	540		
19	SB-SO-18-XX	Reference Laboratory	280		
19	SB-SO-30-XX	Reference Laboratory	290		
19	SB-SO-40-XX	Reference Laboratory	280		
19	SB-SO-53-XX	Reference Laboratory	270		
19	SB-SO-01-MX	Oxford Instrument Analytical X-Met 3000TX	377		
19	SB-SO-10-MX	Oxford Instrument Analytical X-Met 3000TX	434		
19	SB-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX	393		
19	SB-SO-31-MX	Oxford Instrument Analytical X-Met 3000TX	354		
19	SB-SO-45-MX	Oxford Instrument Analytical X-Met 3000TX	399		
20	SB-SO-08-XX	Reference Laboratory	730		
20	SB-SO-11-XX	Reference Laboratory	810		
20	SB-SO-21-XX	Reference Laboratory	740		
20	SB-SO-39-XX	Reference Laboratory	790		
20	SB-SO-42-XX	Reference Laboratory	740		
20	SB-SO-05-MX	Oxford Instrument Analytical X-Met 3000TX	1,038		
20	SB-SO-16-MX	Oxford Instrument Analytical X-Met 3000TX	1,062		
20	SB-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX	1,077		
20	SB-SO-35-MX	Oxford Instrument Analytical X-Met 3000TX	1,087		
20	SB-SO-53-MX	Oxford Instrument Analytical X-Met 3000TX	1,150		
21	SB-SO-22-XX	Reference Laboratory	3,300		
21	SB-SO-25-XX	Reference Laboratory	3,000		
21	SB-SO-27-XX	Reference Laboratory	3,100		
21	SB-SO-35-XX	Reference Laboratory	3,100		
21	SB-SO-44-XX	Reference Laboratory	3,000		
21	SB-SO-08-MX	Oxford Instrument Analytical X-Met 3000TX	2,563		
21	SB-SO-19-MX	Oxford Instrument Analytical X-Met 3000TX	2,604		
21	SB-SO-29-MX	Oxford Instrument Analytical X-Met 3000TX	2,397		
21	SB-SO-40-MX	Oxford Instrument Analytical X-Met 3000TX	2,473		
21	SB-SO-55-MX	Oxford Instrument Analytical X-Met 3000TX	2,578		

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
22	SB-SO-23-XX	Reference Laboratory	8,500		
22	SB-SO-28-XX	Reference Laboratory	8,800		
22	SB-SO-32-XX	Reference Laboratory	8,900		
22	SB-SO-43-XX	Reference Laboratory	7,600		
22	SB-SO-48-XX	Reference Laboratory	8,200		
22	SB-SO-23-MX	Oxford Instrument Analytical X-Met 3000TX	8,087		
22	SB-SO-28-MX	Oxford Instrument Analytical X-Met 3000TX	8,528		
22	SB-SO-32-MX	Oxford Instrument Analytical X-Met 3000TX	8,472		
22	SB-SO-43-MX	Oxford Instrument Analytical X-Met 3000TX	8,034		
22	SB-SO-48-MX	Oxford Instrument Analytical X-Met 3000TX	8,205		
23	SB-SO-02-XX	Reference Laboratory	130 J+		
23	SB-SO-07-XX	Reference Laboratory	270		
23	SB-SO-10-XX	Reference Laboratory	220		
23	SB-SO-26-XX	Reference Laboratory	260		
23	SB-SO-50-XX	Reference Laboratory	200		
23	SB-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX	162		
23	SB-SO-18-MX	Oxford Instrument Analytical X-Met 3000TX	149		
23	SB-SO-30-MX	Oxford Instrument Analytical X-Met 3000TX	152		
23	SB-SO-39-MX	Oxford Instrument Analytical X-Met 3000TX	159		
23	SB-SO-44-MX	Oxford Instrument Analytical X-Met 3000TX	162		
24	SB-SO-01-XX	Reference Laboratory	400		
24	SB-SO-16-XX	Reference Laboratory	480		
24	SB-SO-24-XX	Reference Laboratory	420		
24	SB-SO-45-XX	Reference Laboratory	450		
24	SB-SO-52-XX	Reference Laboratory	430		
24	SB-SO-07-MX	Oxford Instrument Analytical X-Met 3000TX	274		
24	SB-SO-20-MX	Oxford Instrument Analytical X-Met 3000TX	277		
24	SB-SO-27-MX	Oxford Instrument Analytical X-Met 3000TX	261		
24	SB-SO-37-MX	Oxford Instrument Analytical X-Met 3000TX	263		
24	SB-SO-49-MX	Oxford Instrument Analytical X-Met 3000TX	289		

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
25	SB-SO-13-XX	Reference Laboratory	850		
25	SB-SO-19-XX	Reference Laboratory	740		
25	SB-SO-33-XX	Reference Laboratory	870		
25	SB-SO-37-XX	Reference Laboratory	790		
25	SB-SO-55-XX	Reference Laboratory	900		
25	SB-SO-02-MX	Oxford Instrument Analytical X-Met 3000TX	520		
25	SB-SO-11-MX	Oxford Instrument Analytical X-Met 3000TX	534		
25	SB-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX	519		
25	SB-SO-33-MX	Oxford Instrument Analytical X-Met 3000TX	525		
25	SB-SO-50-MX	Oxford Instrument Analytical X-Met 3000TX	507		
26	SB-SO-12-XX	Reference Laboratory	1,400		
26	SB-SO-15-XX	Reference Laboratory	1,100		
26	SB-SO-17-XX	Reference Laboratory	1,200		
26	SB-SO-46-XX	Reference Laboratory	670		
26	SB-SO-54-XX	Reference Laboratory	560		
26	SB-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX	801		
26	SB-SO-15-MX	Oxford Instrument Analytical X-Met 3000TX	814		
26	SB-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX	786		
26	SB-SO-46-MX	Oxford Instrument Analytical X-Met 3000TX	817		
26	SB-SO-54-MX	Oxford Instrument Analytical X-Met 3000TX	777		
32	LV-SE-02-XX	Reference Laboratory		160	
32	LV-SE-10-XX	Reference Laboratory		200	
32	LV-SE-22-XX	Reference Laboratory		170	
32	LV-SE-25-XX	Reference Laboratory		170	
32	LV-SE-31-XX	Reference Laboratory		180	
32	LV-SE-35-XX	Reference Laboratory		170 J-	
32	LV-SE-50-XX	Reference Laboratory		170	
32	LV-SE-02-MX	Oxford Instrument Analytical X-Met 3000TX		0	
32	LV-SE-10-MX	Oxford Instrument Analytical X-Met 3000TX		0	
32	LV-SE-22-MX	Oxford Instrument Analytical X-Met 3000TX		0	
32	LV-SE-25-MX	Oxford Instrument Analytical X-Met 3000TX		9	
32	LV-SE-31-MX	Oxford Instrument Analytical X-Met 3000TX		0	
32	LV-SE-35-MX	Oxford Instrument Analytical X-Met 3000TX		0	
32	LV-SE-50-MX	Oxford Instrument Analytical X-Met 3000TX		0	

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
33	LV-SE-12-XX	Reference Laboratory		71	
33	LV-SE-26-XX	Reference Laboratory		83	
33	LV-SE-33-XX	Reference Laboratory		66	
33	LV-SE-39-XX	Reference Laboratory		74	
33	LV-SE-42-XX	Reference Laboratory		67	
33	LV-SE-01-MX	Oxford Instrument Analytical X-Met 3000TX		0	
33	LV-SE-06-MX	Oxford Instrument Analytical X-Met 3000TX		0	
33	LV-SE-17-MX	Oxford Instrument Analytical X-Met 3000TX		0	
33	LV-SE-37-MX	Oxford Instrument Analytical X-Met 3000TX		0	
33	LV-SE-49-MX	Oxford Instrument Analytical X-Met 3000TX		0	
34	LV-SE-09-XX	Reference Laboratory		55	
34	LV-SE-19-XX	Reference Laboratory		65	
34	LV-SE-27-XX	Reference Laboratory		64	
34	LV-SE-36-XX	Reference Laboratory		70	
34	LV-SE-38-XX	Reference Laboratory		75	
34	LV-SE-03-MX	Oxford Instrument Analytical X-Met 3000TX		0	
34	LV-SE-11-MX	Oxford Instrument Analytical X-Met 3000TX		0	
34	LV-SE-24-MX	Oxford Instrument Analytical X-Met 3000TX		0	
34	LV-SE-32-MX	Oxford Instrument Analytical X-Met 3000TX		0	
34	LV-SE-42-MX	Oxford Instrument Analytical X-Met 3000TX		0	
35	LV-SE-07-XX	Reference Laboratory		58	
35	LV-SE-18-XX	Reference Laboratory		60	
35	LV-SE-23-XX	Reference Laboratory		50 J	
35	LV-SE-45-XX	Reference Laboratory		50 J	
35	LV-SE-48-XX	Reference Laboratory		50 J	
35	LV-SE-07-MX	Oxford Instrument Analytical X-Met 3000TX		0	
35	LV-SE-18-MX	Oxford Instrument Analytical X-Met 3000TX		0	
35	LV-SE-23-MX	Oxford Instrument Analytical X-Met 3000TX		0	
35	LV-SE-45-MX	Oxford Instrument Analytical X-Met 3000TX		0	
35	LV-SE-48-MX	Oxford Instrument Analytical X-Met 3000TX		0	

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
36	LV-SE-01-XX	Reference Laboratory		49	
36	LV-SE-14-XX	Reference Laboratory		46	
36	LV-SE-21-XX	Reference Laboratory		49	
36	LV-SE-24-XX	Reference Laboratory		44	
36	LV-SE-32-XX	Reference Laboratory		47	
36	LV-SE-05-MX	Oxford Instrument Analytical X-Met 3000TX		0	
36	LV-SE-19-MX	Oxford Instrument Analytical X-Met 3000TX		0	
36	LV-SE-27-MX	Oxford Instrument Analytical X-Met 3000TX		0	
36	LV-SE-39-MX	Oxford Instrument Analytical X-Met 3000TX		1	
36	LV-SE-51-MX	Oxford Instrument Analytical X-Met 3000TX		0	
37	LV-SE-08-XX	Reference Laboratory		110	
37	LV-SE-16-XX	Reference Laboratory		110	
37	LV-SE-28-XX	Reference Laboratory		120	
37	LV-SE-30-XX	Reference Laboratory		120	
37	LV-SE-47-XX	Reference Laboratory		120	
37	LV-SE-08-MX	Oxford Instrument Analytical X-Met 3000TX		0	
37	LV-SE-16-MX	Oxford Instrument Analytical X-Met 3000TX		0	
37	LV-SE-28-MX	Oxford Instrument Analytical X-Met 3000TX		0	
37	LV-SE-30-MX	Oxford Instrument Analytical X-Met 3000TX		0	
37	LV-SE-47-MX	Oxford Instrument Analytical X-Met 3000TX		0	
38	LV-SE-11-XX	Reference Laboratory		870	
38	LV-SE-29-XX	Reference Laboratory		860	
38	LV-SE-44-XX	Reference Laboratory		830	
38	LV-SE-46-XX	Reference Laboratory		660	
38	LV-SE-52-XX	Reference Laboratory		910	
38	LV-SE-04-MX	Oxford Instrument Analytical X-Met 3000TX		667	
38	LV-SE-15-MX	Oxford Instrument Analytical X-Met 3000TX		626	
38	LV-SE-20-MX	Oxford Instrument Analytical X-Met 3000TX		685	
38	LV-SE-34-MX	Oxford Instrument Analytical X-Met 3000TX		668	
38	LV-SE-43-MX	Oxford Instrument Analytical X-Met 3000TX		651	

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
46	BN-SO-11-XX	Reference Laboratory			140
46	BN-SO-14-XX	Reference Laboratory			130
46	BN-SO-23-XX	Reference Laboratory			130
46	BN-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX			87
46	BN-SO-12-MX	Oxford Instrument Analytical X-Met 3000TX			86
46	BN-SO-24-MX	Oxford Instrument Analytical X-Met 3000TX			85
47	BN-SO-09-XX	Reference Laboratory			290
47	BN-SO-12-XX	Reference Laboratory			290
47	BN-SO-24-XX	Reference Laboratory			300
47	BN-SO-17-MX	Oxford Instrument Analytical X-Met 3000TX			301
47	BN-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX			297
47	BN-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX			301
48	SB-SO-09-XX	Reference Laboratory	30		
48	SB-SO-20-XX	Reference Laboratory	10		
48	SB-SO-31-XX	Reference Laboratory	32		
48	SB-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX	14		
48	SB-SO-25-MX	Oxford Instrument Analytical X-Met 3000TX	22		
48	SB-SO-56-MX	Oxford Instrument Analytical X-Met 3000TX	26		
49	SB-SO-29-XX	Reference Laboratory	7.9 J		
49	SB-SO-36-XX	Reference Laboratory	36		
49	SB-SO-56-XX	Reference Laboratory	9		
49	SB-SO-04-MX	Oxford Instrument Analytical X-Met 3000TX	0		
49	SB-SO-34-MX	Oxford Instrument Analytical X-Met 3000TX	1		
49	SB-SO-42-MX	Oxford Instrument Analytical X-Met 3000TX	9		
50	SB-SO-04-XX	Reference Laboratory	40		
50	SB-SO-34-XX	Reference Laboratory	36		
50	SB-SO-49-XX	Reference Laboratory	36		
50	SB-SO-22-MX	Oxford Instrument Analytical X-Met 3000TX	50		
50	SB-SO-36-MX	Oxford Instrument Analytical X-Met 3000TX	70		
50	SB-SO-52-MX	Oxford Instrument Analytical X-Met 3000TX	57		

Appendix D2. Analytical Data Summary, Oxford X-Met 3000TX Revised Data Set (Submitted April 20, 2005) and Reference Laboratory Data Set (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se
54	LV-SO-03-XX	Reference Laboratory		2,000	
54	LV-SO-40-XX	Reference Laboratory		1,900	
54	LV-SO-49-XX	Reference Laboratory		2,000	
54	LV-SO-13-MX	Oxford Instrument Analytical X-Met 3000TX		2,376	
54	LV-SO-26-MX	Oxford Instrument Analytical X-Met 3000TX		2,319	
54	LV-SO-40-MX	Oxford Instrument Analytical X-Met 3000TX		2,584	
55	LV-SO-04-XX	Reference Laboratory		2,000	
55	LV-SO-34-XX	Reference Laboratory		1,900 J-	
55	LV-SO-37-XX	Reference Laboratory		1,400	
55	LV-SO-09-MX	Oxford Instrument Analytical X-Met 3000TX		2,763	
55	LV-SO-21-MX	Oxford Instrument Analytical X-Met 3000TX		2,605	
55	LV-SO-46-MX	Oxford Instrument Analytical X-Met 3000TX		2,691	
58	LV-SE-06-XX	Reference Laboratory		360	
58	LV-SE-13-XX	Reference Laboratory		360	
58	LV-SE-41-XX	Reference Laboratory		320	
58	LV-SE-12-MX	Oxford Instrument Analytical X-Met 3000TX		356	
58	LV-SE-36-MX	Oxford Instrument Analytical X-Met 3000TX		345	
58	LV-SE-52-MX	Oxford Instrument Analytical X-Met 3000TX		356	
59	LV-SE-05-XX	Reference Laboratory		400	
59	LV-SE-20-XX	Reference Laboratory		660	
59	LV-SE-43-XX	Reference Laboratory		530	
59	LV-SE-14-MX	Oxford Instrument Analytical X-Met 3000TX		591	
59	LV-SE-33-MX	Oxford Instrument Analytical X-Met 3000TX		555	
59	LV-SE-38-MX	Oxford Instrument Analytical X-Met 3000TX		536	
60	LV-SE-15-XX	Reference Laboratory		230	
60	LV-SE-17-XX	Reference Laboratory		220	
60	LV-SE-51-XX	Reference Laboratory		200	
60	LV-SE-29-MX	Oxford Instrument Analytical X-Met 3000TX		98	
60	LV-SE-41-MX	Oxford Instrument Analytical X-Met 3000TX		114	
60	LV-SE-44-MX	Oxford Instrument Analytical X-Met 3000TX		145	

Notes:
All concentrations reported in milligrams per kilogram (mg/kg), or parts per million (ppm)

J	Estimated concentration
J+	Concentration is considered estimated and biased high
J-	Concentration is considered estimated and biased low
U	Analyte is not detected; the associated concentration value is the sample reporting limit

APPENDIX E

STATISTICAL DATA SUMMARIES

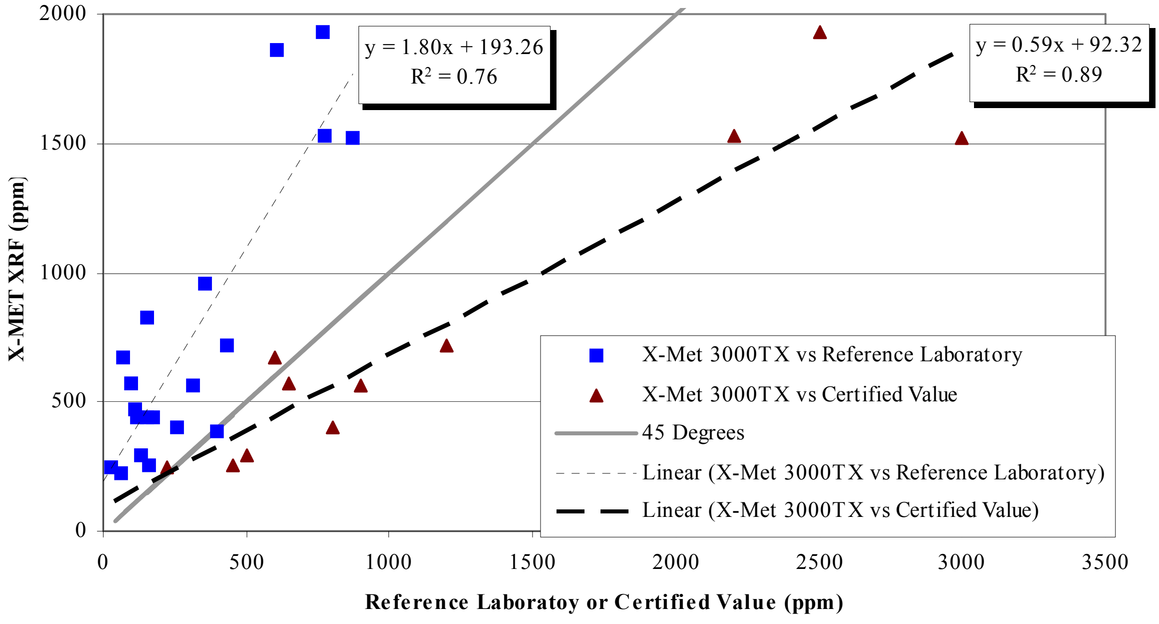


Figure E-1: Linear Correlation Plot for Antimony

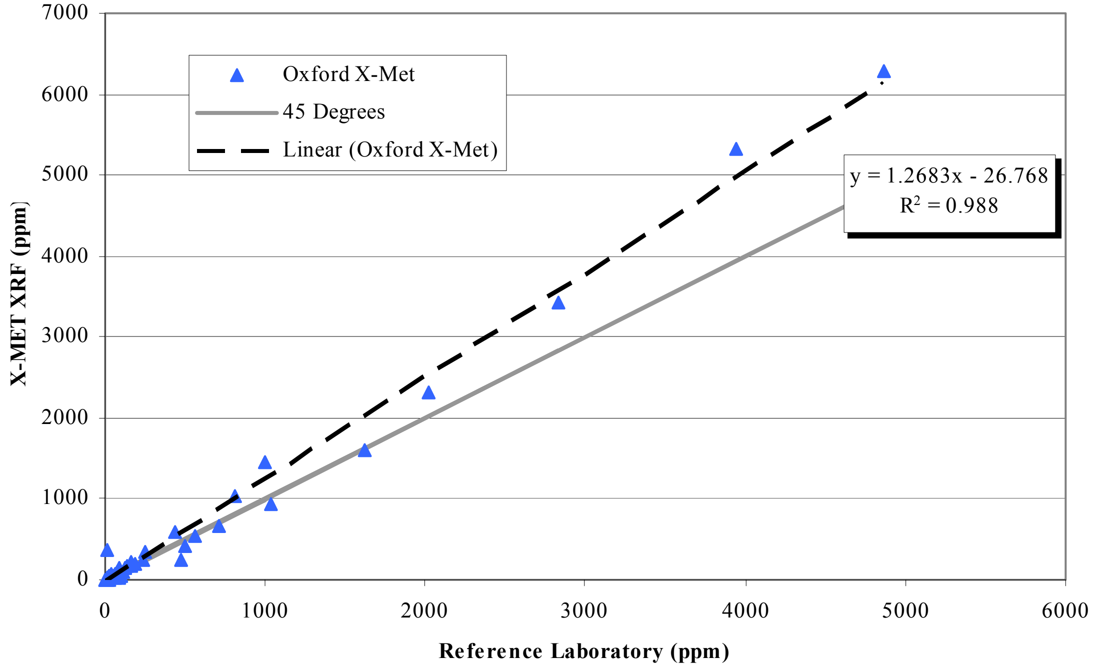


Figure E-2: Linear Correlation Plot for Arsenic

Figure E-3: Linear Correlation Plot for Cadmium

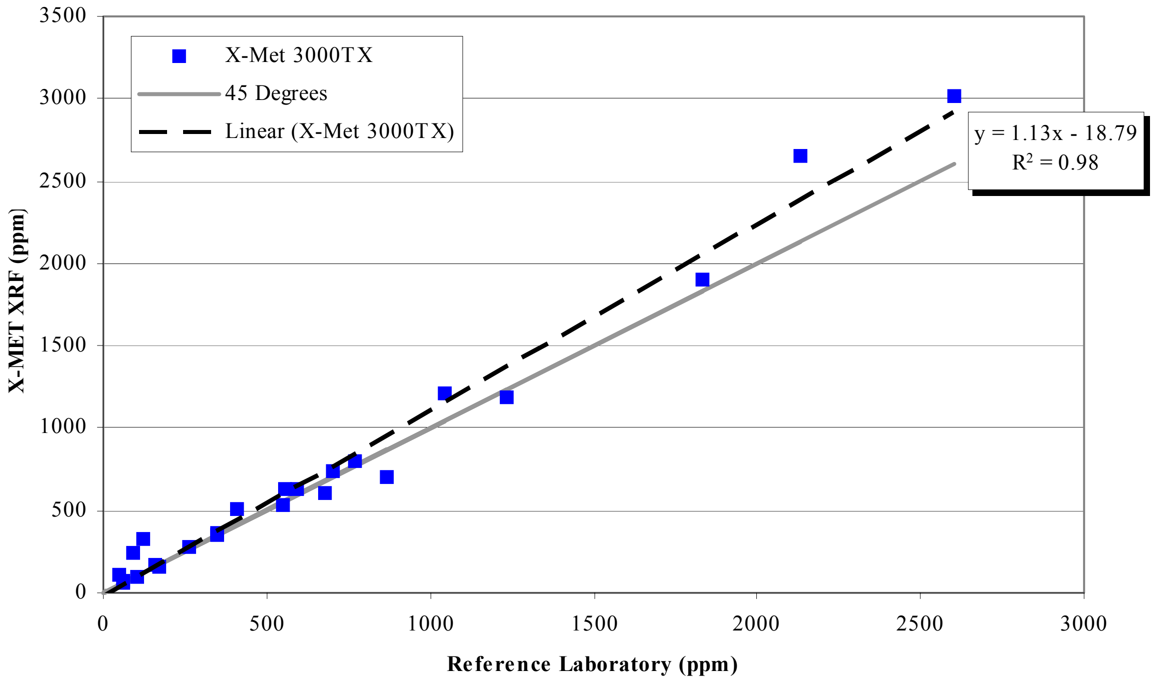


Figure E-4: Linear Correlation Plot for Chromium

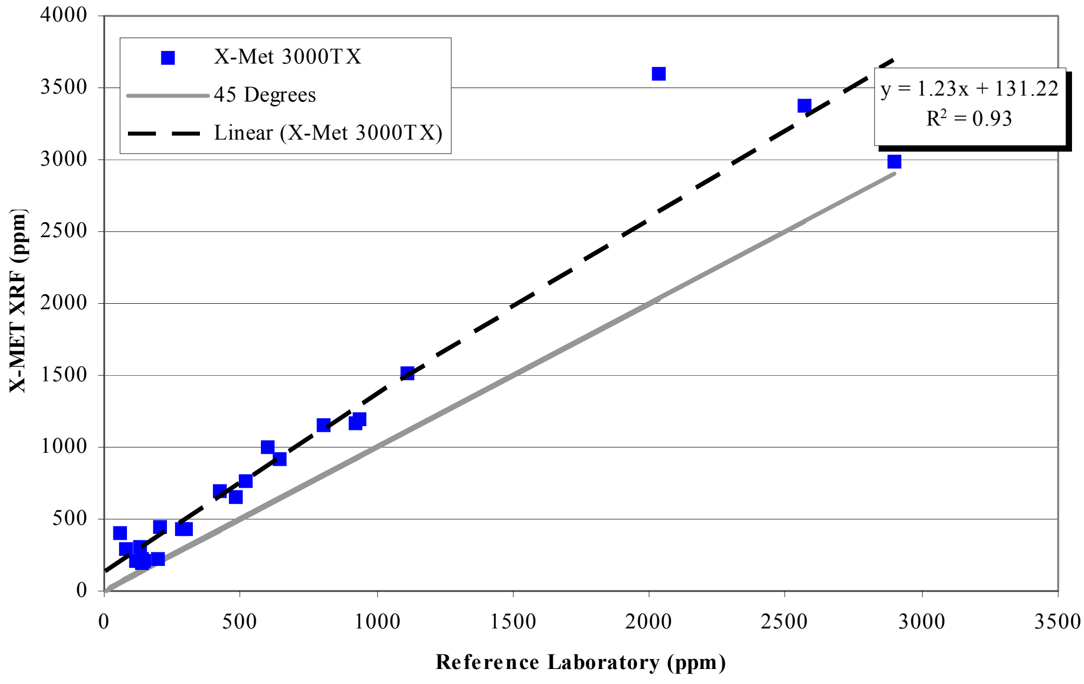


Figure E-5: Linear Correlation Plot for Copper

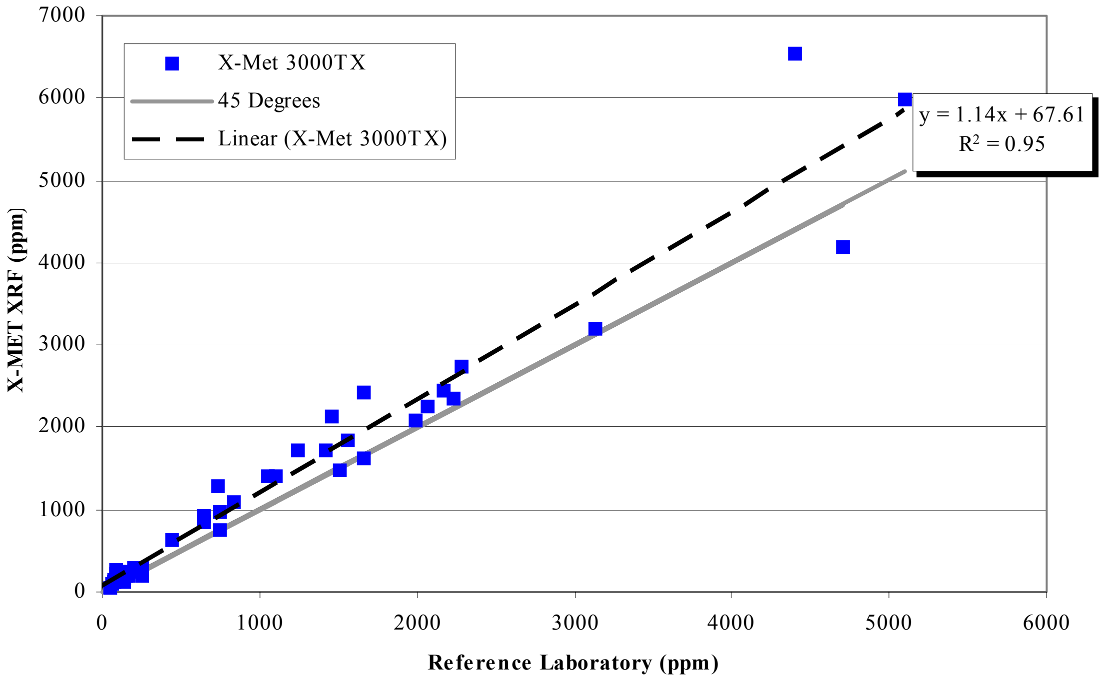


Figure E-6: Linear Correlation Plot for Iron

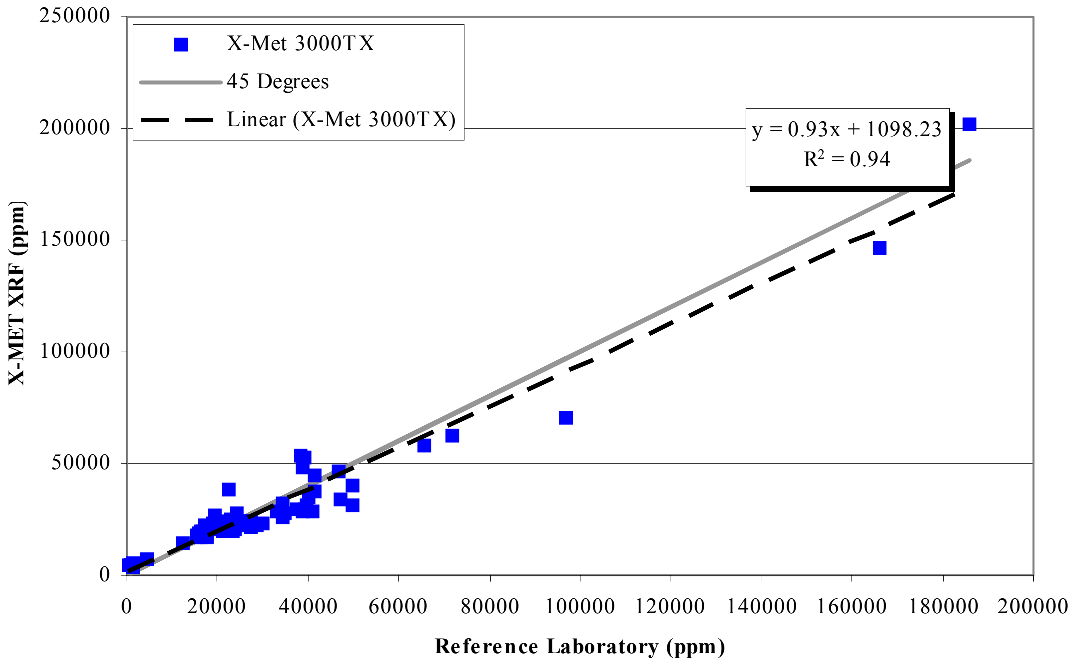


Figure E-7: Linear Correlation Plot for Lead

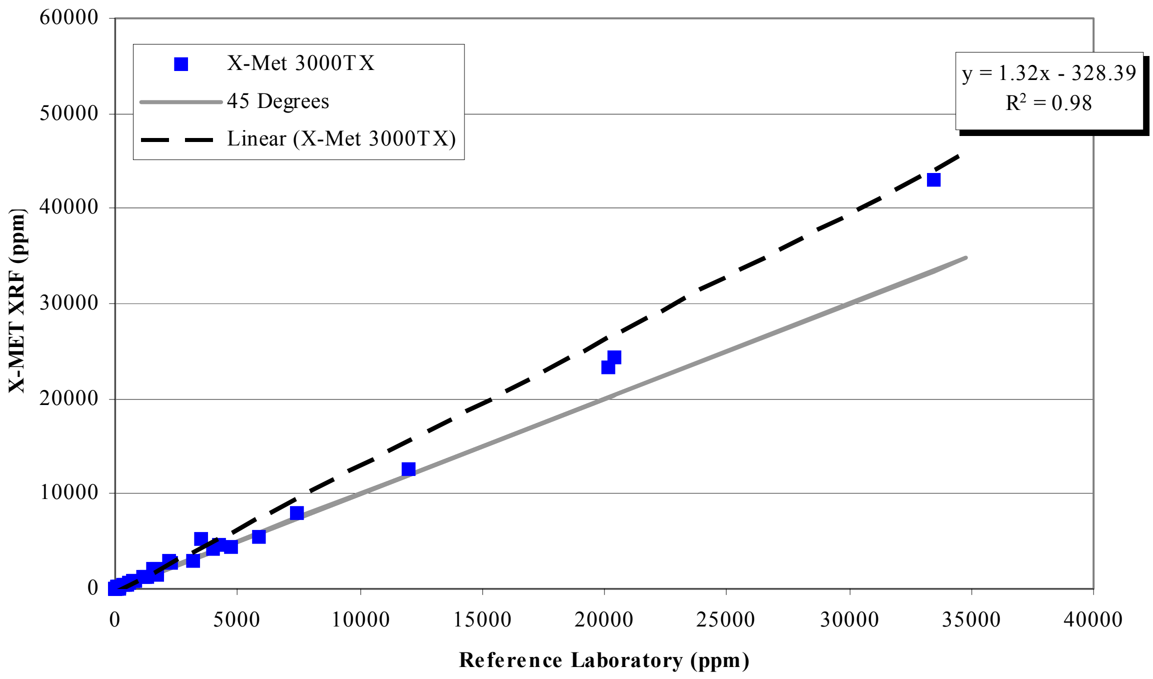


Figure E-8: Linear Correlation Plot for Mercury

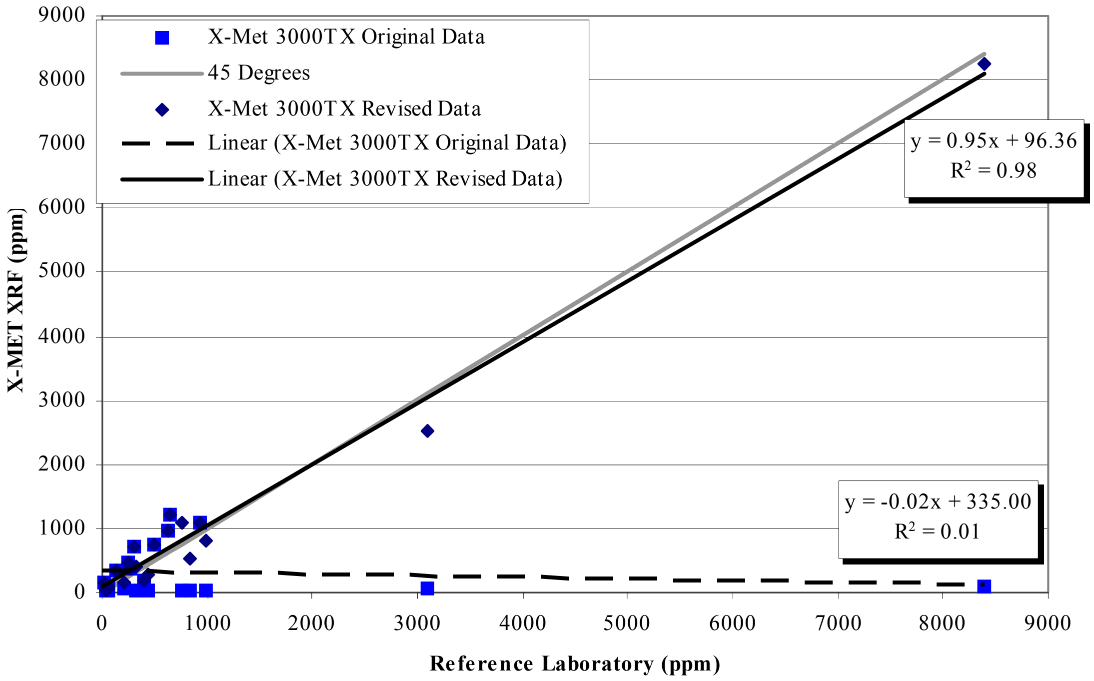


Figure E-9: Linear Correlation Plot for Nickel

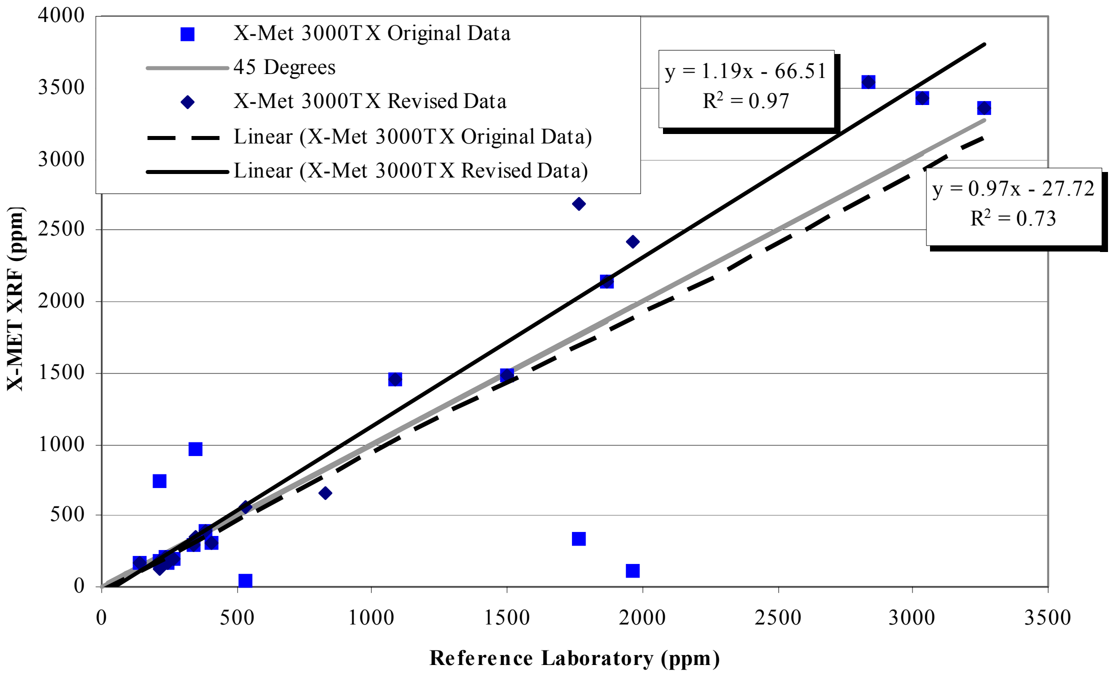


Figure E-10: Linear Correlation Plot for Selenium

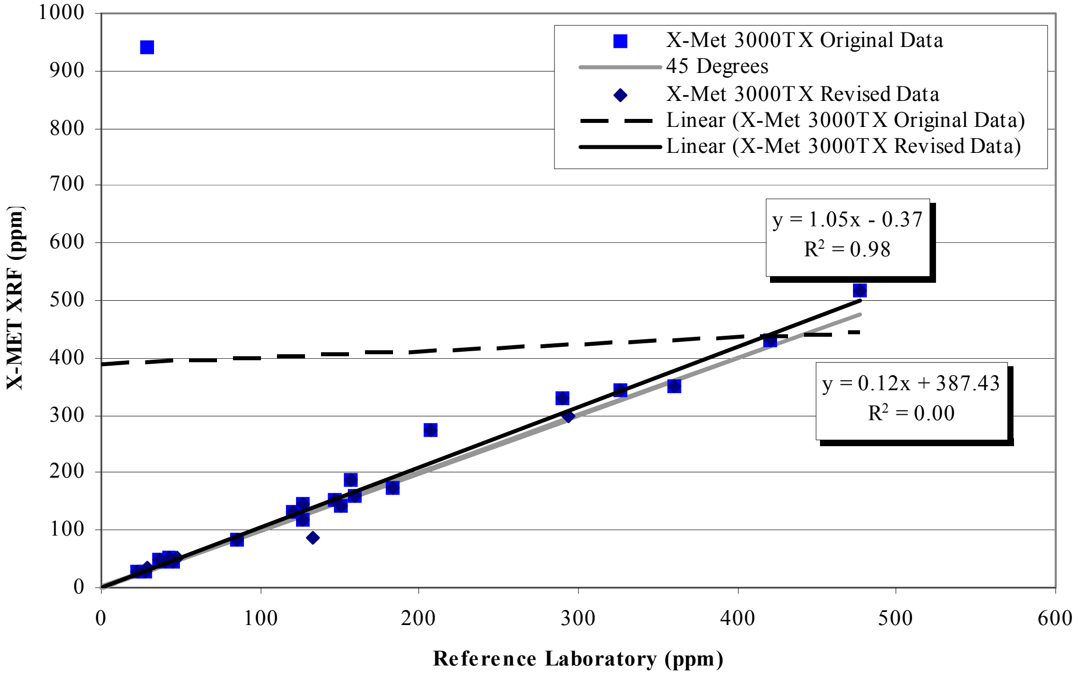


Figure E-11: Linear Correlation Plot for Silver

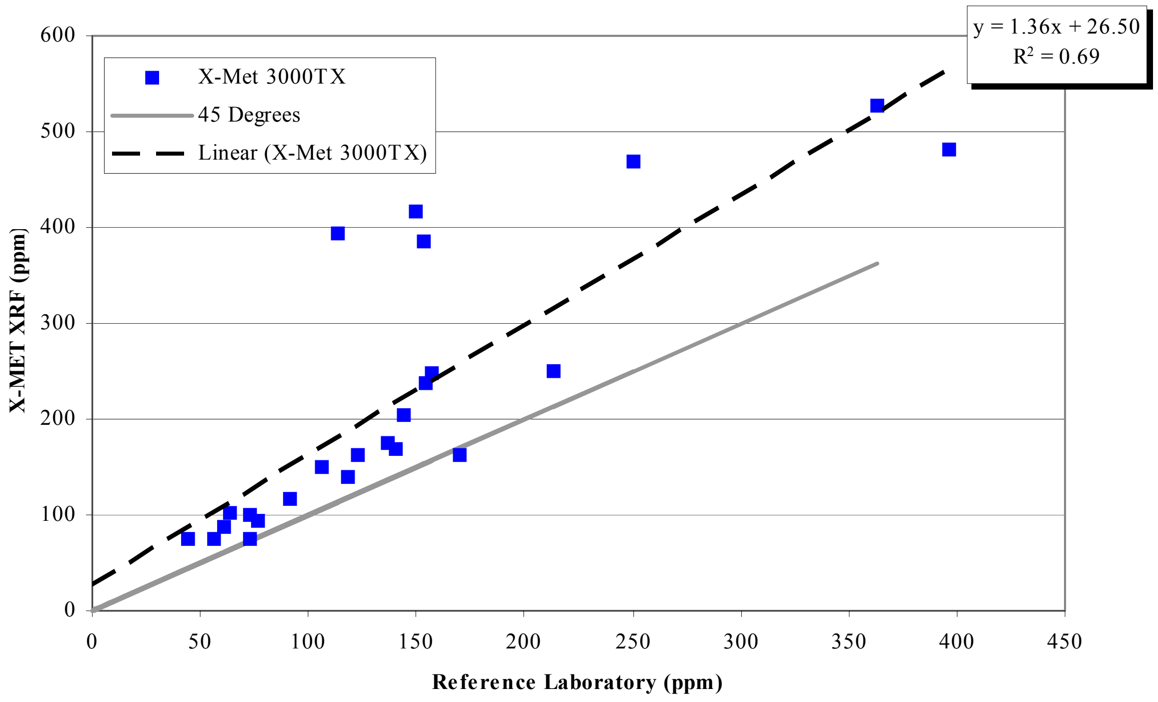


Figure E-12: Linear Correlation Plot for Vanadium

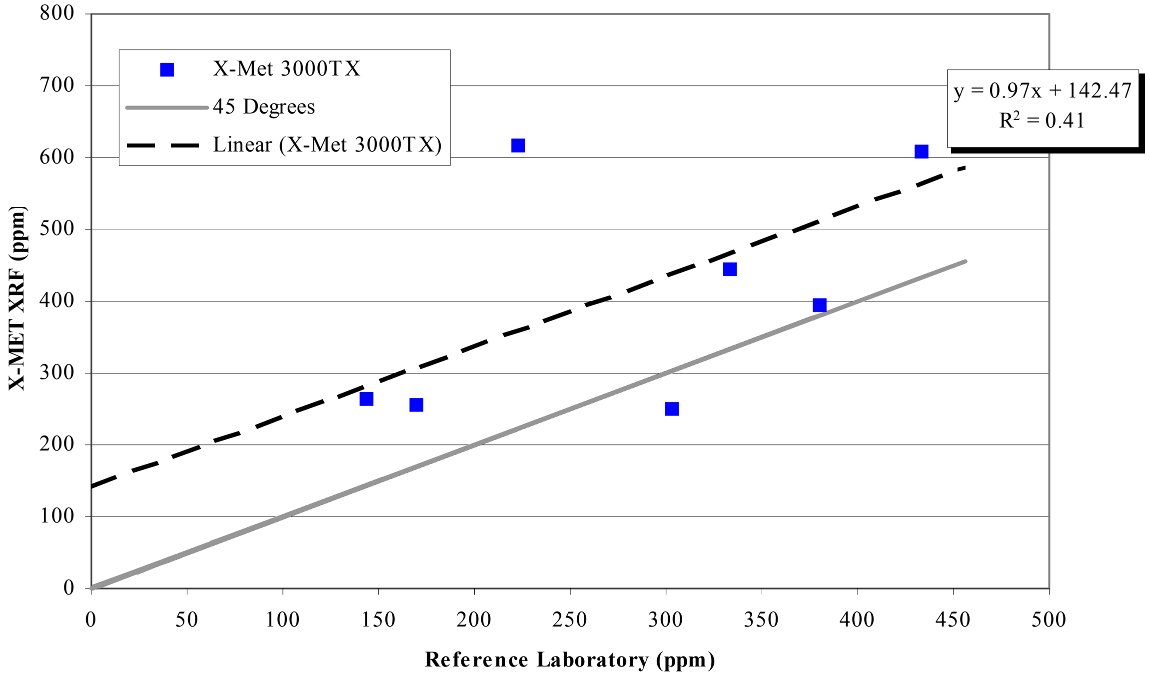
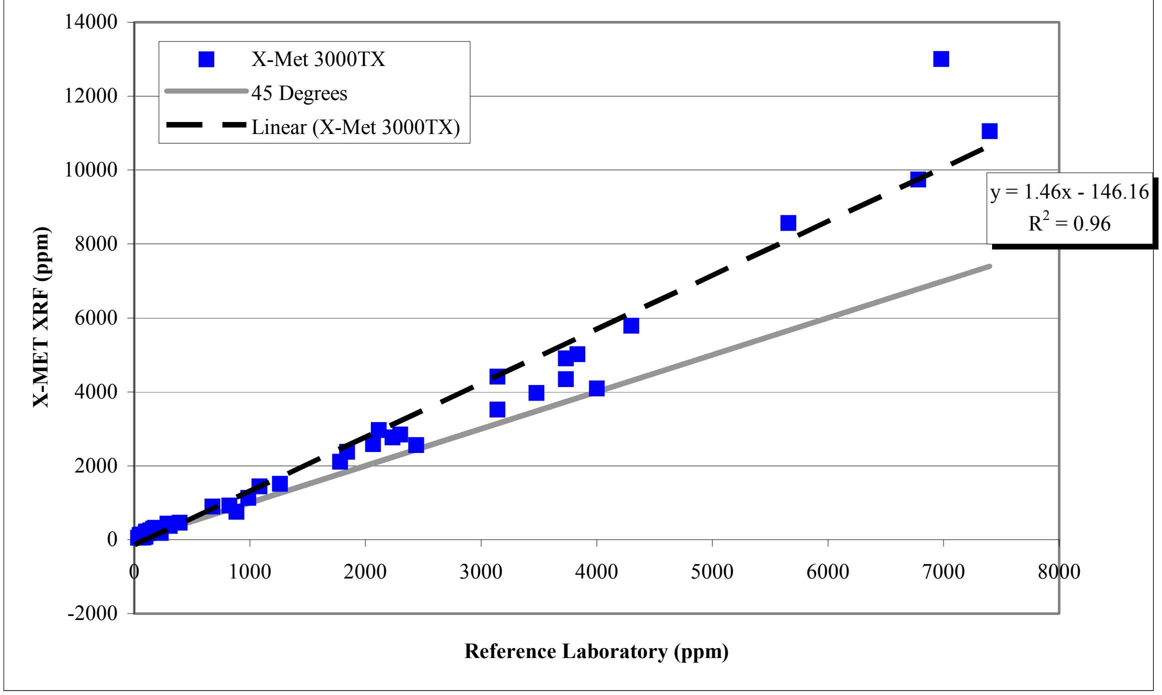
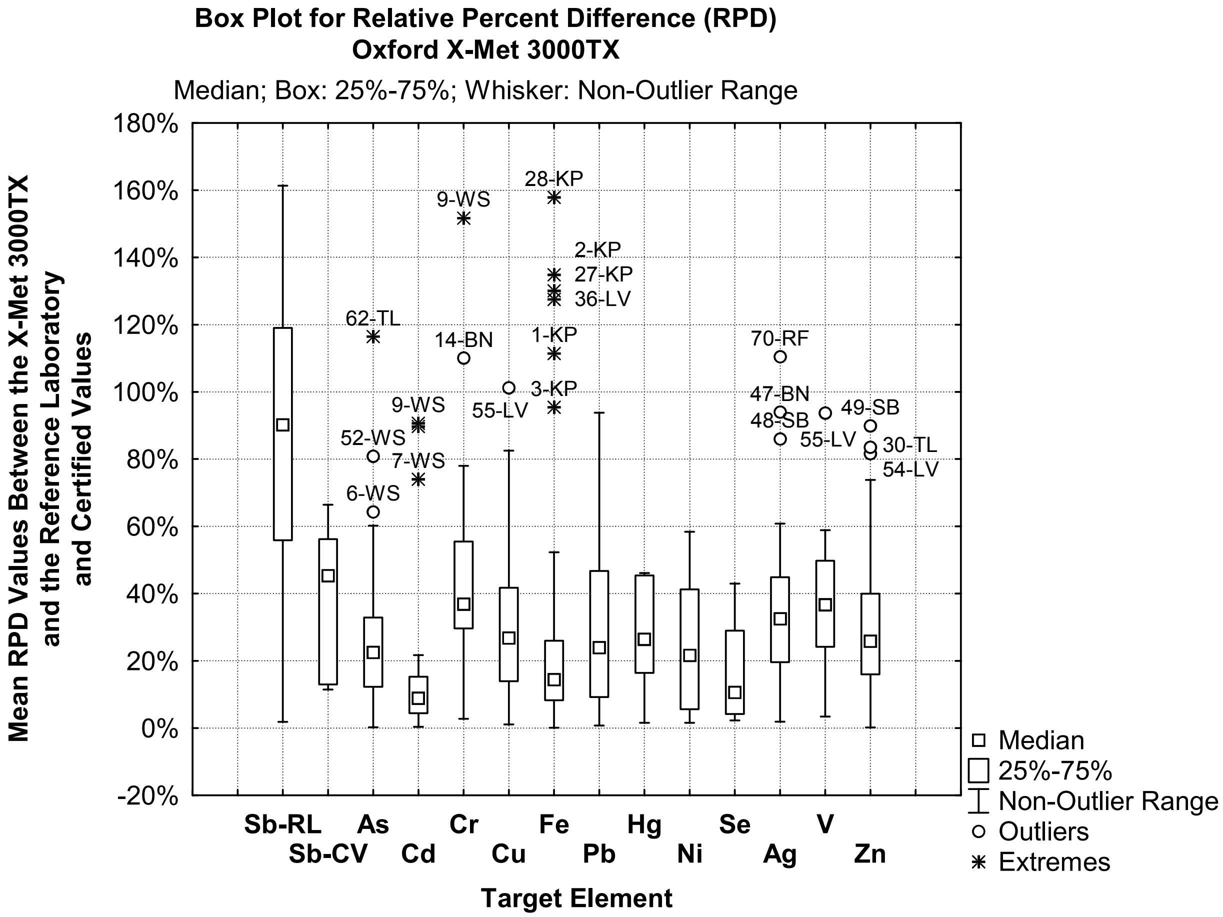


Figure E-13: Linear Correlation Plot for Zinc





Notes:

The “box” in each box plot presents the range of RPD values that lie between the 25th and 75th percentiles (that is, the “quartiles”) of the full RPD population for each element. In essence, the box displays the “interquartile range” of RPD values. The square data point within each box represents the median RPD for the population. The “whiskers” emanating from the top and bottom of each box represent the largest and smallest data points, respectively, that are within 1.5 times the interquartile range. Values outside the whiskers are identified as outliers and extremes.

Some of the more significant extremes and outliers are labeled with the associated Blend numbers and sample site abbreviations (see the footnotes of Table E-5 for definitions). Also refer to Appendix D for the sampling site and analytical data associated with each Blend number.

For antimony data:

RL	Reference Laboratory
CV	Certified Value

Figure E-14. Box and whisker plot for mean RPD values showing outliers and extremes for target elements, Oxford X-Met 3000TX

Table E-1. Evaluation of Sensitivity Method Detection Limits Calculated for the Oxford X-Met 3000TX Using the Original Data Set Submitted at the End of the Field Demonstration on January 28, 2005

		Mercury			Nickel			Selenium		
Matrix	**Blend No.**	**MDL[2]**	**X-Met[3]**	**Ref. Lab[4]**	**MDL[2]**	**X-Met[3]**	**Ref. Lab[4]**	**MDL[2]**	**X-Met[3]**	**Ref. Lab[4]**
Soil	2	NC	ND	ND	**54**	85	83	**6**	4	ND
Soil	5	NC	ND	ND	NC	ND	60	**7**	3	ND
Soil	6	NC	ND	0.83	NC	ND	70	NC	ND	ND
Soil	8	NC	ND	15	NC	ND	57	NC	ND	ND
Soil	10	NC	ND	0.14	NC	ND	60	**13**	45	ND
Soil	12	NC	ND	1.8	NC	ND	91	**94**	533	15
Soil	18	**20**	39	56	NC	ND	213	**4**	3	ND
Sediment	29	NC	ND	0.24	NC	ND	72	**3**	3	ND
Sediment	31	**49**	54	ND	NC	ND	196	**8**	6	ND
Sediment	32	**25**	49	ND	**25**	49	174	**4**	10	4.6
Sediment	39	**43**	89	ND	**62**	82	202	**3**	9	ND
Sediment	65	**41**	91	32	**92**	181	214	**8**	26	22
Mean		**36**			**58**			**15**		

Notes:

1. Bolded cells show calculated MDLs
2. Detection limits and concentrations are milligrams per kilogram (mg/kg), or parts per million (ppm).
3. This column reports the mean concentration reported for this blend by the X-Met 3000TX.
4. This column reports the mean concentration reported for this blend by the Reference Laboratory.

MDL	Method Detection Limit
NC	MDL not calculated due to reference laboratory concentrations greater than 250 ppm or insufficient number of detected concentrations.
ND	One or more results for this blend were reported as "Not Detected." Blends with one or more ND result as reported by the XRF were not used for calculating the MDL for this element.

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Revised Data Set for the Oxford X-Met 3000TX

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
			Ref Lab	ERA Spike									
Soil	Level 1	Number	4	--	11	6	10	16	4	14	5	1	4
		Minimum	1.8%	--	5.1%	2.2%	10.9%	13.0%	39.8%	1.3%	11.2%	15.3%	2.0%
		Maximum	115.1%	--	80.9%	90.6%	151.7%	101.2%	130.1%	93.8%	88.2%	15.3%	29.5%
		Mean	81.2%	--	30.0%	45.8%	60.3%	42.2%	94.2%	44.5%	48.2%	15.3%	13.2%
		Median	104.0%	--	27.0%	42.5%	48.1%	38.5%	103.4%	44.7%	45.4%	15.3%	10.6%
	Level 2	Number	5	1	4	7	4	8	13	4	7	3	5
		Minimum	44.9%	56.2%	1.1%	4.3%	34.1%	1.1%	0.9%	5.9%	16.4%	14.8%	0.8%
		Maximum	137.3%	56.2%	64.3%	21.7%	55.5%	54.2%	33.8%	24.8%	46.1%	27.7%	43.0%
		Mean	97.1%	56.2%	20.0%	12.0%	43.7%	27.9%	11.7%	11.7%	32.3%	23.2%	12.7%
		Median	92.1%	56.2%	7.3%	12.0%	42.7%	28.6%	12.0%	8.0%	31.8%	27.2%	5.4%
	Level 3	Number	4	3	4	2	2	2	13	8	2	6	4
		Minimum	54.1%	25.8%	13.9%	6.3%	2.8%	15.8%	7.0%	3.6%	1.6%	1.6%	2.3%
		Maximum	101.6%	65.4%	29.6%	14.9%	27.3%	39.3%	28.8%	38.7%	20.5%	41.3%	28.4%
		Mean	76.8%	42.4%	22.0%	10.6%	15.0%	27.5%	21.6%	13.4%	11.1%	16.8%	9.7%
		Median	75.8%	35.9%	22.3%	10.6%	15.0%	27.5%	24.0%	8.1%	11.1%	16.6%	4.1%
	Level 4	Number	--	--	--	--	--	--	7	5	--	--	--
		Minimum	--	--	--	--	--	--	0.1%	4.9%	--	--	--
		Maximum	--	--	--	--	--	--	47.1%	39.5%	--	--	--
		Mean	--	--	--	--	--	--	25.2%	20.2%	--	--	--
		Median	--	--	--	--	--	--	31.1%	17.3%	--	--	--
	All Soil	Number	13	4	19	15	16	26	37	31	14	10	13
		Minimum	1.8%	25.8%	1.1%	2.2%	2.8%	1.1%	0.1%	1.3%	1.6%	1.6%	0.8%
		Maximum	137.3%	65.4%	80.9%	90.6%	151.7%	101.2%	130.1%	93.8%	88.2%	41.3%	43.0%
		Mean	86.0%	45.8%	26.2%	25.3%	50.5%	36.7%	26.7%	28.3%	34.9%	18.6%	11.9%
		Median	92.1%	46.0%	20.3%	12.0%	43.9%	31.9%	21.9%	18.8%	30.9%	18.1%	5.6%

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Revised Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Silver	Vanadium	Zinc
Soil	Level 1	Number	3	0	18
		Minimum	1.9%	NC	1.1%
		Maximum	31.4%	NC	89.9%
		Mean	17.7%	NC	28.0%
		Median	19.8%	NC	22.4%
	Level 2	Number	3	0	6
		Minimum	16.6%	NC	11.5%
		Maximum	24.6%	NC	33.7%
		Mean	21.7%	NC	22.6%
		Median	24.0%	NC	21.8%
	Level 3	Number	7	3	9
		Minimum	16.2%	19.6%	2.2%
		Maximum	93.9%	93.7%	60.3%
		Mean	46.9%	47.3%	30.8%
		Median	36.7%	28.7%	29.4%
	Level 4	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Soil	Number	13	3	33
		Minimum	1.9%	19.6%	1.1%
		Maximum	93.9%	93.7%	89.9%
		Mean	34.4%	47.3%	27.8%
		Median	24.6%	28.7%	25.2%

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Revised Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
			Ref Lab	ERA Spike									
Sediment	Level 1	Number	1	1	14	3	1	8	3	14	2	0	5
		Minimum	155.5%	13.0%	0.2%	7.5%	78.0%	13.8%	127.4%	11.8%	95.5%	NC	4.4%
		Maximum	155.5%	13.0%	116.4%	15.0%	78.0%	66.7%	157.9%	89.3%	135.9%	NC	20.4%
		Mean	155.5%	13.0%	27.5%	10.5%	78.0%	40.3%	140.0%	51.3%	115.7%	NC	10.5%
		Median	155.5%	13.0%	20.1%	8.9%	78.0%	38.5%	134.8%	44.9%	115.7%	NC	6.7%
	Level 2	Number	3	3	4	4	3	4	19	4	4	6	4
		Minimum	76.2%	11.5%	8.2%	0.3%	30.3%	24.1%	0.1%	0.7%	41.7%	1.6%	4.7%
		Maximum	161.3%	52.7%	34.9%	21.7%	37.4%	29.2%	52.3%	15.5%	79.6%	58.4%	18.3%
		Mean	126.2%	25.7%	22.8%	9.6%	33.9%	26.9%	10.8%	7.8%	62.4%	21.1%	10.9%
		Median	141.2%	13.0%	24.2%	8.2%	34.1%	27.1%	8.7%	7.5%	64.2%	15.3%	10.4%
	Level 3	Number	3	3	2	3	3	10	4	3	3	4	3
		Minimum	42.7%	45.3%	23.6%	3.8%	23.5%	1.7%	5.9%	11.0%	16.9%	5.6%	2.1%
		Maximum	57.7%	66.4%	36.5%	4.3%	30.9%	36.8%	33.8%	31.6%	61.7%	29.5%	13.0%
		Mean	50.1%	53.9%	30.1%	3.9%	26.3%	10.1%	21.2%	19.9%	40.6%	17.7%	7.9%
		Median	49.9%	49.8%	30.1%	3.8%	24.5%	6.5%	22.5%	17.2%	43.1%	17.9%	8.5%
	Level 4	Number	--	--	--	--	--	--	6	--	--	--	--
		Minimum	--	--	--	--	--	--	8.3%	--	--	--	--
		Maximum	--	--	--	--	--	--	14.4%	--	--	--	--
		Mean	--	--	--	--	--	--	11.9%	--	--	--	--
		Median	--	--	--	--	--	--	12.6%	--	--	--	--
	All Sediment	Number	7	7	20	10	7	22	32	21	9	10	12
		Minimum	42.7%	11.5%	0.2%	0.3%	23.5%	1.7%	0.1%	0.7%	16.9%	1.6%	2.1%
		Maximum	161.3%	66.4%	116.4%	21.7%	78.0%	66.7%	157.9%	89.3%	135.9%	58.4%	20.4%
		Mean	97.8%	36.0%	26.8%	8.2%	37.0%	24.2%	24.4%	38.6%	67.0%	19.8%	10.0%
		Median	76.2%	45.3%	23.1%	6.1%	30.9%	24.8%	10.2%	31.6%	63.4%	15.3%	7.9%

Table E-2. Evaluation of Accuracy Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Revised Data Set for the Oxford X-Met 3000TX

Matrix	Conc Range	Statistic	Silver	Vanadium	Zinc
Sediment	Level 1	Number	4	0	19
		Minimum	27.8%	NC	0.2%
		Maximum	51.2%	NC	83.5%
		Mean	39.8%	NC	43.0%
		Median	40.1%	NC	40.0%
	Level 2	Number	4	2	5
		Minimum	4.4%	40.7%	11.7%
		Maximum	33.5%	58.9%	28.6%
		Mean	21.1%	49.8%	20.0%
		Median	23.2%	49.8%	18.3%
	Level 3	Number	3	3	4
		Minimum	44.9%	3.4%	4.9%
		Maximum	110.4%	39.5%	25.4%
		Mean	72.0%	25.6%	17.1%
		Median	60.8%	33.8%	19.1%
	Level 4	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Sediment	Number	11	5	28
		Minimum	4.4%	3.4%	0.2%
		Maximum	110.4%	58.9%	83.5%
		Mean	41.8%	35.2%	35.2%
		Median	35.4%	39.5%	28.3%

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Revised Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
			Ref Lab	ERA Spike									
All Samples	X-Met 3000TX	Number	20	11	39	25	23	48	69	52	23	20	25
		Minimum	1.8%	11.5%	0.2%	0.3%	2.8%	1.1%	0.1%	0.7%	1.6%	1.6%	0.8%
		Maximum	161.3%	66.4%	116.4%	90.6%	151.7%	101.2%	157.9%	93.8%	135.9%	58.4%	43.0%
		Mean	90.1%	39.5%	26.5%	18.5%	46.4%	30.9%	25.6%	32.5%	47.5%	19.2%	11.0%
		Median	90.1%	45.3%	22.5%	8.9%	36.8%	26.8%	14.4%	23.9%	43.1%	16.0%	6.7%
All Samples	All Instruments	Number	206	110	320	209	338	363	558	392	192	403	195
		Minimum	0.1%	0.1%	0.2%	0.1%	0.1%	0.2%	0.0%	0.1%	0.0%	0.3%	0.0%
		Maximum	181.5%	162.0%	182.8%	168.1%	151.7%	111.1%	190.1%	135.2%	158.1%	146.5%	127.1%
		Mean	80.6%	62.7%	36.6%	29.6%	30.8%	24.6%	35.4%	30.9%	62.5%	31.0%	32.0%
		Median	84.3%	70.6%	26.2%	16.7%	26.0%	16.2%	26.0%	21.5%	58.6%	25.4%	16.7%

Matrix	Conc Range	Statistic	Silver	Vanadium	Zinc
All Samples	X-Met 3000TX	Number	24	8	61
		Minimum	1.9%	3.4%	0.2%
		Maximum	110.4%	93.7%	89.9%
		Mean	37.8%	39.8%	31.2%
		Median	32.4%	36.6%	25.8%
All Samples	All Instruments	Number	177	218	471
		Minimum	0.0%	0.1%	0.0%
		Maximum	129.7%	129.5%	138.0%
		Mean	36.0%	42.2%	26.3%
		Median	28.7%	38.3%	19.4%

Notes:

All RPDs presented in this table are absolute values.

--	No samples reported by the reference laboratory in this concentration range.
Conc	Concentration.
ERA	Environmental Resource Associates, Inc.
NC	Not calculated because of a lack of XRF data.
Number	Number of demonstration samples evaluated.
Ref Lab	Reference laboratory (Shealy Environmental Services, Inc.)
RPD	Relative percent difference.

Table E-3. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Original Data Set for the Oxford X-Met 3000TX

Matrix	Conc Range	Statistic	Mercury	Nickel	Selenium
Soil	Level 1	Number	5	1	4
		Minimum	22.4%	15.3%	2.0%
		Maximum	88.2%	15.3%	188.3%
		Mean	51.2%	15.3%	102.0%
		Median	36.1%	15.3%	108.8%
	Level 2	Number	7	3	5
		Minimum	30.1%	14.8%	0.8%
		Maximum	184.6%	27.7%	185.0%
		Mean	147.7%	23.2%	41.1%
		Median	167.6%	27.2%	5.4%
	Level 3	Number	2	6	3
		Minimum	193.5%	1.6%	2.6%
		Maximum	195.3%	179.7%	28.4%
		Mean	194.4%	59.1%	12.2%
		Median	194.4%	17.3%	5.6%
	Level 4	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Soil	Number	14	10	12
		Minimum	22.4%	1.6%	0.8%
		Maximum	195.3%	179.7%	188.3%
		Mean	119.9%	44.0%	54.2%
		Median	143.4%	18.8%	7.4%

Table E-3. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Original Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Mercury	Nickel	Selenium
Sediment	Level 1	Number	2	0	5
		Minimum	95.5%	NC	4.4%
		Maximum	135.9%	NC	20.4%
		Mean	115.7%	NC	10.5%
		Median	115.7%	NC	6.7%
	Level 2	Number	7	6	4
		Minimum	30.1%	2.0%	4.7%
		Maximum	184.6%	109.7%	18.3%
		Mean	147.7%	45.0%	10.9%
		Median	167.6%	25.3%	10.4%
	Level 3	Number	3	3	3
		Minimum	16.9%	13.4%	2.1%
		Maximum	61.7%	170.8%	13.0%
		Mean	40.6%	71.2%	7.9%
		Median	43.1%	29.5%	8.5%
	Level 4	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Sediment	Number	9	9	12
		Minimum	16.9%	2.0%	2.1%
		Maximum	135.9%	170.8%	20.4%
		Mean	67.0%	53.8%	10.0%
		Median	63.4%	29.5%	7.9%

Table E-3. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated Using the Original Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Mercury	Nickel	Selenium
All	All	Number	23	19	24
		Minimum	16.9%	1.6%	0.8%
		Maximum	195.3%	179.7%	188.3%
		Mean	99.2%	48.6%	32.1%
		Median	79.6%	22.3%	7.9%
All Samples	All Instruments	Number	192	403	195
		Minimum	0.0%	0.3%	0.0%
		Maximum	158.1%	146.5%	127.1%
		Mean	62.5%	31.0%	32.0%
		Median	58.6%	25.4%	16.7%

Notes:

All RPDs presented in this table are absolute values.

--	No samples reported by the reference laboratory in this concentration range.
Conc	Concentration.
ERA	Environmental Resource Associates, Inc.
Number	Number of demonstration samples evaluated.
Ref	Reference laboratory (Shealy Environmental Services, Inc.).
RPD	Relative percent difference.
XRF	X-ray fluorescence

Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated Using the Revised Data Set for the Oxford X-Met 3000TX

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
Soil	Low	Number	4	11	6	10	16	4	14	5	1	4
		Minimum	13.5%	2.3%	4.6%	4.1%	1.7%	1.1%	3.6%	5.7%	15.7%	6.6%
		Maximum	23.3%	18.8%	30.5%	26.1%	11.7%	2.1%	21.4%	17.7%	15.7%	10.7%
		Mean	18.3%	7.8%	12.7%	14.9%	7.4%	1.7%	9.9%	10.7%	15.7%	8.6%
		Median	18.2%	8.2%	8.7%	12.9%	7.9%	1.7%	9.5%	9.8%	15.7%	8.6%
	Medium	Number	5	4	7	4	8	13	4	7	3	5
		Minimum	5.7%	3.5%	0.4%	4.7%	2.4%	0.7%	3.2%	1.1%	9.2%	0.9%
		Maximum	18.4%	9.8%	5.6%	11.8%	12.6%	8.0%	6.4%	7.5%	14.4%	4.4%
		Mean	11.0%	5.9%	2.6%	8.0%	6.1%	3.0%	5.0%	3.5%	11.0%	2.0%
		Median	10.2%	5.2%	2.2%	7.9%	6.0%	2.7%	5.2%	3.8%	9.4%	1.2%
	High	Number	4	4	2	2	2	13	8	2	7	4
		Minimum	6.8%	0.4%	1.1%	1.3%	5.1%	0.7%	1.6%	2.7%	0.9%	0.8%
		Maximum	22.8%	1.6%	4.9%	4.7%	7.4%	3.9%	8.5%	3.4%	5.9%	1.8%
		Mean	12.9%	1.0%	3.0%	3.0%	6.3%	1.9%	3.8%	3.1%	3.4%	1.4%
		Median	10.9%	1.1%	3.0%	3.0%	6.3%	1.6%	3.2%	3.1%	3.0%	1.6%
	Very High	Number	--	--	--	--	--	7	5	--	--	--
		Minimum	--	--	--	--	--	1.2%	1.2%	--	--	--
		Maximum	--	--	--	--	--	4.7%	7.5%	--	--	--
		Mean	--	--	--	--	--	2.4%	3.7%	--	--	--
		Median	--	--	--	--	--	1.7%	3.6%	--	--	--
	All Soil	Number	13	19	15	16	26	37	31	14	11	13
		Minimum	5.7%	0.4%	0.4%	1.3%	1.7%	0.7%	1.2%	1.1%	0.9%	0.8%
		Maximum	23.3%	18.8%	30.5%	26.1%	12.6%	8.0%	21.4%	17.7%	15.7%	10.7%
		Mean	13.8%	6.0%	6.7%	11.7%	6.9%	2.4%	6.7%	6.0%	6.6%	3.9%
		Median	12.2%	5.5%	4.6%	10.9%	7.1%	1.9%	5.3%	4.1%	5.7%	1.8%

Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated Using the Revised Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Silver	Vanadium	Zinc
Soil	Low	Number	3	0	18
		Minimum	11.2%	NC	1.0%
		Maximum	13.9%	NC	28.3%
		Mean	12.5%	NC	10.6%
		Median	12.3%	NC	8.4%
	Medium	Number	3	0	6
		Minimum	1.8%	NC	1.6%
		Maximum	12.1%	NC	4.7%
		Mean	7.9%	NC	3.4%
		Median	9.7%	NC	3.3%
	High	Number	7	3	9
		Minimum	1.8%	11.8%	1.5%
		Maximum	9.8%	25.1%	9.5%
		Mean	6.1%	18.9%	5.2%
		Median	5.7%	19.8%	4.7%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Soil	Number	13	3	33
		Minimum	1.8%	11.8%	1.0%
		Maximum	13.9%	25.1%	28.3%
		Mean	8.0%	18.9%	7.8%
		Median	9.4%	19.8%	6.6%

Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated Using the Revised Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
Sediment	Low	Number	1	14	3	1	8	3	14	2	0	5
		Minimum	4.8%	3.3%	4.8%	28.0%	4.3%	0.7%	1.6%	3.1%	NC	1.3%
		Maximum	4.8%	25.0%	20.3%	28.0%	12.5%	2.3%	20.1%	14.4%	NC	10.1%
		Mean	4.8%	10.4%	10.1%	28.0%	8.7%	1.4%	10.3%	8.7%	NC	5.6%
		Median	4.8%	9.1%	5.2%	28.0%	9.3%	1.1%	10.8%	8.7%	NC	5.6%
	Medium	Number	3	4	4	3	4	19	4	4	6	4
		Minimum	6.7%	2.2%	0.4%	3.9%	6.0%	0.9%	2.1%	2.6%	1.8%	2.2%
		Maximum	11.7%	7.4%	7.2%	16.4%	8.8%	6.8%	7.2%	6.2%	20.1%	5.7%
		Mean	9.8%	4.0%	4.0%	8.4%	7.3%	2.8%	3.7%	3.8%	12.2%	4.1%
		Median	11.0%	3.2%	4.2%	4.9%	7.3%	2.4%	2.7%	3.2%	12.3%	4.2%
	High	Number	3	2	3	3	10	4	3	3	3	3
		Minimum	4.9%	3.8%	0.8%	4.5%	1.2%	1.1%	0.4%	1.0%	0.8%	2.3%
		Maximum	15.4%	5.6%	1.7%	13.5%	7.3%	4.4%	3.5%	5.7%	5.0%	5.4%
		Mean	9.2%	4.7%	1.4%	7.8%	3.0%	2.8%	1.5%	3.3%	3.3%	3.6%
		Median	7.2%	4.7%	1.5%	5.5%	2.7%	2.9%	0.6%	3.2%	4.0%	3.2%
	Very High	Number	--	--	--	--	--	6	--	--	--	--
		Minimum	--	--	--	--	--	0.8%	--	--	--	--
		Maximum	--	--	--	--	--	2.0%	--	--	--	--
		Mean	--	--	--	--	--	1.4%	--	--	--	--
		Median	--	--	--	--	--	1.4%	--	--	--	--
	All Sediment	Number	7	20	10	7	22	32	21	9	9	12
		Minimum	4.8%	2.2%	0.4%	3.9%	1.2%	0.7%	0.4%	1.0%	0.8%	1.3%
		Maximum	15.4%	25.0%	20.3%	28.0%	12.5%	6.8%	20.1%	14.4%	20.1%	10.1%
		Mean	8.8%	8.6%	5.0%	11.0%	5.9%	2.4%	7.8%	4.7%	9.2%	4.6%
		Median	7.2%	7.5%	3.9%	5.5%	5.5%	1.9%	6.4%	3.2%	6.8%	4.9%

Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated Using the Revised Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Silver	Vanadium	Zinc
Sediment	Low	Number	4	0	19
		Minimum	10.2%	NC	0.8%
		Maximum	21.2%	NC	11.8%
		Mean	16.0%	NC	6.3%
		Median	16.3%	NC	6.0%
	Medium	Number	4	2	5
		Minimum	0.8%	8.6%	2.7%
		Maximum	10.6%	13.4%	10.4%
		Mean	6.2%	11.0%	6.4%
		Median	6.8%	11.0%	7.4%
	High	Number	3	3	4
		Minimum	2.8%	6.4%	3.8%
		Maximum	5.1%	14.6%	10.3%
		Mean	3.6%	10.1%	5.9%
		Median	3.0%	9.4%	4.7%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Sediment	Number	11	5	28
		Minimum	0.8%	6.4%	0.8%
		Maximum	21.2%	14.6%	11.8%
		Mean	9.1%	10.5%	6.2%
		Median	8.7%	9.4%	5.7%

Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated Using the Revised Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
All	All	Number	20	39	25	23	48	69	52	23	20	25
		Minimum	4.8%	0.4%	0.4%	1.3%	1.2%	0.7%	0.4%	1.0%	0.8%	0.8%
		Maximum	23.3%	25.0%	30.5%	28.0%	12.6%	8.0%	21.4%	17.7%	20.1%	10.7%
		Mean	12.1%	7.3%	6.0%	11.5%	6.4%	2.4%	7.1%	5.5%	7.8%	4.2%
		Median	11.4%	5.8%	4.6%	10.6%	6.4%	1.9%	5.7%	3.8%	5.8%	3.2%
All Samples	All Instruments	Number	206	320	209	338	363	558	392	192	403	195
		Minimum	0.5%	0.2%	0.4%	0.6%	0.1%	0.1%	0.2%	1.0%	0.3%	0.1%
		Maximum	97.7%	71.7%	92.8%	116.3%	58.3%	101.8%	115.6%	137.1%	164.2%	98.8%
		Mean	8.9%	11.2%	8.2%	15.9%	7.5%	5.2%	9.3%	14.3%	10.8%	7.2%
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%	7.0%	4.5%

Matrix	Conc Range	Statistic	Silver	Vanadium	Zinc
All	All	Number	24	8	61
		Minimum	0.8%	6.4%	0.8%
		Maximum	21.2%	25.1%	28.3%
		Mean	8.5%	13.7%	7.1%
		Median	9.1%	12.6%	6.0%
All Samples	All Instruments	Number	177	218	471
		Minimum	0.6%	0.4%	0.1%
		Maximum	125.3%	86.1%	192.9%
		Mean	10.3%	12.5%	8.0%
		Median	5.2%	8.5%	5.3%

Notes:

--	No samples reported by the reference laboratory in this concentration range.
Conc	Concentration.
NC	Not calculated because of a lack of XRF data.
Number	Number of demonstration samples evaluated.
RSD	Relative standard deviation.

Table E-5. Evaluation of Precision - Relative Standard Deviations Calculated Using the Original Data Set for the Oxford X-Met 3000TX

Matrix	Conc Range	Statistic	Mercury	Nickel	Selenium
Soil	Low	Number	5	1	4
		Minimum	5.7%	15.7%	3.5%
		Maximum	35.6%	15.7%	10.7%
		Mean	15.5%	15.7%	6.4%
		Median	11.3%	15.7%	5.7%
	Medium	Number	7	3	5
		Minimum	1.1%	9.2%	0.4%
		Maximum	21.4%	14.4%	4.4%
		Mean	15.1%	11.0%	1.9%
		Median	18.7%	9.4%	1.2%
	High	Number	2	6	3
		Minimum	6.0%	0.9%	1.5%
		Maximum	9.9%	11.3%	1.8%
		Mean	8.0%	4.7%	1.7%
		Median	8.0%	4.2%	1.7%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Soil	Number	14	10	12
		Minimum	1.1%	0.9%	0.4%
		Maximum	35.6%	15.7%	10.7%
		Mean	14.2%	7.7%	3.3%
		Median	13.7%	7.5%	2.0%

Table E-5. Evaluation of Precision - Relative Standard Deviations Calculated Using the Original Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Mercury	Nickel	Selenium
Sediment	Low	Number	2	0	5
		Minimum	3.1%	NC	1.3%
		Maximum	14.4%	NC	10.1%
		Mean	8.7%	NC	5.6%
		Median	8.7%	NC	5.6%
	Medium	Number	4	6	4
		Minimum	2.6%	1.0%	2.2%
		Maximum	6.2%	20.1%	5.7%
		Mean	3.8%	9.2%	4.1%
		Median	3.2%	7.6%	4.2%
	High	Number	3	3	3
		Minimum	1.0%	0.8%	2.3%
		Maximum	5.7%	6.7%	5.4%
		Mean	3.3%	3.9%	3.6%
		Median	3.2%	4.0%	3.2%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Sediment	Number	9	9	12
		Minimum	1.0%	0.8%	1.3%
		Maximum	14.4%	20.1%	10.1%
		Mean	4.7%	7.4%	4.6%
		Median	3.2%	6.7%	4.9%
			2	0	5

Table E-5. Evaluation of Precision - Relative Standard Deviations Calculated Using the Original Data Set for the Oxford X-Met 3000TX (Continued)

Matrix	Conc Range	Statistic	Mercury	Nickel	Selenium
All	All	Number	23	19	24
		Minimum	1.0%	0.8%	0.4%
		Maximum	35.6%	20.1%	10.7%
		Mean	10.5%	7.6%	4.0%
		Median	6.7%	6.7%	3.3%
All Samples	All Instruments	Number	192	403	195
		Minimum	1.0%	0.3%	0.1%
		Maximum	137.1%	164.2%	98.8%
		Mean	14.3%	10.8%	7.2%
		Median	6.8%	7.0%	4.5%

Notes:

--	No samples reported by the reference laboratory in this concentration range.
Conc	Concentration.
NC	Not calculated because of alack of XRF data.
Number	Number of demonstration samples evaluated.
RSD	Relative standard deviation.

Table E-6. Evaluation of Precision - Relative Standard Deviations Calculated for the Reference Laboratory

Matrix	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
All Soil	Number	17	23	15	34	26	38	33	16	35	13
	Minimum	3.6%	1.4%	0.9%	1.4%	0.0%	1.6%	0.0%	0.0%	0.0%	0.0%
	Maximum	38.0%	45.8%	21.4%	137.0%	21.0%	46.2%	150.0%	50.7%	44.9%	22.7%
	Mean	14.3%	11.7%	11.1%	14.3%	10.1%	10.2%	17.6%	13.8%	11.4%	8.9%
	Median	9.8%	12.4%	9.0%	10.6%	9.1%	8.7%	13.2%	6.6%	10.0%	7.1%
All Sediment	Number	7	24	10	26	21	31	22	10	27	12
	Minimum	2.9%	2.4%	2.9%	4.6%	1.8%	2.7%	0.0%	2.8%	0.6%	1.3%
	Maximum	33.6%	36.7%	37.5%	35.5%	38.8%	37.5%	41.1%	48.0%	35.8%	37.3%
	Mean	14.4%	10.7%	11.4%	9.8%	9.7%	9.9%	11.6%	14.3%	9.4%	10.0%
	Median	9.1%	9.2%	8.2%	7.5%	8.9%	8.1%	7.4%	6.9%	7.3%	7.6%
All	Number	24	47	25	60	47	69	55	26	62	25
	Minimum	2.9%	1.4%	0.9%	1.4%	0.0%	1.6%	0.0%	0.0%	0.0%	0.0%
	Maximum	38.0%	45.8%	37.5%	137.0%	38.8%	46.2%	150.0%	50.7%	44.9%	37.3%
	Mean	14.3%	11.2%	11.2%	12.4%	9.9%	10.1%	15.2%	14.0%	10.6%	9.4%
	Median	9.5%	9.5%	9.0%	8.4%	8.9%	8.5%	8.6%	6.6%	8.2%	7.4%

Table E-6. Evaluation of Precision - Relative Standard Deviations Calculated for the Reference Laboratory (Continued)

Matrix	Statistic	Silver	Vanadium	Zinc
All Soil	Number	13	21	35
	Minimum	2.3%	0.0%	1.0%
	Maximum	37.1%	18.1%	46.5%
	Mean	12.4%	8.4%	10.4%
	Median	7.5%	6.6%	9.1%
All Sediment	Number	10	17	27
	Minimum	1.0%	2.2%	1.4%
	Maximum	21.3%	21.9%	35.8%
	Mean	9.4%	8.4%	8.9%
	Median	6.6%	8.1%	6.9%
All	Number	23	38	62
	Minimum	1.0%	0.0%	1.0%
	Maximum	37.1%	21.9%	46.5%
	Mean	11.1%	8.4%	9.8%
	Median	7.1%	7.2%	7.4%

Notes:

Number	Number of demonstration samples evaluated.
RSD	Relative standard deviation.

Table E-7. Evaluation of the Effects of Interferent Elements on RPDs (Accuracy) of Other Target Elements, Oxford X-Met 3000TX Revised Data Set[1]

Parameter	Statistic	Lead Effects on Arsenic			Copper Effects on Nickel			Nickel Effects on Copper		
Interferent/Element Ratio		<5	5 - 10	>10	<5	5 - 10	>10	<5	5 - 10	>10
Number of Samples		29	7	3	17	1	2	40	0	8
RPD of Target Element[2]	Minimum	-60.2%	-29.6%	-13.9%	-41.3%	14.0%	27.2%	-54.2%	NC	-101.2%
	Maximum	16.9%	64.3%	116.4%	34.0%	14.0%	58.4%	24.5%	NC	-51.5%
	Mean	-21.8%	4.2%	61.1%	-2.9%	14.0%	42.8%	-19.7%	NC	-68.9%
	Median	-22.5%	4.7%	80.9%	-2.7%	14.0%	42.8%	-18.5%	NC	-65.1%
RPD of Target Element[2] (Absolute Value)	Minimum	0.2%	1.1%	13.9%	1.6%	14.0%	27.2%	1.1%	NC	51.5%
	Maximum	60.2%	64.3%	116.4%	41.3%	14.0%	58.4%	54.2%	NC	101.2%
	Mean	23.6%	20.0%	70.4%	16.7%	14.0%	42.8%	23.3%	NC	68.9%
	Median	22.5%	9.8%	80.9%	15.3%	14.0%	42.8%	21.5%	NC	65.1%
Interferent Concentration Range	Minimum	ND	757	1897	28	2418	2454	ND	NC	1458
	Maximum	942	51935	23284	1402	2418	6549	659	NC	3545
	Mean	209	17862	9375	243	2418	4501	282	NC	2564
	Median	86	7946	2944	177	2418	4501	206	NC	2556
Target Element Concentration Range	Minimum	34	110	23	165	206	119	43	NC	140
	Maximum	3425	6291	2322	3545	206	203	6549	NC	255
	Mean	351	2148	796	1388	206	161	1413	NC	210
	Median	140	941	41	659	206	161	1022	NC	225

Table E-7. Evaluation of the Effects of Interferent Elements on RPDs (Accuracy) of Other Target Elements, Oxford X-Met 3000TX Revised Data Set[1] (Continued)

Parameter	**Statistic**	**Zinc Effects on Copper**			**Copper Effects on Zinc**		
Interferent/Element Ratio		<5	5 - 10	>10	<5	5 - 10	>10
Number of Samples		35	2	11	48	3	10
RPD of Target Element[2]	Minimum	-101.2%	-37.8%	-71.2%	-89.9%	-69.7%	-83.5%
	Maximum	14.1%	-13.0%	24.5%	27.8%	-24.8%	-34.3%
	Mean	-25.3%	-25.4%	-36.5%	-19.4%	-52.8%	-52.2%
	Median	-24.1%	-25.4%	-44.9%	-19.8%	-63.8%	-55.1%
RPD of Target Element[2] (Absolute Value)	Minimum	1.1%	13.0%	13.6%	0.2%	24.8%	34.3%
	Maximum	101.2%	37.8%	71.2%	89.9%	69.7%	83.5%
	Mean	27.0%	25.4%	44.4%	25.5%	52.8%	52.2%
	Median	24.1%	25.4%	44.9%	21.3%	63.8%	55.1%
Interferent Concentration Range	Minimum	ND	753	926	ND	1293	1489
	Maximum	9747	11052	13001	2747	1613	6549
	Mean	1416	5903	4130	609	1436	3294
	Median	259	5903	3969	238	1402	2436
Target Element Concentration Range	Minimum	43	182	108	48	212	122
	Maximum	6549	2140	253	13001	267	435
	Mean	1542	1161	175	2214	233	243
	Median	1293	1161	150	825	220	236

Notes:
1. Concentrations are reported in units of milligrams per kilogram (mg/kg), or parts per million (ppm).
2. Table presents statistics for raw (unmodified) RPDs as well as absolute value RPDs.
< Less than.
> Greater than.
RPD Relative percent difference.
NC Not calculated because of a lack of XRF data.
ND Nondetect.
XRF X-ray fluorescence.

Table E-8. Evaluation of the Effects of Interferent Elements on RPDs (Accuracy) of Other Target Elements, Oxford X-Met 3000TX Original Data Set[1]

Parameter	Statistic	Copper Effects on Nickel			Nickel Effects on Copper		
Interferent/Element Ratio		<5	5 - 10	>10	<5	5 - 10	>10
Number of Samples		16	1	2	40	0	8
RPD of Target Element[2]	Minimum	-93.9%	14.0%	-109.7%	-54.2%	NC	-101.2%
	Maximum	179.7%	14.0%	27.2%	24.5%	NC	-51.5%
	Mean	24.4%	14.0%	-41.2%	-19.7%	NC	-68.9%
	Median	-0.2%	14.0%	-41.2%	-18.5%	NC	-65.1%
RPD of Target Element[2]	Minimum	1.6%	14.0%	27.2%	1.1%	NC	51.5%
(Absolute Value)	Maximum	179.7%	14.0%	109.7%	54.2%	NC	101.2%
	Mean	48.3%	14.0%	68.4%	23.3%	NC	68.9%
	Median	19.5%	14.0%	68.4%	21.5%	NC	65.1%
Interferent	Minimum	28	2418	2454	ND	NC	105
Concentration Range	Maximum	1402	2418	6549	743	NC	3545
	Mean	240	2418	4501	312	NC	1980
	Median	164	2418	4501	249	NC	1805
Target Element	Minimum	42	206	203	43	NC	140
Concentration Range	Maximum	3545	206	743	6549	NC	255
	Mean	1147	206	473	1413	NC	210
	Median	365	206	473	1022	NC	225

Notes:

1. Concentrations are reported in units of milligrams per kilogram (mg/kg), or parts per million (ppm).
2. Table presents statistics for raw (unmodified) RPDs as well as absolute value RPDs.

< Less than.
> Greater than.
RPD Relative percent difference.
NC Not calculated because of a lack of XRF data.
ND Nondetect.
XRF X-ray fluorescence.

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set

Matrix	Site	Matrix Description	Statistic	Antimony				Arsenic	
				Reference Laboratory		Certified Value		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	--	--	--	--
			Minimum	--	--	--	--	--	--
			Maximum	--	--	--	--	--	--
			Mean	--	--	--	--	--	--
			Median	--	--	--	--	--	--
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	1	1	6	6
			Minimum	-137.3%	85.8%	25.8%	25.8%	-18.9%	1.1%
			Maximum	-85.8%	137.3%	25.8%	25.8%	9.8%	18.9%
			Mean	-114.6%	114.6%	25.8%	25.8%	-2.1%	9.0%
			Median	-117.6%	117.6%	25.8%	25.8%	2.9%	7.5%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	1	1	1	1
			Minimum	-44.9%	44.9%	56.2%	56.2%	-44.0%	44.0%
			Maximum	-44.9%	44.9%	56.2%	56.2%	-44.0%	44.0%
			Mean	-44.9%	44.9%	56.2%	56.2%	-44.0%	44.0%
			Median	-44.9%	44.9%	56.2%	56.2%	-44.0%	44.0%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	1	1	--	--	--	--
			Minimum	1.8%	1.8%	--	--	--	--
			Maximum	1.8%	1.8%	--	--	--	--
			Mean	1.8%	1.8%	--	--	--	--
			Median	1.8%	1.8%	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	3	3	3	3	9	9
			Minimum	-76.2%	42.7%	35.9%	35.9%	-60.2%	3.7%
			Maximum	-42.7%	76.2%	66.4%	66.4%	16.9%	60.2%
			Mean	-61.5%	61.5%	51.7%	51.7%	-19.4%	24.9%
			Median	-65.8%	65.8%	52.7%	52.7%	-17.7%	17.7%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

				Cadmium		**Chromium**		**Copper**	
				Reference Laboratory		**Reference Laboratory**		**Reference Laboratory**	
Matrix	**Site**	**Matrix Description**	**Statistic**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**
Soil	AS	Fine to medium sand (steel processing)	Number	2	2	2	2	3	3
			Minimum	-15.3%	11.0%	-49.2%	10.9%	-15.8%	15.8%
			Maximum	11.0%	15.3%	-10.9%	49.2%	24.5%	24.5%
			Mean	-2.2%	13.1%	-30.0%	30.0%	9.2%	19.7%
			Median	-2.2%	13.1%	-30.0%	30.0%	18.8%	18.8%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	5	5	3	3	6	6
			Minimum	-7.1%	4.3%	-110.0%	2.8%	-71.2%	15.1%
			Maximum	20.8%	20.8%	-2.8%	110.0%	-15.1%	71.2%
			Mean	4.0%	8.6%	-49.6%	49.6%	-34.3%	34.3%
			Median	4.4%	6.3%	-35.9%	35.9%	-22.0%	22.0%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	1	1	3	3
			Minimum	-2.2%	2.2%	-71.7%	71.7%	-54.2%	1.1%
			Maximum	12.0%	12.0%	-71.7%	71.7%	-1.1%	54.2%
			Mean	4.9%	7.1%	-71.7%	71.7%	-29.1%	29.1%
			Median	4.9%	7.1%	-71.7%	71.7%	-32.1%	32.1%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	1	1	2	2
			Minimum	--	--	-40.7%	40.7%	-54.2%	18.4%
			Maximum	--	--	-40.7%	40.7%	-18.4%	54.2%
			Mean	--	--	-40.7%	40.7%	-36.3%	36.3%
			Median	--	--	-40.7%	40.7%	-36.3%	36.3%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	5	5	4	4	4	4
			Minimum	-21.7%	3.8%	-55.5%	30.9%	-101.2%	11.7%
			Maximum	3.8%	21.7%	-30.9%	55.5%	-11.7%	101.2%
			Mean	-8.8%	10.3%	-43.3%	43.3%	-47.6%	47.6%
			Median	-8.9%	8.9%	-43.5%	43.5%	-38.6%	38.6%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

				Iron		Lead		Mercury	
		Matrix		Reference Laboratory		Reference Laboratory		Reference Laboratory	
Matrix	Site	Description	Statistic	RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	3	3	3	3	--	--
			Minimum	-16.3%	16.3%	-35.3%	9.2%	--	--
			Maximum	47.1%	47.1%	9.3%	35.3%	--	--
			Mean	20.6%	31.5%	-5.6%	17.9%	--	--
			Median	31.1%	31.1%	9.2%	9.3%	--	--
Soil	BN	Sandy loam, low organic (ore residuals)	Number	7	7	6	6	--	--
			Minimum	5.5%	5.5%	-18.8%	4.9%	--	--
			Maximum	16.3%	16.3%	4.9%	18.8%	--	--
			Mean	10.5%	10.5%	-7.3%	8.9%	--	--
			Median	9.4%	9.4%	-7.5%	7.5%	--	--
Soil	CN	Sandy loam (burn pit residue)	Number	3	3	3	3	2	2
			Minimum	-15.5%	7.5%	-59.3%	6.7%	-30.1%	22.4%
			Maximum	34.6%	34.6%	84.4%	84.4%	-22.4%	30.1%
			Mean	8.9%	19.2%	10.6%	50.1%	-26.2%	26.2%
			Median	7.5%	15.5%	6.7%	59.3%	-26.2%	26.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	5	5	6	6	--	--
			Minimum	-157.9%	95.4%	-41.6%	1.3%	--	--
			Maximum	-95.4%	157.9%	7.0%	41.6%	--	--
			Mean	-125.9%	125.9%	-12.6%	15.4%	--	--
			Median	-130.1%	130.1%	-11.6%	12.1%	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	12	12	6	6	4	4
			Minimum	-127.4%	0.1%	-83.5%	31.6%	-88.2%	41.7%
			Maximum	14.4%	127.4%	-31.6%	83.5%	-41.7%	88.2%
			Mean	-15.6%	23.7%	-57.7%	57.7%	-61.6%	61.6%
			Median	-8.3%	13.4%	-59.0%	59.0%	-58.4%	58.4%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

				Nickel		Selenium		Silver	
				Reference Laboratory		Reference Laboratory		Reference Laboratory	
Matrix	Site	Matrix Description	Statistic	RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	1	1	1	1
			Minimum	--	--	0.8%	0.8%	-36.7%	36.7%
			Maximum	--	--	0.8%	0.8%	-36.7%	36.7%
			Mean	--	--	0.8%	0.8%	-36.7%	36.7%
			Median	--	--	0.8%	0.8%	-36.7%	36.7%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	2	2	4	4	4	4
			Minimum	-22.3%	1.6%	-15.0%	2.3%	-93.9%	16.6%
			Maximum	1.6%	22.3%	43.0%	43.0%	-16.6%	93.9%
			Mean	-10.4%	12.0%	4.9%	16.6%	-41.6%	41.6%
			Median	-10.4%	12.0%	-4.2%	10.6%	-28.0%	28.0%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	2	2	2	2
			Minimum	27.7%	27.7%	-29.5%	5.4%	-24.0%	1.9%
			Maximum	27.7%	27.7%	5.4%	29.5%	-1.9%	24.0%
			Mean	27.7%	27.7%	-12.1%	17.4%	-13.0%	13.0%
			Median	27.7%	27.7%	-12.1%	17.4%	-13.0%	13.0%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	1	1	--	--	--	--
			Minimum	-15.3%	15.3%	--	--	--	--
			Maximum	-15.3%	15.3%	--	--	--	--
			Mean	-15.3%	15.3%	--	--	--	--
			Median	-15.3%	15.3%	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	6	6	5	5	4	4
			Minimum	-41.3%	1.6%	-28.4%	2.1%	-60.8%	16.2%
			Maximum	58.4%	58.4%	4.5%	28.4%	-16.2%	60.8%
			Mean	1.9%	25.0%	-10.7%	12.5%	-36.5%	36.5%
			Median	-3.6%	21.7%	-9.2%	9.2%	-34.4%	34.4%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

Matrix	Site	Matrix Description	Statistic	Vanadium		Zinc	
				Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	3	3
			Minimum	--	--	-60.3%	13.1%
			Maximum	--	--	-13.1%	60.3%
			Mean	--	--	-38.1%	38.1%
			Median	--	--	-41.1%	41.1%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	1	1	7	7
			Minimum	-28.7%	28.7%	-35.9%	1.1%
			Maximum	-28.7%	28.7%	-1.1%	35.9%
			Mean	-28.7%	28.7%	-24.0%	24.0%
			Median	-28.7%	28.7%	-26.8%	26.8%
Soil	CN	Sandy loam (burn pit residue)	Number	--	--	3	3
			Minimum	--	--	-25.2%	11.5%
			Maximum	--	--	-11.5%	25.2%
			Mean	--	--	-19.6%	19.6%
			Median	--	--	-22.2%	22.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	1	1
			Minimum	--	--	-69.7%	69.7%
			Maximum	--	--	-69.7%	69.7%
			Mean	--	--	-69.7%	69.7%
			Median	--	--	-69.7%	69.7%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	10	10
			Minimum	-93.7%	33.8%	-89.9%	0.2%
			Maximum	-33.8%	93.7%	0.2%	89.9%
			Mean	-51.9%	51.9%	-43.8%	43.8%
			Median	-40.1%	40.1%	-30.7%	30.7%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

				Antimony				Arsenic	
				Reference Laboratory		Certified Value		Reference Laboratory	
Matrix	Site	Matrix Description	Statistic	RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Sediment	RF	Silty fine sand (tailings)	Number	2	2	2	2	11	11
			Minimum	-57.7%	49.9%	45.3%	45.3%	-36.5%	0.2%
			Maximum	-49.9%	57.7%	49.8%	49.8%	-0.2%	36.5%
			Mean	-53.8%	53.8%	47.6%	47.6%	-21.9%	21.9%
			Median	-53.8%	53.8%	47.6%	47.6%	-23.6%	23.6%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	4	4	1	1	5	5
			Minimum	-101.6%	54.1%	65.4%	65.4%	-33.8%	19.5%
			Maximum	-54.1%	101.6%	65.4%	65.4%	-19.5%	33.8%
			Mean	-84.0%	84.0%	65.4%	65.4%	-25.7%	25.7%
			Median	-90.1%	90.1%	65.4%	65.4%	-27.0%	27.0%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	3	3	3	1	1
			Minimum	-161.3%	141.2%	-13.0%	11.5%	116.4%	116.4%
			Maximum	-141.2%	161.3%	13.0%	13.0%	116.4%	116.4%
			Mean	-152.7%	152.7%	-3.8%	12.5%	116.4%	116.4%
			Median	-155.5%	155.5%	-11.5%	13.0%	116.4%	116.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	2	2	--	--	6	6
			Minimum	-115.1%	95.8%	--	--	-29.6%	11.7%
			Maximum	-95.8%	115.1%	--	--	80.9%	80.9%
			Mean	-105.4%	105.4%	--	--	10.7%	37.7%
			Median	-105.4%	105.4%	--	--	-12.8%	27.6%
	All		Number	20	20	11	11	39	39
			Minimum	-161.3%	1.8%	-13.0%	11.5%	-60.2%	0.2%
			Maximum	1.8%	161.3%	66.4%	66.4%	116.4%	116.4%
			Mean	-89.9%	90.1%	35.1%	39.5%	-10.8%	26.5%
			Median	-90.1%	90.1%	45.3%	45.3%	-18.9%	22.5%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

Matrix	Site	Matrix Description	Statistic	Cadmium		Chromium		Copper	
				Reference Laboratory		Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Sediment	RF	Silty fine sand (tailings)	Number	5	5	4	4	13	13
			Minimum	-21.7%	0.3%	-34.1%	23.5%	-66.7%	16.9%
			Maximum	-0.3%	21.7%	-23.5%	34.1%	-16.9%	66.7%
			Mean	-9.0%	9.0%	-28.1%	28.1%	-36.2%	36.2%
			Median	-4.3%	4.3%	-27.4%	27.4%	-35.6%	35.6%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	1	1	5	5	4	4
			Minimum	-14.9%	14.9%	-55.7%	27.3%	-82.5%	13.6%
			Maximum	-14.9%	14.9%	-27.3%	55.7%	14.1%	82.5%
			Mean	-14.9%	14.9%	-39.3%	39.3%	-33.4%	40.4%
			Median	-14.9%	14.9%	-36.8%	36.8%	-32.5%	32.8%
Sediment	TL	Silt and clay (slag-enriched)	Number	2	2	1	1	7	7
			Minimum	-4.7%	4.7%	-78.0%	78.0%	-8.1%	1.7%
			Maximum	7.5%	7.5%	-78.0%	78.0%	11.5%	11.5%
			Mean	1.4%	6.1%	-78.0%	78.0%	-0.5%	5.1%
			Median	1.4%	6.1%	-78.0%	78.0%	-2.1%	4.3%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	3	2	2	6	6
			Minimum	-90.6%	74.0%	-151.7%	34.1%	-44.9%	13.0%
			Maximum	-74.0%	90.6%	-34.1%	151.7%	-13.0%	44.9%
			Mean	-84.8%	84.8%	-92.9%	92.9%	-33.8%	33.8%
			Median	-89.8%	89.8%	-92.9%	92.9%	-37.0%	37.0%
	All		Number	25	25	23	23	48	48
			Minimum	-90.6%	0.3%	-151.7%	2.8%	-101.2%	1.1%
			Maximum	20.8%	90.6%	-2.8%	151.7%	24.5%	101.2%
			Mean	-13.2%	18.5%	-46.4%	46.4%	-27.9%	30.9%
			Median	-4.7%	8.9%	-36.8%	36.8%	-26.8%	26.8%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

				Iron		**Lead**		**Mercury**	
				Reference Laboratory		**Reference Laboratory**		**Reference Laboratory**	
Matrix	**Site**	**Matrix Description**	**Statistic**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**
Sediment	RF	Silty fine sand (tailings)	Number	13	13	11	11	5	5
			Minimum	-15.4%	0.6%	-89.3%	5.0%	-135.9%	61.7%
			Maximum	14.4%	15.4%	-5.0%	89.3%	-61.7%	135.9%
			Mean	-3.0%	7.1%	-32.7%	32.7%	-87.2%	87.2%
			Median	-3.7%	5.9%	-22.1%	22.1%	-79.6%	79.6%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	12	12	6	6	10	10
			Minimum	-39.8%	0.1%	-93.8%	7.2%	-45.4%	1.6%
			Maximum	33.6%	39.8%	-7.2%	93.8%	46.1%	46.1%
			Mean	17.4%	24.0%	-54.3%	54.3%	5.9%	27.5%
			Median	23.9%	24.5%	-60.2%	60.2%	11.1%	26.4%
Sediment	TL	Silt and clay (slag-enriched)	Number	7	7	4	4	2	2
			Minimum	-52.3%	8.8%	-71.3%	0.7%	-16.9%	16.9%
			Maximum	12.8%	52.3%	0.7%	71.3%	65.1%	65.1%
			Mean	-16.1%	22.6%	-30.9%	31.2%	24.1%	41.0%
			Median	-10.0%	12.8%	-26.4%	26.4%	24.1%	41.0%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	7	7	7	7	--	--
			Minimum	-21.9%	0.9%	-45.6%	3.6%	--	--
			Maximum	25.1%	25.1%	-3.6%	45.6%	--	--
			Mean	3.8%	12.7%	-25.8%	25.8%	--	--
			Median	0.9%	8.4%	-25.1%	25.1%	--	--
	All		Number	69	69	52	52	23	23
			Minimum	-157.9%	0.1%	-93.8%	0.7%	-135.9%	1.6%
			Maximum	47.1%	157.9%	84.4%	93.8%	65.1%	135.9%
			Mean	-8.3%	25.6%	-27.7%	32.5%	-27.3%	47.5%
			Median	0.9%	14.4%	-23.0%	23.9%	-30.1%	43.1%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

Matrix	Site	Matrix Description	Statistic	Nickel		Selenium		Silver	
				Reference Laboratory		Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Sediment	RF	Silty fine sand (tailings)	Number	6	6	5	5	4	4
			Minimum	-29.5%	2.0%	-16.7%	4.7%	-110.4%	18.4%
			Maximum	34.0%	34.0%	-4.7%	16.7%	-18.4%	110.4%
			Mean	3.3%	18.3%	-9.9%	9.9%	-56.2%	56.2%
			Median	6.0%	15.3%	-8.5%	8.5%	-48.0%	48.0%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	2	2	3	3	1	1
			Minimum	-12.2%	2.7%	2.0%	2.0%	-86.0%	86.0%
			Maximum	-2.7%	12.2%	5.0%	5.0%	-86.0%	86.0%
			Mean	-7.4%	7.4%	3.2%	3.2%	-86.0%	86.0%
			Median	-7.4%	7.4%	2.6%	2.6%	-86.0%	86.0%
Sediment	TL	Silt and clay (slag-enriched)	Number	--	--	4	4	4	4
			Minimum	--	--	-20.4%	4.4%	-44.9%	4.4%
			Maximum	--	--	7.3%	20.4%	4.4%	44.9%
			Mean	--	--	-7.7%	11.4%	-24.1%	26.3%
			Median	--	--	-8.9%	10.4%	-27.9%	27.9%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	2	2	1	1	4	4
			Minimum	14.8%	14.8%	-5.6%	5.6%	-42.3%	19.4%
			Maximum	27.2%	27.2%	-5.6%	5.6%	-19.4%	42.3%
			Mean	21.0%	21.0%	-5.6%	5.6%	-28.9%	28.9%
			Median	21.0%	21.0%	-5.6%	5.6%	-27.0%	27.0%
	All		Number	20	20	25	25	24	24
			Minimum	-41.3%	1.6%	-29.5%	0.8%	-110.4%	1.9%
			Maximum	58.4%	58.4%	43.0%	43.0%	4.4%	110.4%
			Mean	2.5%	19.2%	-5.3%	11.0%	-37.4%	37.8%
			Median	-1.8%	16.0%	-5.6%	6.7%	-32.4%	32.4%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

				Vanadium		Zinc	
				Reference Laboratory		Reference Laboratory	
Matrix	Site	Matrix Description	Statistic	RPD	RPD ABS Val	RPD	RPD ABS Val
Sediment	RF	Silty fine sand (tailings)	Number	2	2	13	13
			Minimum	-58.9%	3.4%	-63.8%	4.9%
			Maximum	-3.4%	58.9%	-4.9%	63.8%
			Mean	-31.1%	31.1%	-27.3%	27.3%
			Median	-31.1%	31.1%	-20.5%	20.5%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	--	--	10	10
			Minimum	--	--	-35.0%	2.2%
			Maximum	--	--	27.8%	35.0%
			Mean	--	--	5.3%	15.7%
			Median	--	--	9.5%	14.1%
Sediment	TL	Silt and clay (slag-enriched)	Number	--	--	7	7
			Minimum	--	--	-59.1%	24.8%
			Maximum	--	--	-24.8%	59.1%
			Mean	--	--	-44.2%	44.2%
			Median	--	--	-40.0%	40.0%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	1	1	7	7
			Minimum	19.6%	19.6%	-55.7%	16.2%
			Maximum	19.6%	19.6%	23.5%	55.7%
			Mean	19.6%	19.6%	-21.7%	33.0%
			Median	19.6%	19.6%	-33.7%	33.7%
	All		Number	8	8	61	61
			Minimum	-93.7%	3.4%	-89.9%	0.2%
			Maximum	19.6%	93.7%	27.8%	89.9%
			Mean	-34.9%	39.8%	-26.4%	31.2%
			Median	-36.6%	36.6%	-25.4%	25.8%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Revised Data Set (Continued)

Site Abbreviations:

AS	Alton Steel Mill
BN	Burlington Northern Railroad/ASARCO East
CN	Naval Surface Warfare Center, Crane Division
KP	KARS Park -- Kennedy Space Center
LV	Leviathan Mine/Aspen Creek
RF	Ramsey Flats – Silver Bow Creek
SB	Sulfur Bank Mercury Mine
TL	Torch Lake Superfund Site
WS	Wickes Smelter Site

Other Notes:

--	No samples reported by the reference laboratory in this concentration range.
Number	Number of demonstration samples evaluated.
RPD	Relative percent difference (unmodified).
RPD Abs Val	Relative percent difference (absolute value).

Table E-10. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Original Data Set

				Mercury		Nickel		Selenium	
				Reference Laboratory		Reference Laboratory		Reference Laboratory	
Matrix	**Site**	**Matrix Description**	**Statistic**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	--	--	1	1
			Minimum	--	--	--	--	0.8%	0.8%
			Maximum	--	--	--	--	0.8%	0.8%
			Mean	--	--	--	--	0.8%	0.8%
			Median	--	--	--	--	0.8%	0.8%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	--	--	2	2	3	3
			Minimum	--	--	-22.3%	1.6%	-188.3%	185.0%
			Maximum	--	--	1.6%	22.3%	-185.0%	188.3%
			Mean	--	--	-10.4%	12.0%	-187.2%	187.2%
			Median	--	--	-10.4%	12.0%	-188.2%	188.2%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	1	1	2	2
			Minimum	-30.1%	22.4%	27.7%	27.7%	-29.5%	5.4%
			Maximum	-22.4%	30.1%	27.7%	27.7%	5.4%	29.5%
			Mean	-26.2%	26.2%	27.7%	27.7%	-12.1%	17.4%
			Median	-26.2%	26.2%	27.7%	27.7%	-12.1%	17.4%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	1	1	--	--
			Minimum	--	--	-15.3%	15.3%	--	--
			Maximum	--	--	-15.3%	15.3%	--	--
			Mean	--	--	-15.3%	15.3%	--	--
			Median	--	--	-15.3%	15.3%	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	5	5	5	5
			Minimum	-88.2%	41.7%	-109.7%	93.9%	-28.4%	2.1%
			Maximum	-41.7%	88.2%	179.7%	179.7%	4.5%	28.4%
			Mean	-61.6%	61.6%	56.6%	138.0%	-10.7%	12.5%
			Median	-58.4%	58.4%	136.2%	136.2%	-9.2%	9.2%

Table E-10. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Original Data Set (Continued)

				Mercury		Nickel		Selenium	
				Reference Laboratory		Reference Laboratory		Reference Laboratory	
Matrix	**Site**	**Matrix Description**	**Statistic**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**	**RPD**	**RPD ABS Val**
Sediment	RF	Silty fine sand (tailings)	Number	5	5	6	6	5	5
			Minimum	-135.9%	61.7%	-29.5%	2.0%	-16.7%	4.7%
			Maximum	-61.7%	135.9%	34.0%	34.0%	-4.7%	16.7%
			Mean	-87.2%	87.2%	3.3%	18.3%	-9.9%	9.9%
			Median	-79.6%	79.6%	6.0%	15.3%	-8.5%	8.5%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	10	10	2	2	3	3
			Minimum	35.9%	35.9%	-12.2%	2.7%	2.0%	2.0%
			Maximum	195.3%	195.3%	-2.7%	12.2%	5.0%	5.0%
			Mean	146.5%	146.5%	-7.4%	7.4%	3.2%	3.2%
			Median	174.1%	174.1%	-7.4%	7.4%	2.6%	2.6%
Sediment	TL	Silt and clay (slag-enriched)	Number	2	2	--	--	4	4
			Minimum	-16.9%	16.9%	--	--	-20.4%	4.4%
			Maximum	65.1%	65.1%	--	--	7.3%	20.4%
			Mean	24.1%	41.0%	--	--	-7.7%	11.4%
			Median	24.1%	41.0%	--	--	-8.9%	10.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	--	2	2	1	1
			Minimum	--	--	14.8%	14.8%	-5.6%	5.6%
			Maximum	--	--	27.2%	27.2%	-5.6%	5.6%
			Mean	--	--	21.0%	21.0%	-5.6%	5.6%
			Median	--	--	21.0%	21.0%	-5.6%	5.6%
	All		Number	23	23	19	19	24	24
			Minimum	-135.9%	16.9%	-109.7%	1.6%	-188.3%	0.8%
			Maximum	195.3%	195.3%	179.7%	179.7%	7.3%	188.3%
			Mean	33.8%	99.2%	16.9%	48.6%	-29.8%	32.1%
			Median	-16.9%	79.6%	1.6%	22.3%	-7.6%	7.9%

Table E-10. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements, Oxford X-Met 3000TX Original Data Set (Continued)

Site Abbreviations:

AS	Alton Steel Mill
BN	Burlington Northern Railroad/ASARCO East
CN	Naval Surface Warfare Center, Crane Division
KP	KARS Park -- Kennedy Space Center
LV	Leviathan Mine/Aspen Creek
RF	Ramsey Flats -- Silver Bow Creek
SB	Sulfur Bank Mercury Mine
TL	Torch Lake Superfund Site
WS	Wickes Smelter Site

Other Notes:

--	No samples reported by the reference laboratory in this concentration range.
Number	Number of demonstration samples evaluated.
RPD	Relative percent difference (unmodified).
RPD Abs Val	Relative percent difference (absolute value).