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Innovative Technology Verification Report

XRF Technologies for Measuring Trace Elements in Soil and Sediment

Oxford ED2000 XRF Analyzer



Innovative Technology Verification Report

Oxford ED2000 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.

Gary Foley, Ph.D.
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Abstract

The Oxford ED2000 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 ED2000 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 ED2000 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 Oxford ED2000 bench-top XRF analyzer is an energy dispersive XRF analyzer that can be operated in a mobile laboratory or similar setting. The ED2000 can analyze up to 75 elements in a variety of sample matrices, including contaminated soils and sediments, liquids, powders, granules, filter papers, or films. The measurement of light-end elements (sodium to iron) can be determined when the samples are prepared as pressed pellets. Oxford provides a calibration service as an option to customers for specific projects and applications using this analyzer.

This report describes the results of the evaluation of the ED2000 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 ED2000 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 ED2000 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	XLt 700 Series XLi 700 Series	XLt XLi
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, Inc., 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.

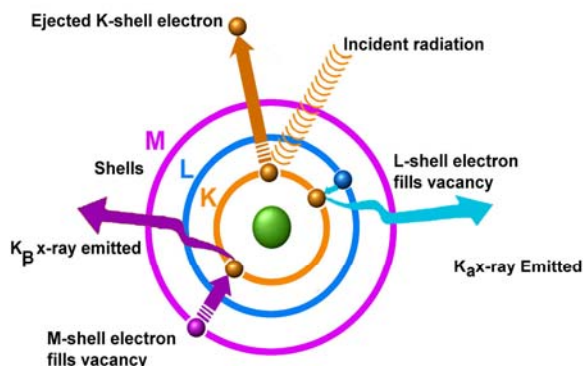


Figure 1-1. The XRF process.

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 reduced 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 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	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					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
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			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.

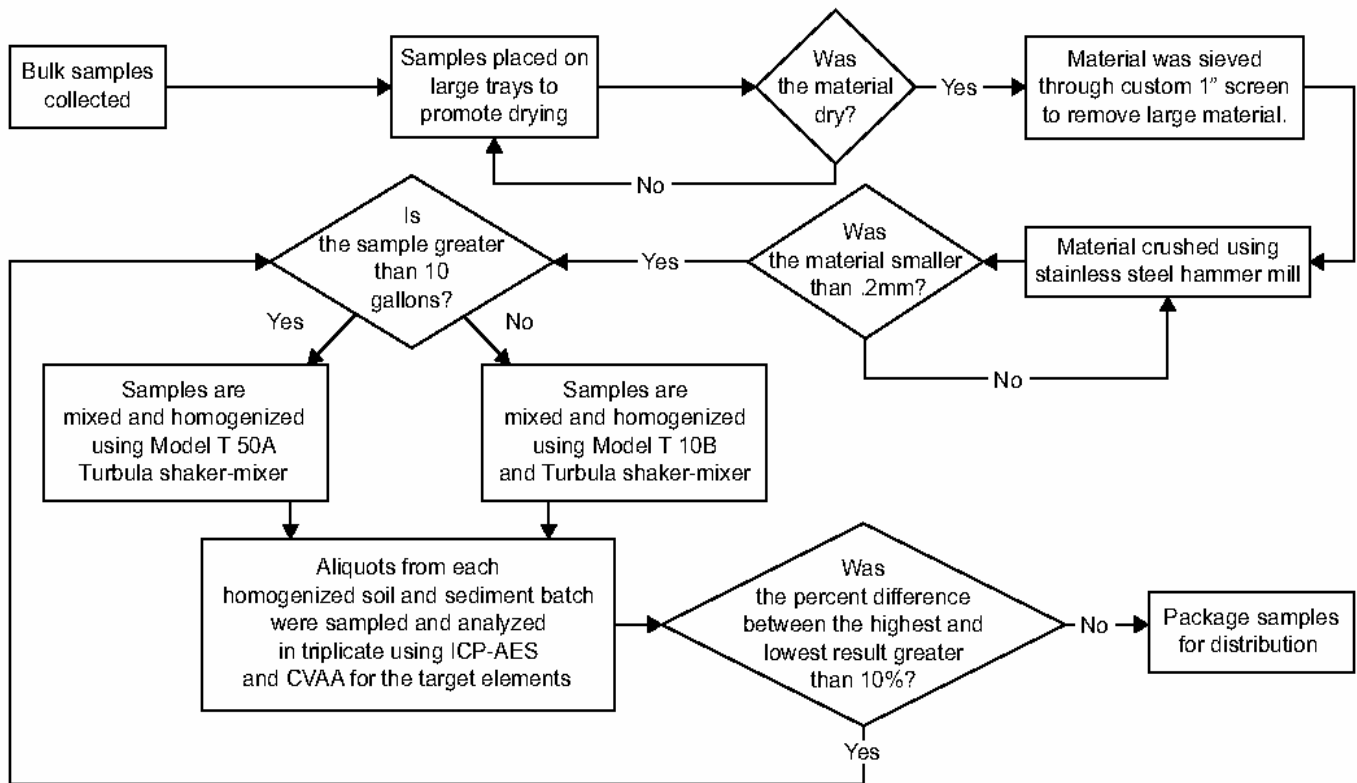


Figure 3-1. Bulk sample processing diagram.

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
Ramsay 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:

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

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:

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

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:

$$RSD = \left| \frac{SD}{\bar{C}} \right| \times 100$$

where

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

The standard deviation was calculated using the equation:

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

where

SD = Standard deviation
n = Number of replicate samples
 C_k = Concentration of sample K
 \bar{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 therefore 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 siliceous 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.

Table 5-1. Number of Validation Qualifiers

Element	Number and Percentage of Qualified Results per QC type ¹					
	Method Blank		MS/MSD		Serial Dilution	
	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).

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.

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:

¹Values 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:

¹Values 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 ED2000 XRF analyzer is manufactured by Oxford Instruments Analytical Ltd (Oxford). This chapter provides a technical description of the ED2000 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 Oxford ED2000 is an energy dispersive XRF analyzer that can be operated as a bench-top unit in a mobile laboratory or similar setting. The entire analyzer system includes three major components: (1) XRF spectrometer, (2) vacuum pump, and (3) personal computer. Each ED2000 unit comes equipped with a modem so that the instrument can be controlled remotely for ease of operation. This feature also allows qualified technicians to evaluate system functionality and provide troubleshooting guidance for inexperienced users from a remote location.

The ED2000 can analyze up to 75 elements in a variety of sample matrices, including contaminated soils and sediments, liquids, powders, granules, filter papers, or films. The measurement of light-end elements (sodium to iron) can be determined when the samples are prepared as pressed pellets. Oxford provides a calibration service as an option to customers using this analyzer.

The ED2000 system includes a SMART digital pulse processor to handle count rates as high as 90,000 counts per second (CPS). The high count rates and high detector resolutions provide improved precision and lower detection levels compared to older Oxford XRF analyzers. Oxford's XpertEase 32 software is designed for detecting elements across the full specification and allows automated processing to free up the operator during routine analyses. Special features of the ED2000 include a 16-position automatic sample tray, 10 liter dewar to hold liquid nitrogen to cool the detector, vacuum pump to evacuate the sample chamber of the instrument to

reduce the formation of oxides in the sample matrix, and a personal computer loaded with Microsoft Windows® XP and instrument calibration and operational software. The ED2000 is shown in a bench-top configuration in Figure 6-1. Technical specifications for the ED2000 are provided in Table 6-1.



Figure 6-1. Oxford ED2000 analyzer set up for bench-top analysis.

For the demonstration, a sample crusher/mixer and sample press was included with the analyzer to prepare sample pellets for analysis. However, it should be noted that processing samples into pellets is not required for routine sample analysis.

6.2 Instrument Operations during the Demonstration

The ED2000 and accessories were shipped to the demonstration site on three 4-foot by 4-foot wooden pallets. One pallet contained the analyzer, which was over-packed in a large cardboard box with Styrofoam padding and strapped to the pallet. The two other pallets contained the sample crusher/mixer, the sample press, a computer, monitor, printer, and disposable analytical supplies. Additionally, a local supplier delivered a tank of liquid nitrogen, which is used to cool the detector during analyses. Unpacking

the three pallets and carrying the analyzer and accessories into the demonstration building required the help of four individuals. The analyzer, sample

crusher/mixer, and sample press each weighed over 150 pounds (70 kg).

Table 6-1. Oxford ED2000 XRF Analyzer Technical Specifications

Weight:	75 kilograms (XRF spectrometer only).
Dimensions:	570 millimeters (mm) wide, 500 mm deep, and 200 mm high.
Excitation Source:	X-ray tube programmable 4-50kV, 1-1,000 μ A (maximum 50 watts). Stability <0.2%/8 hrs. Silver x-ray tube target.
Filters:	Fully programmable; 8 filter positions.
Detector:	Patented Pentafet detector and digital pulse processor. Guaranteed resolution of <150eV with 17,000 cps input rate. Output count rate >90,000 cps. Liquid Nitrogen Dewar: 10-liter capacity.
Software:	Oxford owns the XpertEase Windows software package, which allows qualitative, semi-quantitative, and full quantitative analysis. Special features include pre-programmed analytical parameters; full spectrometer control, data library, x-ray mathematical models.
Element Range:	Sodium to uranium.
Number of Elements:	Up to 75 elements for qualitative analysis and full quantitative analysis.
Concentration Range:	ppm to 100%.
Sample Form:	Solids, liquids, powders, granules, filter papers, films.
Sample Sizes:	From 0.2 mm to 250 mm diameter.
Sample Chamber:	Air path, helium/vacuum options. 250 mm diameter x 90 mm deep. Standard automated 16-position sample carousel. Options include 8-position sample carousel and sample spinner.
Computer:	IBM compatible computer, 2.8 GHz Pentium IV processor, 80 GB hard disk, 128 MB RAM, including 15-inch SVGA color monitor, 105-key keyboard, two-button mouse and associated ink jet printer.
Interface:	External RS232 port.
Operating Environment:	Temperature: 5 to 30 °C; 20 to 80% relative (non-condensing).
Power Requirements:	110-125 or 220-250V AC, 50/60 Hz 10 amps.

6.2.1 Set up and Calibration

Unpacking the analyzer and accessories required approximately 1 hour and the primary assembling of the XRF unit required an additional 2 hours. Due to an unfortunate electrical shortage when the main laptop PC was plugged in (European voltage turned on, rather than U.S. voltage), the laptop PC was damaged. As a result, the initial empirical calibration curve, which was developed using the pre-demonstration samples and stored on the laptop PC hard drive, was not immediately available. The empirical calibration information was sent to the Oxford demonstration team via email and loaded onto a second laptop PC the next day. For the demonstration, approximately 6 hours were needed to make the ED2000 operational. Oxford states that an experienced technician can set up and calibrate the ED2000 in one to four hours. The Oxford XpertEase software was used to set up and operate the ED2000. Each menu helps guide the user through the process of turning on the x-ray tube and initializing the spectrometer optics and detector. The elements and their characteristic energy wavelengths for analysis and the measurement units are selected using the software. The Oxford empirical and factory calibration curves were verified by analyzing some of the pre-demonstration samples and National Institute of Standards and Technology (NIST) standards. The empirical calibration information was used for all sample analyses during the demonstration.

6.2.2 Demonstration Sample Processing

Oxford sent a two-man team to the demonstration site to process the demonstration samples using the ED2000. The field team including a senior instrument specialist, who operated the instrument and reduced the data, and a senior sales representative, who served as the sample preparation technician.

Sample preparation by Oxford for this demonstration involved pressing ground and homogenized soil and sediment samples into pellets for analysis. However, the ED2000 can accommodate non-pressed samples in polyethylene cups and covered with Mylar® film. The initial pre-demonstration calibration curve was developed using pelletized samples and Oxford determined that using the same sample preparation

techniques was important for this demonstration in order to minimize error and maximize precision and accuracy.

The sample processing steps included weighing 9 grams of soil, adding five wax pellets, and placing the mixture in a stainless steel dish. A titanium plug was also placed in the dish with the sample and wax to aid in crushing. The dish was covered and the sample vigorously shaken in the sample crusher/mixer for approximately 10 seconds. The crushing and mixing helped to homogenize the sample with the crushed wax, and further reduced the particle size to approximately 70 microns. The mixture was then placed in a stainless steel cylinder and the sample pressed into a cylindrical shaped pellet using an aluminum sample cup and a hydraulic press (Figures 6-2 and 6-3.) The aluminum sample cup did not affect the XRF analysis because the x-rays only penetrate the sample approximately 2 millimeters and the pelletized samples were approximately 10 millimeters thick. All re-usable parts in contact with a soil or sediment sample were cleaned with water and paper towels. For particularly stubborn particles, a small amount of denatured alcohol was used. The total time required to prepare a sample for this demonstration ranged from 9 to 15 minutes.



Figure 6-2. Oxford ED2000 soil pellet sample preparation.

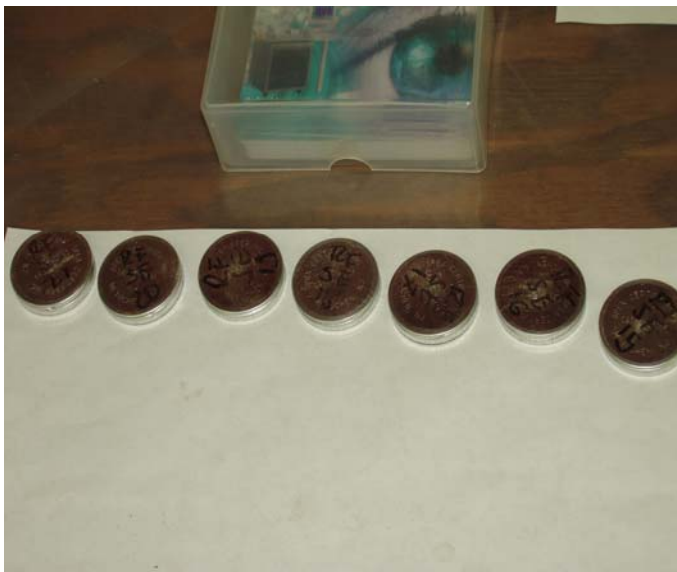


Figure 6-3. Oxford ED2000 pelletized samples for XRF analysis.

A combination of NIST standards and pre-demonstration samples were used to create the calibration curve for the demonstration. The calibration curve had 19 points of concentration/response and was developed by analyzing the known concentration samples for 215 seconds. Each demonstration analysis batch involved filling the 16 position auto-sampler with 15 demonstration samples and one standard. Actual XRF analysis time for each sample varied between five to nine minutes with an average run time of around eight minutes. Differences in analysis run times resulted from the analyzer optimizing each individual sample by selecting the number of filters used and the number of counts per second. Samples with large variability in element concentrations and density required longer analysis times. After a sixteen sample batch sequence was completed, Oxford would review the data and save it to the ED2000 operating system.

Another batch of 16 samples was loaded into the autosampler and the analyses started. The XRF demonstration analytical results were transferred to

the laptop PC daily for storage and manipulation. Final demonstration data were transferred at the end of the demonstration from a universal serial bus (USB) portable storage drive to the database maintained by Tetra Tech for all demonstration data.

The ED2000 operating system did experience several software glitches that stopped the XRF analysis runs. When this occurred, the system was rebooted and the incomplete sample analyses were restarted. In addition, the auto-sampler slot 8 experienced some repeated problems accommodating samples which required some system checks and runs of only 15 samples.

6.3 General Demonstration Results

The ED2000 required substantial effort to unpack, assemble, and prepare for operation due to the number of components and both the size and weight of these components. Oxford prepared and analyzed all 326 demonstration samples in 4 days (January 25 through 28, 2005) following a day spent resolving the computer problems identified in Section 6.2.1. On this basis, the observer estimated a routine throughput of 50 to 60 samples per 8-hour day, depending on the specific processing steps and the analysis run time.

6.4 Contact Information

Additional information on Oxford's ED2000 XRF analyzer is available from the following source:

Dr. John I.H. Patterson
Oxford Instruments Analytical
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 25 and 28, 2005. The samples were analyzed in batches of 16 using the instrument autosampler. Final data were transferred at the end of the demonstration from a USB portable storage drive to the database Tetra Tech maintained for all demonstration data. All the data Oxford provided at the close of the demonstration are tabulated and compared with the reference laboratory data and the ERA-certified spike concentrations, as applicable, in Appendix D.

The ED2000 data set was reviewed and evaluated in accordance with the primary and secondary objectives of the demonstration. The findings of the evaluation for each objective are presented below.

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 no concentration data for some of the target elements in these blends. In these cases, the ED2000's quantitation algorithms could not resolve a signal for a specific element that was greater than instrument "noise" at an acceptable level of statistical significance. Additional information on instrument algorithms and detection thresholds is available from the developer; contact information is provided at the end of Chapter 6. In selecting samples from among the 12 blends to

calculate MDLs, blends were not used where the developer reported no concentration for one or more of the seven replicates.

The MDLs calculated for the ED2000 are presented in Table 7-1. As shown, the data for the MDL blends allowed only two MDLs (one for soil and one for sediment) to be calculated for mercury. Between six and 12 MDLs were calculated for the remaining target elements. The mean MDLs in Table 7-1 are classified as follows:

- Very low (1 to 20 ppm): antimony, cadmium, copper, selenium, silver, and vanadium.
- Low (20 to 50 ppm): arsenic, chromium, lead, mercury, nickel, and zinc.
- Medium (50 to 100 ppm): none.
- High (greater than 100 ppm): none.

No trends could be discerned in the MDLs calculated in terms of sample matrix (soil versus sediment) or blend. For antimony, however, the mean calculated MDL of 18 ppm was biased high by an extreme value of 69 ppm calculated for Blend 65. This MDL was more than four times higher than the next highest MDL of 16 ppm calculated for antimony in Blend 12. Review of the analytical data for Blend 65 indicated that the high MDL was the result of a high relative degree of imprecision in the replicate results the vendor reported for this blend.

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Oxford ED2000¹

Matrix	Blend No.	Antimony			Arsenic			Cadmium			Chromium		
		ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴
Soil	2	7	17	17	23	121	1.5	NC	ND	ND	67	351	167
Soil	5	NC	ND	ND	21	69	47	3	2	1.9	81	107	121
Soil	6	11	33	8	NC	886	477	3	10	12	79	109	133
Soil	8	NC	481	118	NC	17,111	3,943	7	49	91	74	71	55
Soil	10	7	3	ND	10	55	39	NC	ND	0.96	16	101	116
Soil	12	16	241	62	NC	1,229	559	14	212	263	18	81	101
Soil	18	5	8	ND	25	20	9	NC	ND	ND	40	228	150
Sediment	29	NC	ND	ND	25	23	10	NC	ND	ND	25	79	63
Sediment	31	NC	ND	ND	35	38	11	NC	ND	ND	28	160	133
Sediment	32	6	3	ND	38	47	31	NC	ND	ND	12	89	75
Sediment	39	NC	ND	ND	14	24	14	3	1	ND	99	107	102
Sediment	65	69	54	11	41	309	250	3	47	44	39	329	303
Mean ED2000 MDL		18			26			6			48		
Matrix	Blend No.	Copper			Lead			Mercury			Nickel		
		ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴
Soil	2	9	47	47	NC	1,033	1,200	NC	ND	ND	17	123	83
Soil	5	15	58	49	68	95	78	NC	ND	ND	16	78	60
Soil	6	26	179	160	NC	3,273	3,986	NC	ND	0.83	14	101	70
Soil	8	NC	2,716	1,243	NC	29,881	33,429	NC	ND	15	84	277	57
Soil	10	12	40	31	30	70	72	NC	ND	0.14	10	78	60
Soil	12	NC	953	747	NC	3,745	4,214	NC	ND	1.8	33	137	91
Soil	18	25	51	50	13	17	17	11	54	56	31	289	213
Sediment	29	NC	1,997	1,986	27	41	33	NC	ND	0.24	13	154	72
Sediment	31	NC	1,696	1,514	20	56	51	NC	ND	ND	34	377	196
Sediment	32	9	41	36	12	36	26	NC	ND	ND	18	194	174
Sediment	39	20	111	94	13	45	27	NC	ND	ND	24	263	202
Sediment	65	27	87	69	20	43	25	34	24	32	29	325	214
Mean ED2000 MDL		18			25			23			27		

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Oxford ED2000¹ (Continued)

Matrix	Blend No.	Selenium			Silver			Vanadium			Zinc		
		ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴	ED2000 MDL ²	ED2000 Conc ³	Ref. Lab Conc ⁴
Soil	2	3	2	ND	NC	ND	ND	NC	29	1.2	30	34	24
Soil	5	NC	ND	ND	NC	ND	0.93	NC	84	55	31	254	229
Soil	6	NC	ND	ND	6	11	14	6	79	56	NC	889	886
Soil	8	NC	ND	ND	7	69	144	7	65	34	NC	11,812	5,657
Soil	10	NC	ND	ND	NC	ND	ND	NC	70	51	27	111	92
Soil	12	3	12	15	2	28	38	2	60	45	NC	2,745	2,114
Soil	18	4	1	ND	NC	ND	ND	NC	123	67	22	106	90
Sediment	29	NC	ND	ND	3	3	ND	3	73	96	55	190	160
Sediment	31	NC	ND	ND	3	4	6.2	3	81	76	45	163	137
Sediment	32	3	5	4.6	NC	ND	ND	NC	81	57	29	96	69
Sediment	39	6	3	ND	2	1	ND	2	62	38	34	164	137
Sediment	65	7	16	22	7	39	41	7	64	31	NC	2175	1,843
Mean ED2000 MDL		4			4			4			34		

Notes and abbreviations:

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

² MDLs calculated from the 12 MDL sample blends for the ED2000 in this technology demonstration (in bold typeface for emphasis).

³ This column lists the mean concentration reported for this MDL sample blend by the ED2000.

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

Conc Concentration

MDL Method detection limit.

NC The MDL was not calculated because reference laboratory concentrations exceeded five times the expected MDL range of 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.” 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.

The mean MDLs calculated for the ED2000 are compared in Table 7-2 with the mean MDLs for all developers that participated in the demonstration and the mean MDLs derived from performance data presented in EPA Method 6200 (EPA 1998e). As shown, the mean MDLs for the ED2000 are comparable to or lower than the demonstration-wide means for all the target elements. The greatest differences between the Oxford ED 2000 data and the demonstration data as a whole are observed for antimony, cadmium, silver, and vanadium, where the ED2000 MDLs are one-third or less of the

demonstration-wide means. The ED2000 MDLs are also significantly lower than the mean MDLs calculated from EPA Method 6200 data for all of the target elements. Reasons for the slightly increased sensitivity of the ED2000 relative to other field portable XRF instruments may include: (1) the high-resolution, nitrogen-cooled detector that was used, (2) the evacuation of the sample chamber in the instrument to limit x-ray scattering by air during analysis, and (3) the program-specific calibration by the vendor using pre-demonstration samples (Chapter 6).

Table 7-2. Comparison of ED2000 MDLs to All –Instrument Mean MDLs and EPA Method 6200 Data¹

Element	Oxford ED2000 Mean MDLs²	All XRF Instrument Mean MDLs³	EPA Method 6200 Mean Detection Limits⁴
Antimony	18	61	55 ⁵
Arsenic	26	26	92
Cadmium	6	70	NR
Chromium	48	83	376
Copper	18	23	171
Lead	25	40	78
Mercury	23	23	NR
Nickel	27	50	100 ⁵
Selenium	4	8	NR
Silver	4	42	NR
Vanadium	4	28	NR
Zinc	34	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 participating in the technology demonstration.

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

⁵ Only one value reported.

EPA U.S. Environmental Protection Agency.

MDL Method detection limit.

NR No MDLs or LODs reported for this element.

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 generally in the range of 40 to 70 for most elements. However, somewhat low numbers of acceptable blends were noted for antimony (29), cadmium (26), mercury (26), selenium (25), and silver (24). RPDs between the mean concentrations reported by the ED2000 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 or RPD results used to calculate the median, for each target element. These statistics are provided for the demonstration as a whole, 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-1).

Accuracy was classified as follows for the target elements based on the overall median RPDs (for all demonstration blends and spikes):

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

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 (Appendix E).

Review of the median RPD values for the various media and concentration subpopulations in Table 7-3 indicates that RPDs were generally higher in soil than

in sediment for arsenic, cadmium, chromium, and silver. Therefore, the demonstration data set implies that the ED2000 attained a lower level of accuracy for these elements in soil. The only significant difference or trend noted in terms of sample concentration levels was a high relative median RPD of 49.4 percent observed for mercury in Level 2 concentration soil samples (200 to 1,000 ppm). Review of the data indicated that this value was biased by high RPDs for multiple blends from the Sulphur Bank mine site (Blends 23 through 26).

Section 5.3.3 indicated that reference laboratory data for antimony were consistently biased low when compared with the ERA-certified spike concentrations. This effect may be caused when antimony compounds used for spiking volatilize, resulting in loss of antimony during the sample digestion process at the reference laboratory. Therefore, Table 7-3 includes a second accuracy evaluation for antimony, comparing the ED2000 results with the ERA-certified values. As shown, this comparison indicates far better performance for antimony than does the comparison to the reference laboratory results; the overall median RPD using the ERA-certified values was 3.9 percent, compared with an overall median of 117 percent using the reference laboratory data. Compensating for potential laboratory bias, use of the ERA-certified values improves apparent XRF performance for antimony from “poor” to “very good.”

As an additional comparison, Table 7-3 presents the overall average of the median RPDs for all eight XRF instruments. Complete summary statistics for the RPDs across all eight XRF instruments are included in Appendix E (Table E-1). Table 7-3 indicates that the median RPDs for the ED2000 were equivalent to or lower than the all-instrument medians for the majority of the target elements. For mercury and nickel, the ED2000 median RPDs were significantly lower, strongly implying a higher level of accuracy. In contrast, higher median RPDs for the ED2000 relative to the all-instrument medians (indicating lower accuracy) were observed for antimony, arsenic, iron, and selenium.

In addition to calculating RPDs, the evaluation of accuracy included preparing linear correlation plots of ED2000 concentration values against the reference laboratory values. These plots are presented for the

Table 7-3. Evaluation of Accuracy — Relative Percent Differences Versus Reference Laboratory Data for the Oxford ED2000

Matrix	Sample Group	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc	
			Ref Lab	ERA Spike													
Soil	Level 1	Number	9	1	15	7	28	16	5	16	7	24	4	3	13	20	
		Median	118.3%	20.3%	55.2%	39.5%	40.5%	14.7%	175.1%	17.1%	4.6%	9.5%	30.9%	43.4%	41.4%	19.3%	
	Level 2	Number	5	1	4	7	4	7	13	4	7	5	5	3	4	6	
		Median	115.5%	26.0%	74.8%	28.8%	12.4%	29.6%	62.5%	13.1%	49.4%	10.5%	32.3%	39.3%	22.7%	24.3%	
	Level 3	Number	4	3	3	2	2	2	13	8	2	6	4	6	4	8	
		Median	100.4%	7.1%	103.0%	27.4%	30.7%	8.9%	61.0%	13.1%	19.7%	3.8%	27.7%	18.2%	27.8%	23.6%	
	Level 4	Number	--	--	--	--	--	--	7	4	--	--	--	--	--	--	
		Median	--	--	--	--	--	--	51.4%	23.8%	--	--	--	--	--	--	
	All Soil	Number	18	5	22	16	34	25	38	32	16	35	13	12	21	34	
		Median	114.9%	20.3%	68.7%	35.6%	39.7%	17.1%	62.3%	14.7%	20.9%	8.4%	31.5%	41.1%	31.6%	22.7%	
	Sediment	Level 1	Number	4	4	17	3	20	7	3	15	3	17	5	5	6	18
			Median	131.9%	2.9%	42.3%	10.8%	24.8%	15.3%	183.5%	16.1%	28.8%	19.7%	30.7%	11.7%	19.6%	22.2%
Level 2		Number	3	3	4	4	3	4	18	4	4	6	4	4	8	5	
		Median	147.6%	0.4%	12.0%	1.1%	6.2%	11.9%	90.3%	5.9%	22.1%	4.8%	27.3%	16.4%	32.9%	8.8%	
Level 3		Number	3	3	2	3	3	10	4	3	3	4	3	3	3	4	
		Median	98.7%	5.2%	19.9%	13.5%	5.4%	7.3%	84.1%	2.7%	23.1%	3.9%	31.4%	34.5%	18.0%	7.8%	
Level 4		Number	--	--	--	--	--	--	6	--	--	--	--	--	--	--	
		Median	--	--	--	--	--	--	65.9%	--	--	--	--	--	--	--	
All Sediment		Number	11	11	23	10	26	21	31	22	10	27	12	12	17	27	
		Median	131.5%	2.2%	37.3%	6.3%	17.9%	10.3%	88.7%	11.5%	23.3%	8.6%	31.0%	13.5%	27.2%	17.4%	
All Samples		ED2000	Number	29	16	45	26	60	46	69	54	26	62	25	24	38	61
			Median	117.7%	3.9%	49.7%	18.3%	24.8%	13.5%	78.1%	14.3%	23.3%	8.6%	31.4%	23.0%	30.1%	19.3%
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 RPDs presented in this table are absolute values.

-- 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.

individual target elements in Figures E-1 through E-13 of Appendix E. The plots include a 45-degree line that shows the “ideal” relationship between the ED2000 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 close to zero (that is, plus or minus the mean MDL in Table 7-1). Table 7-4 lists the results for these three correlation parameters and highlights in bold each target element that met all three accuracy criteria. This table shows that the results for mercury and nickel met all three of these criteria. The correlation plot for nickel is displayed in Figure 7-1 as an example of the correlations obtained for these elements.

Table 7-4. Summary of Correlation Evaluation for the ED2000

Target Element	m	b	r ²	Correlation	Bias
Antimony (vs. Reference Lab)	2.99	60	0.84	Moderate	High
Antimony (vs. ERA Certified Value)	0.90	100	0.79	Moderate	--
Arsenic	1.75	106	0.69	Moderate	High
Cadmium	0.93	-18	0.98	High	--
Chromium	0.83	50	0.89	Moderate	--
Copper	0.98	107	0.89	Moderate	--
Iron	1.91	8735 ¹	0.83	Moderate	High
Lead	1.06	-218 ¹	0.90	High	--
Mercury	0.88	-4	0.99	High	--
Nickel	1.02	21	0.99	High	--
Selenium	0.73	1	0.99	High	Low
Silver	0.95	-1	0.85	Moderate	--
Vanadium	1.12	18	0.93	High	--
Zinc	1.66	-271 ¹	0.85	Moderate	High

Notes:

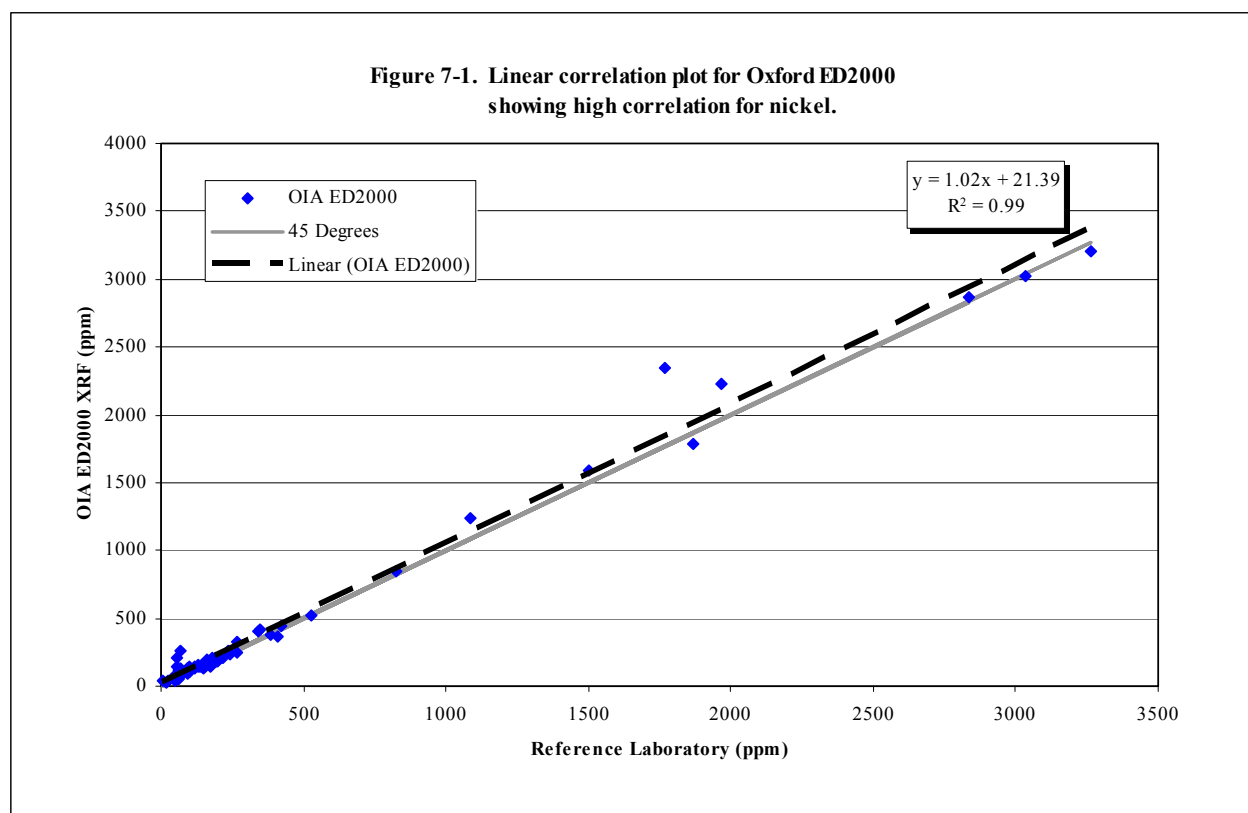
¹ For iron, no MDL was calculated and the high intercept value was the result of the extreme range of concentrations in the demonstration samples. To a lesser extent, high intercepts were also produced in the lead and zinc plots from the large concentration ranges in the demonstration samples.

-- No bias observed.

b Y-intercept of correlation line.

m Slope of correlation line.

r² Correlation coefficient of correlation line.



General observations from the correlation plots are as follows:

- The elements with a high relative degree of correlation between the ED2000 and the reference laboratory ($r^2 > 0.95$) included cadmium, lead, mercury, nickel, selenium, and vanadium. Correlations for four other elements (chromium, copper, silver, and zinc) also were fairly high, with r^2 values between 0.85 and 0.95. Further review of the data indicated that removal of lone high outliers from complex Blend 9 (Wickes Smelter slag) improve the r^2 values for copper, lead, and zinc to above 0.95. Thus, the linear correlation evaluation corresponds with the RPD evaluation in assessing the accuracy of the XRF instrument as “good” to “fair” for all these elements.
- Elements with low relative correlations (r^2 less than 0.85) included antimony, arsenic, and iron.

The plot for arsenic is presented in Figure 7-2 as an example of instrument performance for these three elements, showing a high overall level of scatter in the data (see Figures E-1 and E-6 for plots of antimony and iron). The correlation plots again confirm the findings of the RPD evaluation, which found high and variable median RPDs for these elements (Table 7-3).

- Figure E-1 shows a second correlation analysis was performed for antimony, comparing the mean ED2000 concentrations for spiked blends with the ERA-certified values rather than with the mean concentrations reported by the reference laboratory. However, as a result of a few outlier data points, this analysis did not improve the correlation coefficient for antimony, which remained near 0.8. Use of the certified values drastically improved the observed XRF bias for antimony, however, reducing the slope of the correlation line from 2.99 to near 0.9.

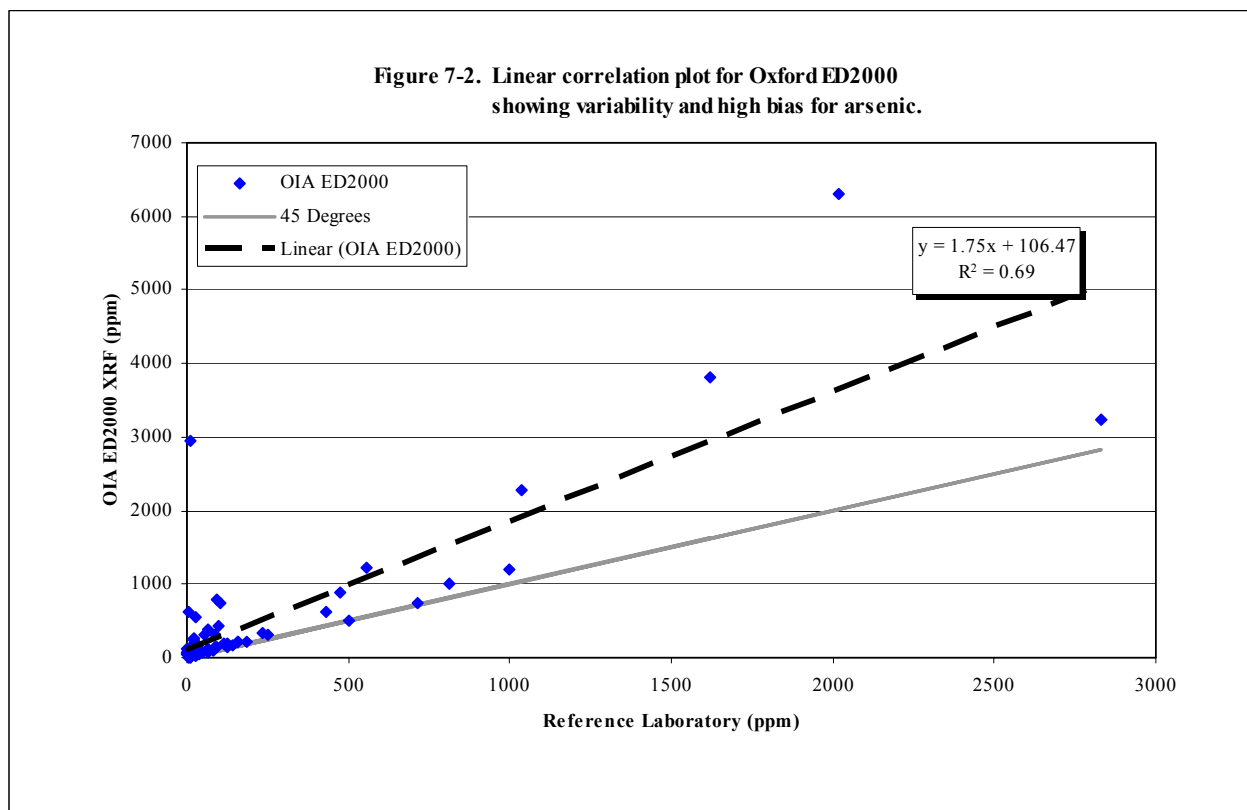
Further review of the correlation plots reveals a generalized bias in the XRF measurements for some elements versus the laboratory method. With a slope of 1.91 and a y-intercept of 8735 ppm, the best-fit correlation line for iron displayed the highest positive bias (Figure E-6). Similar positive biases (indicated by slopes of 1.6 or more) were apparent for arsenic and zinc. A positive bias is reasonable in XRF data for many elements, given that the XRF measures total element concentrations in the bulk soil. The laboratory methods, conversely, measure only the elements that can be extracted and solubilized from the soil by the digestion process. However, the plots for most of the other elements reveal only slight biases (both high and low). A somewhat low bias was observed for selenium with a slope of 0.73.

In conclusion, the evaluations of accuracy were similar to the MDL evaluation in Section 7.1 in showing an acceptable overall level of performance by the ED2000 for the target elements. Correlations with the reference laboratory were generally high, and median RPDs were better for most of the

elements than those obtained by the eight demonstration instruments combined. Factors such as the high-resolution detection system and program-specific calibration protocol (Chapter 6) may have contributed to the high relative level of accuracy attained by the ED2000. In contrast to vendor claims, however, the ED2000 appeared to offer no special advantage in performance for lighter elements such as vanadium, chromium, iron, or even arsenic.

7.3 Primary Objective 3 — Precision

As outlined in Section 4.2.3, precision of the ED2000 data set 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) are presented in Table 7-5. The table also presents the median RSDs for the demonstration data set as a whole for the ED2000. Additional summary statistics for the RSDs (including minimum, maximum, and mean) are provided in Appendix E (Table E-2).



The RSD calculation found a high level of precision for the ED2000 across all the target elements; the highest median RSD was only 12.5 percent (vanadium). The ranges into which the median RSDs fell are summarized below:

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

The median RSDs calculated for the soil and sediment subsets were also below 10 percent for all the elements except vanadium, where the median RSDs were in the 10 to 17 percent range. No significant differences were observed between the RSDs for soil and sediment. Use of the mean RSDs (Appendix E) as opposed to the median RSDs indicated a similarly high level of precision in the ED2000 results; mean RSDs were below 25 percent for essentially all elements and data subpopulations. The high overall level of precision may have been facilitated by the high level of processing (homogenizing, sieving, crushing, and drying) performed on the sample blends before the demonstration (Chapter 3). This observation is consistent with previous SITE MMT 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 MMT 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 greatest for lead, mercury, and vanadium, where the median RSDs increased to between 10 and 20 percent in Level 1 blends. This observation indicates that, to a minor extent, analytical precision for the ED2000 results is concentration-dependent.

As an additional comparison, Table 7-5 also presents the median RSDs calculated for all XRF instruments that were part of the demonstration. Additional summary statistics for the RSDs calculated across all XRF instruments are included in Appendix E. Table 7-5 indicates that the median RSDs for the ED2000 were equivalent to or below the all-instrument medians for all elements with the exception of lead and vanadium, where slightly higher median RSDs were observed. This observation indicates that the additional sample processing and pelletization steps Oxford performed during the demonstration may have slightly improved data precision overall.

Table 7-6 presents median RSD statistics for the reference laboratory for comparison to the ED2000 data and the results attained for all eight XRF instruments combined. (Additional summary statistics for the reference laboratory RSDs are provided in Appendix E, Table E-3.) The median RSDs attained by the ED2000 were lower than the reference laboratory RSDs for all target elements except vanadium. In comparison, the median RSDs for all XRF instruments combined 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 ED2000

Matrix	Sample Group	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	9	15	7	28	16	5	16	7	24	4	3	13	20
		Median	2.2%	4.7%	5.4%	5.7%	5.3%	0.7%	13.5%	14.8%	5.6%	4.0%	3.8%	17.2%	6.5%
	Level 2	Number	5	4	7	4	7	13	4	7	5	5	3	4	6
		Median	2.8%	1.7%	2.3%	5.4%	1.4%	1.4%	2.5%	4.0%	3.7%	3.2%	2.4%	3.7%	1.7%
	Level 3	Number	4	4	2	2	2	13	8	2	6	4	6	4	8
		Median	1.8%	0.9%	1.9%	1.3%	2.3%	3.1%	1.7%	3.6%	1.7%	2.0%	2.3%	5.2%	1.0%
	Level 4	Number	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	1.8%	23.2%	--	--	--	--	--	--
	All Soil	Number	18	23	16	34	25	38	33	16	35	13	12	21	34
		Median	2.6%	3.1%	2.9%	5.6%	4.2%	1.6%	8.4%	6.0%	4.7%	2.7%	2.5%	10.9%	3.7%
Sediment	Level 1	Number	4	17	3	21	8	3	16	3	18	5	5	6	19
		Median	4.9%	5.5%	2.6%	9.2%	4.8%	0.3%	8.4%	16.4%	4.6%	5.1%	4.2%	18.3%	10.3%
	Level 2	Number	4	4	4	3	4	19	4	4	6	4	4	8	5
		Median	1.6%	1.3%	2.2%	3.8%	1.7%	1.5%	1.9%	2.6%	3.0%	2.4%	1.7%	17.5%	1.7%
	Level 3	Number	3	2	3	3	10	4	3	3	4	3	3	3	4
		Median	3.6%	9.4%	3.1%	2.4%	1.4%	1.9%	0.4%	1.6%	2.8%	1.8%	2.9%	7.1%	1.6%
	Level 4	Number	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	2.1%	--	--	--	--	--	--	--
	All Sediment	Number	11	23	10	27	22	32	23	10	28	12	12	17	28
		Median	2.8%	5.0%	2.6%	7.3%	2.0%	1.5%	4.9%	2.9%	4.1%	3.2%	3.0%	16.5%	6.1%
All Samples	ED2000	Number	29	46	26	61	47	70	56	26	63	25	24	38	62
		Median	2.7%	4.0%	2.7%	5.6%	2.6%	1.5%	6.3%	4.7%	4.3%	2.8%	2.8%	12.5%	4.7%
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 ED2000 and All Demonstration Instruments

Matrix		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	ED2000	Number	29	46	26	61	47	70	56	26	63	25	24	38	62
		Median	2.7%	4.0%	2.7%	5.6%	2.6%	1.5%	6.3%	4.7%	4.3%	2.8%	2.8%	12.5%	4.7%
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:

Ref. Lab. Reference laboratory (Shealy Environmental Services, Inc.).

XRF X-ray fluorescence.

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 interferences are described in Section 4.2.4.

Interferent-to-element ratios were calculated using the mean concentrations the reference laboratory reported for each blend and are 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-4). The table indicates a clear increase in the median RPD for arsenic at the higher lead-to-arsenic ratios. Specifically, a median RPD of 35 percent at low interferent ratios increases to 75 percent at moderate ratios and further to 138 percent in the high interferent ratios. Using the criteria applied in Section 7.2, high concentrations of lead therefore diminish the accuracy of the ED2000 from “fair” to “poor” for arsenic. Similarly, Table 7-7 indicates that high concentrations of copper reduce instrument accuracy for nickel, although overall accuracy remains “good” for the high-

ratio blends (with a median RPD of 24 percent). In presenting statistics for unmodified RPDs as well as the absolute values of the RPDs, Appendix E further shows that the interferences by lead and copper tend to increase the positive bias of the results for arsenic and nickel (as indicated by more negative unmodified RPDs).

Table 7-7 and Appendix E reveal no other trends in RPDs that would indicate significant potential interferences. Although interference effects were limited for the ED2000, the data show significant potential effects of high lead concentrations on results for arsenic, despite the program-specific calibration of the instrument that was based on pre-demonstration samples.

7.5 Primary Objective 5 — Effects of Soil Characteristics

The population of RPDs between the results for the ED2000 and the reference laboratory were further evaluated against sampling site and soil type. Separate sets of summary statistics were developed for the median RPDs associated with each sampling site for comparison to the other sites and to the demonstration data set as a whole. The site-specific median RPDs are presented in Table 7-8, along with descriptions of soil and sediment type from observations during sampling at each site. Complete RPD summary statistics for each soil type (minimum, maximum, and mean) are presented in Appendix E (Table E-5).

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 extreme values with sample types or locations for multiple elements across the data set. Some 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 ED2000¹

Parameter	Lead Effects on Arsenic			Copper Effects on Nickel			Nickel Effects on Copper			Zinc Effects on Copper			Copper Effects on Zinc		
	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10
Interferent/ Element Ratio															
Number of Samples	29	6	9	43	5	14	37	1	8	34	1	11	48	3	10
Median RPD of Target Element ²	34.7%	74.8%	138.3%	7.2%	8.5%	23.6%	13.5%	16.1%	12.6%	12.4%	11.3%	15.9%	21.0%	20.9%	17.3%
Median Interferent Concentration	76	5087	2239	141	1160	2409	156	378	2284	207	889	3940	179	1259	2329
Median Target Element Concentration	148	1057	373	191	156	149	1006	92	121	1093	179	135	619	146	177

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 ED2000

Matrix	Site	Matrix Description	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead
Soil	AS	Fine to medium sand (steel processing)	Number	--	1	3	2	3	3	--
			Median	--	182.8%	39.5%	70.8%	16.2%	42.1%	--
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	7	5	7	6	7	1
			Median	120.7%	74.5%	35.2%	22.9%	24.5%	59.5%	0.0%
Soil	CN	Sandy loam (burn pit residue)	Number	2	1	2	2	3	3	2
			Median	88.7%	49.7%	30.5%	63.8%	21.9%	60.8%	9.7%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	2	--	--	4	2	6	--
			Median	8.6%	--	--	64.4%	21.6%	179.6%	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	11	5	11	4	12	4
			Median	107.7%	38.7%	7.4%	14.8%	9.1%	87.6%	1.3%
Sediment	RF	Silty fine sand (tailings)	Number	5	12	5	12	13	13	5
			Median	130.9%	37.0%	4.6%	11.8%	13.6%	86.1%	23.5%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	7	5	1	11	4	12	11
			Median	115.5%	32.7%	0.7%	41.6%	10.3%	60.8%	28.9%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	2	2	5	7	7	3
			Median	150.9%	123.2%	12.4%	23.2%	7.6%	98.4%	87.7%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	2	6	2	6	5	7	--
			Median	122.4%	114.0%	51.1%	15.9%	37.5%	78.1%	--
	All		Number	29	45	25	60	47	70	26
			Median	117.7%	49.7%	16.8%	24.8%	13.6%	78.4%	23.3%

Table 7-8. Effect of Soil Type on the RPDs (Accuracy) for Target Elements, Oxford ED2000 (Continued)

Matrix	Site	Matrix Description	Statistic	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	AS	Fine to medium sand (steel processing)	Number	--	3	1	1	1	3
			Median	--	5.2%	32.3%	17.3%	27.2%	12.4%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	1	6	4	4	4	7
			Median	0.0%	12.3%	30.9%	41.1%	27.5%	23.2%
Soil	CN	Sandy loam (burn pit residue)	Number	2	3	2	2	1	3
			Median	9.7%	10.5%	29.4%	36.2%	3.7%	22.6%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	3	--	--	--	2
			Median	--	4.4%	--	--	--	27.1%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	11	5	4	9	10
			Median	1.3%	19.5%	28.2%	14.2%	30.0%	34.6%
Sediment	RF	Silty fine sand (tailings)	Number	5	13	5	5	3	13
			Median	23.5%	5.4%	31.4%	11.7%	35.7%	13.8%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	11	11	3	1	10	11
			Median	28.9%	4.0%	35.7%	65.4%	51.6%	16.9%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	6	4	4	7	7
			Median	87.7%	28.5%	25.8%	23.0%	12.0%	17.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	7	1	3	3	6
			Median	--	19.1%	28.9%	54.6%	36.4%	20.9%
	All		Number	26	63	25	24	38	62
			Median	23.3%	8.6%	31.4%	23.0%	30.1%	19.3%

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 reveals few extremes in performance of the XRF instrument based on sample matrix. Although elevated RPDs were observed in some blends relative to the remainder of the data sets for the target elements, few extremes were observed that would indicate clear matrix effects. Moreover, no specific sample sites appear to correlate with abnormally good or poor performance across many or all of the target elements. Instead, elevated median RPDs for some target elements were observed on a more limited basis in specific site blends. For example, cadmium, copper, and silver displayed elevated median RPDs in blends from the Wickes Smelter site, whereas antimony, mercury, and nickel displayed similar RPDs in blends from the Torch Lake site. Iron exhibited an extreme median RPD of 179.6 percent in KARS Park blends, while vanadium displayed a high relative median RPD of 51.6 percent in blends from the Sulphur Bank Mercury Mine. In some cases, the identification of a potential matrix affect based on elevated or extreme RPDs was limited by low sample numbers. For example, a high RPD of 182.8 percent was observed for arsenic in an Alton Steel site blend, but only a single sample from this site had been assessed as acceptable for accuracy evaluation (using the approach described in Section 4.2.2). Thus, the evaluation of matrix affects based on median RPD was complex and showed no clear or general trends.

Review of the box and whiskers plot (Figure E-14) and the correlation plots from the accuracy evaluation revealed no other general trends in RPDs relative to sampling site. The outliers and extreme values apparent in Figure E-14 were broadly distributed among seven of the nine sampling sites. This evaluation verified the slight prevalence of outliers in the KARS Park blends (for iron) and in Wickes Smelter blends (for multiple metals). As discussed in Section 2.3, the KARS Park site was contaminated by former gun range operations. Soil samples from this site consisted of fine to medium quartz sand with anticipated contamination from antimony, arsenic, chromium, copper, lead, and zinc. In comparison, the soil matrix from the Wickes Smelter site was described as roaster slag, consisting of a black, fairly coarse sand and gravel material that again contained high concentrations of multiple target elements.

A smaller number of outlier blends apparent on Figure E-14 were associated with the Leviathan Mine site. However, sample matrix appeared to have a

minor effect on the overall accuracy of the XRF data. The box and whiskers plot in Figure E-14 shows that the broad overall distributions of RPDs for many elements, such that relatively few high outliers or extreme values could be identified. The distributions of RPDs were sufficiently broad that no high outliers or extreme values were discernable for antimony, arsenic, and vanadium.

7.6 Primary Objective 6 — Sample Throughput

The Oxford two-person field team was able to analyze all 326 demonstration samples in 5 days at the demonstration site. Once the ED2000 instrument had been set up and operations had been streamlined, the Oxford field team was able to analyze a maximum of 123 samples during an extended work day. This sample throughput was achieved by using different members of the field team to perform sample preparation and instrumental analysis and by using the autosampler to process samples through the XRF spectrometer. Without an extended work day, and taking into account instrument set-up and demobilization time, it was estimated that the Oxford field team would have averaged about 56 samples per day.

This estimated sample throughput for a normal working day was lower than that observed for the other instruments that participated in the demonstration (average of 66 samples per day). The lower sample throughput was primarily the result of the unique sample preparation process employed, which involved pelletizing each sample prior to instrumental analysis. If a powdered sample would have been used instead, the sample throughput would have increased.

A detailed discussion of the time required to complete the various steps of sample analysis using the ED2000 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 fully described in Chapter 8, Economic Analysis.

7.8 Secondary Objective 1 — Training Requirements

The instrument operator must be suitably trained to safely set up and operate the instrument to successfully use XRF and obtain the level of data quality required for specific projects. The amount of training required depends on the complexity of the instrument and the associated software.

Oxford recommends that the operator have a high school diploma and basic operational training. Field or laboratory technicians are generally qualified to operate the instrument. The ED2000 comes equipped with a modem that allows qualified Oxford technicians to evaluate system functionality and to provide troubleshooting guidance from a remote location. During the demonstration, the Oxford staff members who operated the instrument held Ph.D. degrees in chemistry, with about 5 years of experience in operation of the ED2000.

Oxford provides free on-site training for all purchasers of the instrument. Topics vary based on the end users' intended applications. The training generally lasts 3 days; however, the training period can be extended for very complex applications.

In addition to the general instrument operational instruction and training, the operator and data manager must be familiar with using a windows-based personal computer (PC) to operate the ED2000 software (Xpert Ease).

7.9 Secondary Objective 2 — Health and Safety

The health and safety requirements for operation of the instrument were identified. Included in the evaluation were the potential risks from exposure to radiation and to reagents. However, not included in the evaluation were potential risks from exposure to site-specific hazardous materials or to physical safety hazards.

Two potential areas for operator risks were evaluated: (1) radiation from the instrument itself, and (2) exposure to any reagents used in preparing and analyzing the samples. As mentioned above, any potential risks from sample contaminants were not addressed, simply because of the wide range in site conditions where the instrument may be used.

The ED2000 contains an x-ray tube that is positioned to deliver x-rays into a lead-shielded, sealed sample chamber. Each instrument is equipped with a sample chamber lock, and large lights indicate when x-rays are being generated. The instrument will not operate if the lock is not latched or if the lights are burned out. The sample chamber lock, lead-shielded sample chamber, and safety lights are designed to minimize possible exposure to the x-ray radiation.

The second potential source of risk to XRF instrument operators is exposure to reagent chemicals. The two reagents used during the demonstration are wax (for sample pellet preparation) and liquid nitrogen (for cooling the detector). The wax is not hazardous; however, care must be taken when filling the detector's 10-liter reservoir (Dewar) with liquid nitrogen because of its extremely low temperature (-196°C or -321°F). The instrument loses approximately 1 liter per day, requiring the Dewar to be refilled every 10 days. In addition, the instrument will not operate without enough liquid nitrogen to cool the detectors. The risks from exposure to radiation or to liquid nitrogen are minimal when the instrument is operated according to the manufacturer's recommendations, however.

7.10 Secondary Objective 3 — Portability

Portability depends on the size, weight, number of components, and power requirements of the instrument, and the reagent 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 were also recorded. 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.
- (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 the dimensions and power requirements, the ED2000 is defined as transportable. It is capable of being transported to a field trailer or other fixed location with the required power supply and a stable, weatherproof environment.

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) Oxford offers a 12-month limited warranty on parts and labor. Additional warranties, optional extended warranties, and service contracts vary by country. 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 the ED2000 is approximately 10,000 hours, which is equivalent to approximately 7 years.

Oxford is continually upgrading both the instrument and software to enhance environmental analysis. It is expected that Oxford will continue to provide upgrades to instruments and software as long as there is a market for improved technologies.

The ED2000 instrument is made with hard-tool plastic that is durable and impact-resistant under nearly all field applications. The instrument is not weatherproof and must be located in a stable, weatherproof environment.

7.12 Secondary Objective 5 — Availability

Oxford Instruments Analytical has offices worldwide. New instruments are available from the Oxford offices in Concord, Massachusetts, and Elk Grove Village, Illinois. Oxford provides product support for all instruments through service contracts tailored to the client's needs. A network of 30 service representatives provide service and customer support for instrument owners.

The ED2000 is available for lease or for long-term rental on a case-specific basis. The ED2000 is not available from third party vendors for lease or rental.

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Chapter 8 Economic Analysis

This chapter provides cost information for the Oxford ED2000 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 ED2000.

8.1 Equipment Costs

Capital equipment costs include either purchase or rental of the ED2000 and any ancillary equipment that is generally needed for sample analysis. (See Chapter 6 for a description of available accessories.) Information on purchase price for the analyzer and accessories was obtained from Oxford.

The ED2000 instrument costs between \$65,000 and \$85,000, depending on the configuration. The cost of the unit used for the XRF demonstration is approximately \$80,000, including peripherals (autosampler, sample crusher and mixer, sample press, computer, monitor, and printer). At the time of the demonstration, Oxford indicated that models are not available for rental. For evaluation and comparison purposes later in this Chapter, an estimated rental cost was derived based on similar XRF technologies where both purchase and rental prices were available. Long-term lease programs are also available through Oxford. Purchased models include a 1-year parts and labor warranty; this warranty may be extended for \$9,000 per year. The lifespan of the x-ray tube is about 5 to 7 years for normal usage.

The purchase price and shipping cost for the Oxford ED2000 exceed the average costs for all XRF instruments that participated in the demonstration, as shown in Table 8-1.

Table 8-1. Equipment Costs

Cost Element	ED2000	XRF Demonstration Average ¹
Shipping	\$750	\$410
Capital Cost (Purchase)	\$80,000	\$54,300
Weekly Rental (estimated)	\$3,700	\$2,813
Autosampler (for Overnight Analysis)	Included	N/A

Notes:

¹ Average for all eight instruments in the demonstration

N/A Not available or not applicable for this comparison

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. Additional costs include purchase of liquid nitrogen for detector cooling and rental or purchase of sample preparation equipment, if required for the intended use.

The ED2000 was operated for 4 days to complete the analysis of the demonstration sample set (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 \$250 for 326 samples or \$0.75 per sample.

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 estimated time to complete each of these activities using the ED2000 is listed in Table 8-2. 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 all sample analysis using the ED2000 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. In

comparison to other XRF analyzers, the ED2000 exhibited higher-than-average times for initial setup and calibration, sample preparation and analysis, and end of project packing. The ED2000 exhibited a lower-than-average time for daily shutdown and startup because the instrument was not shut down each night.

Sample preparation included pressing samples into pellets to maximize data quality. However, the ED2000 does not require pelletized samples and can accommodate powdered samples in polyethylene cups, covered with Mylar[®] film, for analysis. (The majority of the technology developers in this demonstration used powdered samples.) The sample preparation time could have been reduced from 7 minutes to 2 minutes by changing to the powdered sample approach.

Table 8-2. Time Required to Complete Analytical Activities¹

Activity	ED2000	Average ²
Initial Setup and Calibration	85	54
Sample Preparation	7.0	3.1
Sample Analysis	7.9	6.7
Daily Shutdown/Startup	0	10
End of Project Packing	115	43
Total Processing Time per Sample	15.5	10.0

Notes:

¹ All estimates are in minutes

² Average for all eight XRF instruments in the demonstration

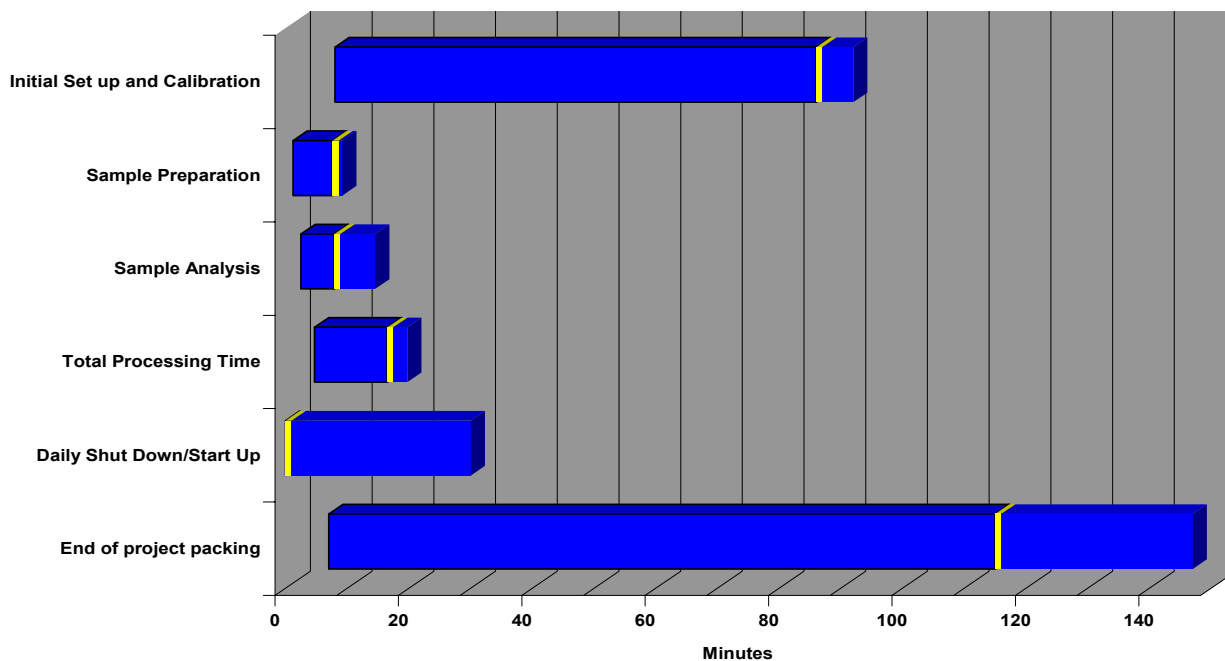


Figure 8-1. Comparison of activity times for the Oxford ED2000 versus other XRF instruments.

The Oxford field team expended about 93 labor hours to complete all sample processing activities during the field demonstration using the ED2000. This was significantly higher than the overall average of 69 labor hours for all instruments that participated in the demonstration. The primary reasons that labor hours were higher for the ED2000 include:

- The unique sample preparation process employed, which involved pelletizing each sample prior to instrumental analysis. If a powdered sample would have been used instead, the labor hours would have decreased.
- The additional time required to set up the multiple components of the ED2000 system and to maintain these components during sample processing.

8.4 Comparison of XRF Analysis and Reference Laboratory Costs

Two scenarios were evaluated to compare the cost for XRF analysis using the ED2000 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 ED2000 was based on equipment rental for 1 week, along with labor and supplies estimates established during the

field demonstration. The estimate used for rental of the ED2000 was a hypothetical weekly rental rate based on a survey of rental versus purchase costs of other XRF instruments. Labor costs were estimated based on the number of people in the field team and the time spent during the field demonstration to complete the analysis of the 326 demonstration samples. 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. 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 ED2000 analysis cost was not adjusted for one element versus 13 elements.

Table 8-3 summarizes the costs for the ED2000 versus the cost for analysis in a fixed laboratory. This comparison shows that the ED2000 compares favorably to a fixed laboratory in terms of overall cost when a large number of elements are to be determined. The ED2000 compares unfavorably to a fixed laboratory when one element is to be determined. Use of the ED2000 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 ED2000 in the example scenario (326 samples) was estimated at \$11,645. This estimate compares with the average of \$8,932 for all XRF instruments that participated in the demonstration. However, it should be noted that bench-top instruments, such as the ED2000, are known to cost more than the hand-held instruments that were included in the average cost for all XRF instruments. In comparison to other bench-top XRF instruments, the cost of the ED2000 for the example scenario was similar.

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

Analytical Approach	Quantity	Item	Unit Rate	Total
ED2000 (1 to 13 elements)				
Shipping	1	Roundtrip	\$750	\$750
Weekly Rental	1	Week	\$3,700 ¹	\$4,100
Supplies	326	Sample	\$0.75	\$245
Labor	148	Hours	\$43.8	\$6,460
IDW	N/A	Each	N/A	\$90
Total ED2000 Analysis Cost (1 to 13 elements)				\$11,645
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

Notes:

¹ Estimated value as Oxford currently does not have a rental rate for the ED2000.

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 ED2000 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. The vendor performed additional processing during the demonstration to homogenize and press the samples into uniform pellets. 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 ED2000 for each primary and secondary objective are summarized in Tables 9-1 and 9-2. The ED2000 and the combined performance of all eight vendors that participated in the XRF technology evaluation program are compared in Figure 9-1. The comparison in Figure 9-1 indicates that, when compared with the program as a whole, the ED2000 showed:

- Equivalent or better MDLs for all 12 of the target elements evaluated (iron was not included in the MDL evaluation).
- Equivalent or better accuracy (RPDs) for 9 of the 13 target elements (exceptions include antimony, arsenic, iron, and selenium). 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 program as whole.
- Equivalent or better precision (RSDs) for 11 of the 13 target elements (exceptions include lead and vanadium).

Factors that may have contributed to high relative level of performance include: (1) samples were processed into uniform pellets before they were analyzed, (2) a program-specific instrument calibration was generated using pre-demonstration samples, (3) the sample analysis chamber was evacuated to limit x-ray scattering by air, (4) a high-resolution, cryogenically cooled detector was used, and (5) the overall stability that is expected of a bench-top XRF. As a bench-top instrument, however, the ED2000 is not fully portable and requires a stable operating environment.

Table 9-1. Summary of Oxford ED2000 Performance – Primary Objectives

Objective	Performance Summary
P1: Method Detection Limits	<ul style="list-style-type: none"> • Mean MDLs for the target elements ranged as follows: <ul style="list-style-type: none"> ○ MDLs of 1 to 20 ppm: antimony, cadmium, copper, selenium, silver, and vanadium. ○ MDLs of 20 to 50 ppm: arsenic, chromium, lead, mercury, nickel, and zinc. ○ MDLs greater than 50 ppm: none. (Iron was not included in the MDL evaluation.) • The MDL calculation for mercury was based on limited data (two MDL sample blends). • No significant differences were noted between MDLs for soil and sediment, or among different sample blends. • The MDLs calculated were significantly lower than reference MDL data from EPA Method 6200.
P2: Accuracy and Comparability	<ul style="list-style-type: none"> • Median RPDs between the ED2000 and reference laboratory data revealed the following, with lower RPDs indicating greater accuracy: <ul style="list-style-type: none"> ○ RPDs of 1 to 10 percent: nickel. ○ RPDs of 10 to 25 percent: cadmium, chromium, copper, lead, mercury, silver, and zinc. ○ RPDs of 25 to 50 percent: arsenic, selenium, and vanadium. ○ RPDs of greater than 50 percent: antimony and iron. • 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. RPDs for antimony were high when the ED2000 data were compared with the reference laboratory data (with a median RPD of 117 percent) but improved considerably when compared with certified spike values (where the median RPD was 3.9 percent). Thus, the ED2000 appeared to be more accurate in terms of the true concentration of antimony than the reference laboratory. • Higher RPDs (that is, lower accuracy) were observed in soil than in sediment for arsenic, cadmium, chromium, and silver. • Correlation plots relative to reference laboratory data indicated: <ul style="list-style-type: none"> ○ Moderate to high correlations for all target elements. ○ Positive biases for arsenic, iron, and zinc. ○ Negative bias for selenium.
P3: Precision	<ul style="list-style-type: none"> • Median RSDs were good for all target elements, as follows: <ul style="list-style-type: none"> ○ RSDs below 5 percent: antimony, arsenic, cadmium, copper, iron, mercury, nickel, selenium, silver, and zinc. ○ RSDs between 5 and 10 percent: chromium and lead. ○ The RSD between 10 and 20 percent: vanadium. • Median RSDs for the ED2000 for all elements except vanadium were lower than the RSDs calculated for the reference laboratory data, indicating slightly better precision for the XRF instrument.
P4: Effects of Sample Interferences	<ul style="list-style-type: none"> • High relative concentrations of lead reduced accuracy for arsenic; median RPDs for arsenic increased from 35 percent to 138 percent as the concentration of lead increased. The lead interference produced a positive bias in the arsenic results. • High relative concentrations of copper (more than 10 times) slightly reduced accuracy for nickel; the median RPDs increased from 7 percent to 24 percent. A positive bias in the nickel results was produced by the copper interference.

Table 9-1. Summary of Oxford ED2000 Performance – Primary Objectives (Continued)

Objective	Performance Summary
P5: Effects of Soil Type	<ul style="list-style-type: none"> • 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. • High RPD outliers were also observed for copper, lead, nickel, selenium, and zinc in blends from the Wickes Smelter site, a complex roaster slag matrix that contained high concentrations of many elements.
P6: Sample Throughput	<ul style="list-style-type: none"> • Oxford’s sample preparation protocol during the demonstration included palletizing each sample prior to analysis and took an average of 7 minutes per sample. • With an average instrument analysis time of 7.9 minutes per sample, the total sample processing time was 15.5 minutes per sample. • A maximum sample throughput of 123 samples was achieved during the field demonstration on one extended work day. A typical average sample throughput was estimated to be 53 samples per day for an 8-hour work day.
P7: Costs	<ul style="list-style-type: none"> • The purchase cost was \$80,000 for the ED2000 as used in the demonstration. This cost included an optional autosampler and processing equipment to create sample pellets. Although long-term leases are available, the vendor does not currently offer short-term rental. • The Oxford field team expended approximately 93 labor hours to complete the processing of the demonstration sample set (326 samples). In comparison, the average for all participating XRF instruments was 69 labor hours. By approximating a 1-week rental cost (based on similar XRF instruments) and adding labor and shipping/supplies costs, a total project cost of \$11,645 was estimated for a project the size of the demonstration using the ED2000. In comparison, the average project cost for all participating XRF instruments was \$8,932 and the cost for fixed-laboratory analysis of all 13 elements was \$63,896.

Table 9-2. Summary of Oxford ED2000 Performance – Secondary Objectives

Objective	Performance Summary
S1: Training Requirements	<ul style="list-style-type: none"> • Field or laboratory technicians with a high school diploma and basic operational training are generally qualified to operate the ED2000. • Oxford offers free training for instrument purchasers that generally lasts about 3 days. • The ED2000 comes equipped with a modem that allows qualified technicians to remotely troubleshoot the instrument and guide operators.
S2: Health and Safety	<ul style="list-style-type: none"> • The ED2000 is equipped with safety measures to minimize possible exposure to emissions from the x-ray tube. The instrument cannot be operated if these safety measures are disabled. • Users of the ED2000 must be able to safely manage and dispense cryogenic liquids (nitrogen) to operate the detector.
S3: Portability	<ul style="list-style-type: none"> • Based on dimensions, weight, and power requirements, the ED2000 is a transportable (as opposed to fully portable) instrument. It is best used in a field trailer or other fixed location with the required power supply and a stable, weatherproof environment.
S4: Durability	<ul style="list-style-type: none"> • The ED2000 instruments have a 12-month limited warranty for parts and labor. Additional optional warranties and service contracts are available, depending on the country where the instrument is purchased and used. • The average lifespan of an x-ray tube in the ED2000 is anticipated to be 10,000 hours (7 years) • The ED2000 is encased in durable hard-tool plastic but is not weatherproof. It must be used in a stable environment.
S5: Availability	<ul style="list-style-type: none"> • New instruments are available from the Oxford offices in Concord, Massachusetts, and Oak Park, Illinois. A world-wide network of 30 service representatives provides service and customer support. • The ED2000 is available for lease or for long-term rental on a case-specific basis. It is not available from third-party vendors.

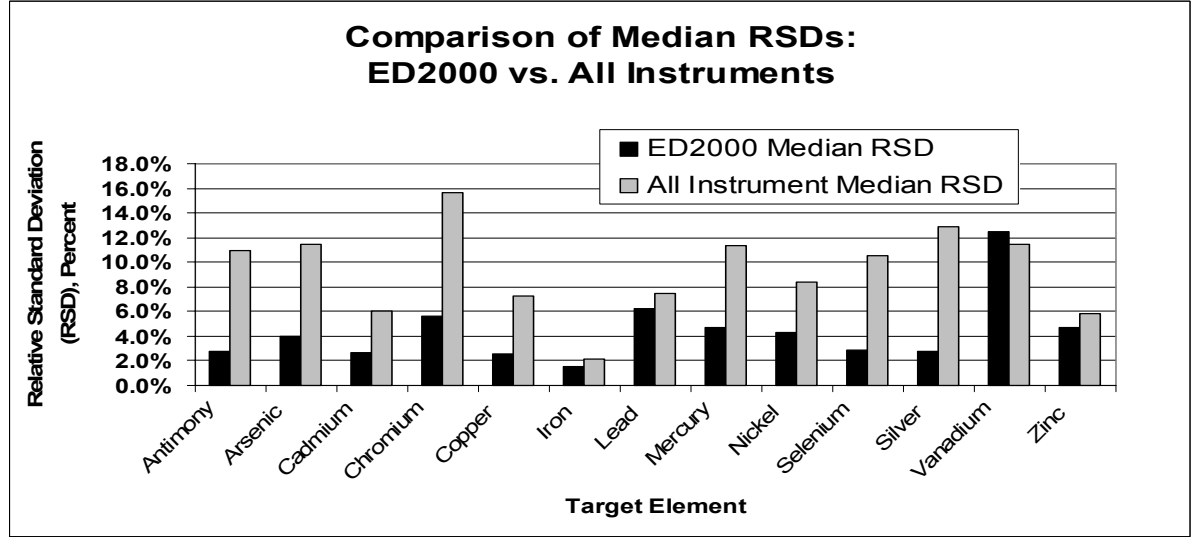
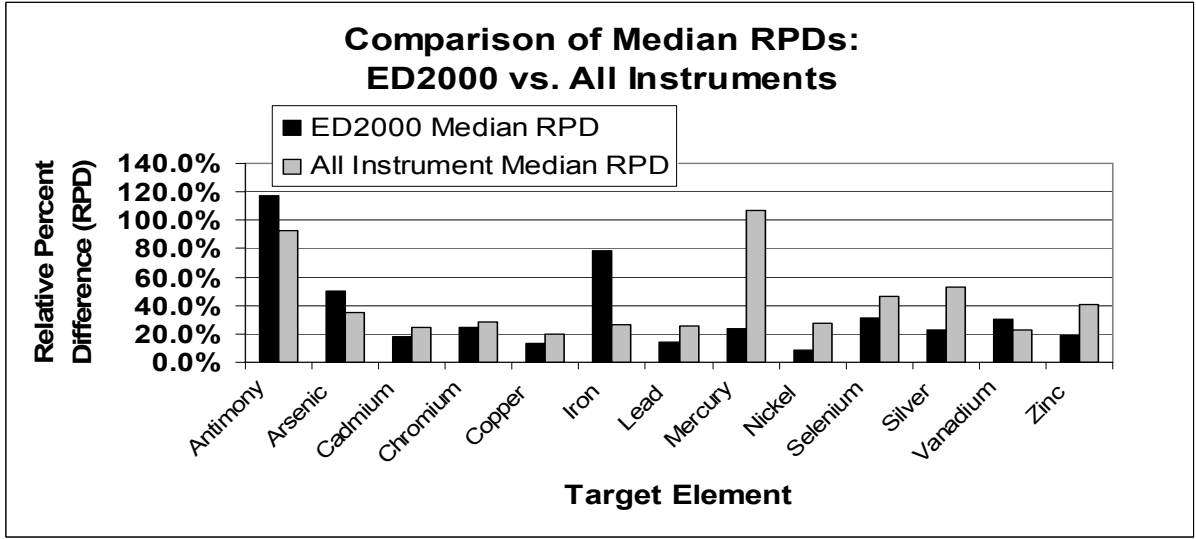
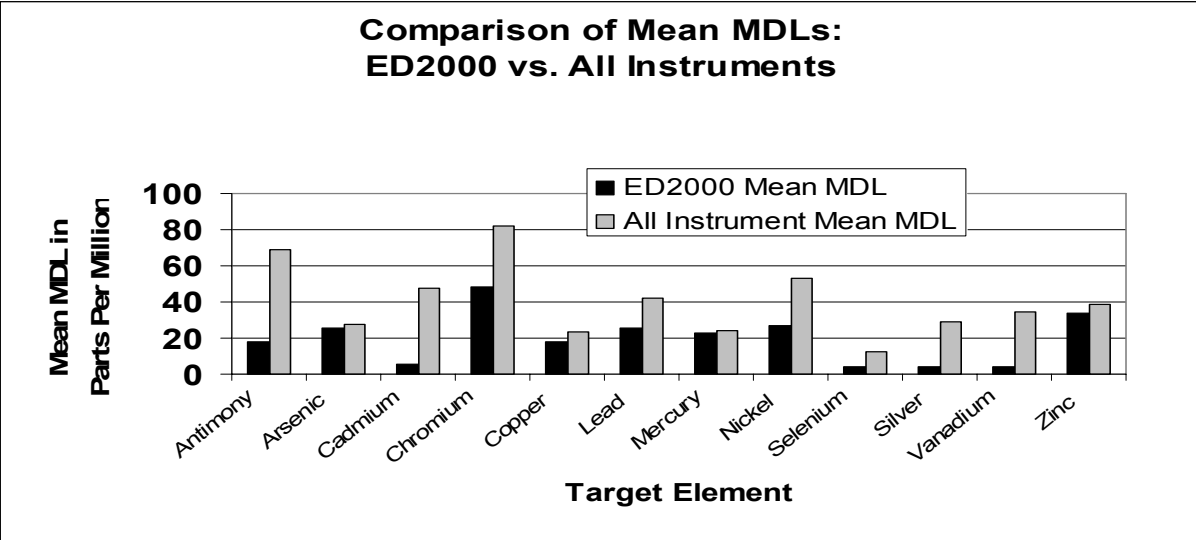


Figure 9-1. Method detection limits (sensitivity), accuracy, and precision of the ED2000 in comparison to the average of all eight XRF instruments.

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Chapter 10 References

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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:	ED2000 XRF Analyzer
COMPANY:	Oxford Instruments Analytical
ADDRESS:	945 Busse Road Elk Grove Village, IL 60007
PHONE:	1-800-678-1117
WEB SITE:	www.oxford-instruments.com
E-MAIL:	sales@msys.oxinst.com

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 (Oxford) ED2000 bench-top 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 technologies to measure 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 technology developer, including Oxford, 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 Oxford ED2000 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 ED2000 XRF Analyzer (EPA/540/R-06/007).

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 Oxford ED2000 XRF analyzer is an energy-dispersive XRF analyzer that can be operated as a bench-top unit in a mobile or on-site laboratory. The ED2000 can analyze up to 75 elements in a variety of sample matrices, including contaminated soils and sediments, liquids, powders, granules, filter papers, or films. Light-end elements (sodium to iron) can be measured when the samples are prepared as pressed pellets. Samples were pressed into pellets for this demonstration; however, this step is not required for routine analysis of soil or sediment samples.

The Oxford ED2000 analyzer system includes a SMART digital pulse processor to handle count rates as high as 90,000 counts per second (CPS). The high count rates and high detector resolutions provide improved precision and lower detection levels when compared with older Oxford XRF analyzers. Oxford also provides a calibration service as an option to customers using this analyzer. Special features of the Oxford ED2000 include a 16-position automatic sample tray, a 10 liter Dewar to hold liquid nitrogen to cool the detector, a vacuum pump to evacuate the sample chamber of the XRF to reduce formation of oxides in the sample matrix, and a personal computer loaded with Oxford’s instrument calibration and XpertEase 32 software for automated data processing.

VERIFICATION OF PERFORMANCE

Method Detection Limit: MDLs were calculated using seven replicate analyses from each of 12 low-concentration 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 ED2000 are summarized below (lower MDL values indicate higher sensitivity).

Relative Sensitivity	Mean MDL	Target Elements
High	1 - 20 ppm	Antimony, Cadmium, Copper, Selenium, Silver, and Vanadium.
Moderate	20 – 50 ppm	Arsenic, Chromium, Lead, Mercury, Nickel, and Zinc.
Low	50 – 100 ppm	None.
Very Low	> 100 ppm	None.

Notes: 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 ED2000 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%	Nickel.
Moderate	10% - 25%	Cadmium, Chromium, Copper, Lead, Mercury, Silver, and Zinc.
Low	25% - 50%	Arsenic, Selenium, and Vanadium.
Very Low	> 50%	Antimony* and Iron.

* Calculation of RPDs versus sample spike concentrations rather than reference laboratory results (due to potential low bias in the reference laboratory results for antimony) improves accuracy from Very Low to High.

Accuracy was also assessed through correlation plots between the mean ED2000 and mean reference laboratory concentrations for the various sample blends. Correlation coefficients (r2) 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	0.84	0.69	0.98	0.89	0.89	0.83	0.90	0.99	0.99	0.99	0.85	0.93	0.85
Bias	High	High	--	--	--	High	--	--	--	Low	--	--	High

Notes: -- = No significant bias. * Correlation is 0.79 with no observed bias when assessed versus sample spike concentrations.

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 ED2000 was classified from high to very low for the target elements, as indicated in the table below, based on the overall median RSDs. These RSDs indicated a higher level of precision in the ED2000 than in the reference laboratory data for all target elements except vanadium.

Relative Precision	Median RSD	Target Elements
High	0% - 5%	Antimony, Arsenic, Cadmium, Copper, Iron, Mercury, Nickel, Selenium, Silver, and Zinc
Moderate	5% - 10%	Chromium and Lead.
Low	10% - 20%	Vanadium.
Very Low	> 20%	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. This evaluation found that accuracy for arsenic was reduced from “low” (median RPDs between 25 and 50 percent) to “very low” (median RPDs greater than 50 percent) by high relative concentrations of lead (greater than 10X the arsenic concentration). An existing high bias in the arsenic results was increased by the interference. A more minor but similar effect was observed for copper as an interferent for nickel.

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 outlier RPD values indicating low relative accuracy for iron in blends of sandy soil from the KARS Park site, a former gun range. High RPD outliers were also observed for multiple elements in blends from the Wickes Smelter site, a complex roaster slag matrix.

Sample Throughput: The total processing time per sample was estimated at 15.5 minutes, which included 7.0 minutes of sample preparation and 7.9 minutes of instrument analysis time. On this basis, a sample throughput of 53 samples per 8-hour work day was estimated with the use of the instrument's autosampler. As noted above, however, the sample blends had undergone rigorous pre-processing before the demonstration. Sample throughput would have decreased if these sample preparation steps (grinding, drying, sieving) had been performed during the demonstration; these steps can add from 10 minutes to 2 hours to the sample processing time.

Costs: A cost assessment for the ED2000 identified a purchase cost of \$80,000, plus \$750 shipping, as equipped for the demonstration. Using a hypothetical rental cost approximated from similar types of instruments, a total cost of \$11,645 (with a labor cost of \$6,460 at \$43.75/hr) was estimated for a project similar to the demonstration (326 samples of soil and sediment). In comparison, the project cost averaged \$8,932 for all eight XRF instruments participating in the demonstration and \$63,896 for fixed-laboratory analysis of all 13 target elements.

Skills and training required: Field or laboratory technicians with a high school diploma are generally qualified to operate the ED2000. Oxford offers free training for instrument purchasers that generally lasts about 3 days, and the instrument is equipped with a modem for remote troubleshooting and guidance.

Health and Safety Aspects: The ED2000 is equipped with safety measures to minimize possible exposure to emissions from the x-ray tube. The instrument cannot be operated if these safety measures are disabled. Users of the ED2000 must be able to safely manage and dispense cryogenic liquids (nitrogen) to operate the detector.

Portability: Based on dimensions, weight, and power requirements, the ED2000 is a transportable (as opposed to fully portable) instrument. It is best used in a field trailer or other fixed location with the required power supply and a stable, weatherproof environment.

Durability: The ED2000 is encased in durable hard-tool plastic but is not weatherproof. Oxford instruments have a 12-month limited warranty for parts and labor. The developer estimates that the average lifespan of the x-ray tube source is 10,000 hours or 7 years.

Availability: New instruments are available from the Oxford offices in Concord, Massachusetts, and Oak Park, Illinois. A world-wide network of 30 service representatives provides service and customer support. Although long-term leasing is possible, instruments are not currently available for rental.

RELATIVE PERFORMANCE

The overall performance of the ED2000 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	●	●	●	NC	●	Same	●	●	●	●	●
Accuracy	○	○	Same	Same	Same	○	●	●	●	○	●	●	Same
Precision	●	●	●	●	●	●	○	●	●	●	●	○	●

Key: ● Better ○ Worse NC No MDL Calculated.

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 the 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 instrumentation for the investigation of heavy metal contamination in soil.

The ED2000 is a field transportable EDXRF instrument based on a high resolution detector which is designed for operation in almost any environment. As such, this analyzer can be used either for direct screening of samples at the site in a RV/mobile lab or it may be set up in a tent or make shift field laboratory. For this evaluation, the instrument was set up in a field laboratory. Under normal operating conditions, such as a mobile laboratory, the instrument can be operational in 10 minutes. If the instrument is shipped on site in different components, then unpacking and assembly can be done within two hours and the instrument made operational in less than 2 ½ hrs. During this SITE program demonstration, unfortunately the computer power supply was shorted out; replacing the computer with one of the existing lap-top computers compatible to the instrument and loading the available empirical calibration files took some time.

The ED2000 offers optimized excitation conditions for different element groups. This increased the analysis time per sample but provides better overall results. Based on the three criteria of MDL, Precision and Accuracy, ED2000 performed well compare to other XRF instruments (as evident by Tables 7-2 to 7-5) due to the optimized excitation, better counting statistics and sample preparation. One can reduce the sample preparation time to one minute by creating an empirical calibration as powders and measuring subsequent samples as powders. Powder results can be improved further by tapping the samples in the sample cups to take out the air pockets.

The results of this SITE study were essentially as expected. The MDLs calculated from the data for ED2000 were at least a factor of two better than the average of all other XRF instruments for antimony, cadmium, lead, selenium, silver and vanadium and better than the MDL for the remaining elements except arsenic which was equal in MDL of other XRF units. The precision data were better than reference lab and the average of the other XRF instruments except for the lead and vanadium (11 elements out of 13). The precision for lead was slightly worse than the average of all instruments at 6.3% vs. the average of 4.9%, however, this is quite acceptable for environmental analysis and better than the precision obtained by the reference laboratory (8.6% RSD). The vanadium excitation conditions were not optimized, as was done for other elements, in order to reduce the analysis time (again, because of the time constraint caused by start-up problems). Adding a set of excitation conditions will greatly improve the precision of the vanadium measurement at the expense of additional measurement time. The accuracy data based on RPD values were better than or equal to average values of other XRF instrments for nine elements out of thirteen. The accuracy can be improved significantly if site specific empirical calibration is used. 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. Antimony results for ED 2000 were closer to the real ERA spike value than any other XRF instruments or even the reference laboratory values.

In summary, the performance of the ED2000 was very good when compared to the other instruments in this study. Even greater accuracy can be achieved by fine tuning of calibration parameters and use of matrix-matched site-specific calibrations. The precision and MDL of the ED2000 was one of the best in the study; therefore, the improvements available for the calibration will make the ED2000 one of the best instruments available for the measurement of heavy metals in soil.

APPENDIX C

DATA VALIDATION SUMMARY REPORT

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APPENDIX

DATA VALIDATION REPORTS

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	<ol style="list-style-type: none"> 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 \leq 25	<ol style="list-style-type: none"> 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 \leq 20	<ol style="list-style-type: none"> 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	<ol style="list-style-type: none"> 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 \leq 20	<ol style="list-style-type: none"> 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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: SERIAL DILUTION 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

**TABLE 5: DATA QUALIFICATION: SERIAL DILUTION EXCEEDANCES
(Continued)**

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
LV-SO-34-XX	Antimony	870	mg/kg	J-	j
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

**TABLE 5: DATA QUALIFICATION: SERIAL DILUTION EXCEEDANCES
(Continued)**

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
SB-SO-15-XX	Iron	51000	mg/kg	J-	j
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 D. Analytical Data Summary, Oxford ED2000 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+	0.7 J-	0.1 U	290.0	26.0	1400.0	620.0
1	KP-SO-10-XX	Reference Laboratory	6.1 J+	0.7 J-	0.1 U	300.0	26.0	1600.0	560.0
1	KP-SO-15-XX	Reference Laboratory	6.3 J+	0.8 J-	0.1 U	340.0	26.0	1600.0	510.0
1	KP-SO-18-XX	Reference Laboratory	6.7 J+	0.6 J-	0.1 U	250.0	24.0	1200.0	500.0
1	KP-SO-22-XX	Reference Laboratory	8.3 J+	0.7 J-	0.1 U	260.0	29.0	1300.0	650.0
1	KP-SO-06-OI	Oxford Instrument Analytical ED 2000	8.9	77.0	1.6	505.2	32.0	21280.0	519.4
1	KP-SO-10-OI	Oxford Instrument Analytical ED 2000	7.4	70.3		574.0	27.9	21670.0	496.7
1	KP-SO-15-OI	Oxford Instrument Analytical ED 2000	4.2	60.4	0.2	513.1	23.3	21450.0	457.5
1	KP-SO-18-OI	Oxford Instrument Analytical ED 2000	7.4	49.5	0.2	512.2	27.9	21390.0	484.8
1	KP-SO-22-OI	Oxford Instrument Analytical ED 2000	10.3	53.4		506.6	28.5	21330.0	569.8
2	KP-SO-07-XX	Reference Laboratory	17.0 J+	2.0 J-	0.1 U	170.0	48.0	990.0	1200.0
2	KP-SO-13-XX	Reference Laboratory	16.0 J+	1.4 J-	0.0 U	180.0	52.0	980.0	1200.0
2	KP-SO-20-XX	Reference Laboratory	19.0 J+	1.5 J-	0.1 U	160.0	46.0	910.0	1300.0
2	KP-SO-24-XX	Reference Laboratory	17.0 J+	1.4 J-	0.1 U	160.0	49.0	900.0	1100.0
2	KP-SO-27-XX	Reference Laboratory	15.0 J+	1.3 J-	0.1 U	170.0	45.0	970.0	1200.0
2	KP-SO-29-XX	Reference Laboratory	18.0 J+	1.5 J-	0.1 U	150.0	42.0	870.0	1200.0
2	KP-SO-32-XX	Reference Laboratory	16.0 J+	1.6 J-	0.0 U	180.0	50.0	970.0	1200.0
2	KP-SO-07-OI	Oxford Instrument Analytical ED 2000	17.3	108.4		370.0	47.8	21010.0	1055.6
2	KP-SO-13-OI	Oxford Instrument Analytical ED 2000	19.0	119.3	0.1	318.7	47.4	20610.0	1029.9
2	KP-SO-20-OI	Oxford Instrument Analytical ED 2000	20.5	131.8		381.1	48.2	20950.0	1052.7
2	KP-SO-24-OI	Oxford Instrument Analytical ED 2000	16.2	116.3		341.6	42.0	20740.0	999.5
2	KP-SO-27-OI	Oxford Instrument Analytical ED 2000	13.0	122.2		358.4	50.2	20890.0	1002.9
2	KP-SO-29-OI	Oxford Instrument Analytical ED 2000	18.3	125.9		335.1	43.3	20740.0	1057.7
2	KP-SO-32-OI	Oxford Instrument Analytical ED 2000	17.6	122.9		352.1	48.6	20730.0	1032.0
3	KP-SO-04-XX	Reference Laboratory	94.0 J+	2.8	0.0 U	180.0	200.0	1300.0	5800.0
3	KP-SO-16-XX	Reference Laboratory	93.0 J+	2.9	0.1 U	200.0	230.0	1400.0	6100.0
3	KP-SO-23-XX	Reference Laboratory	86.0 J+	2.6	0.0 U	180.0	190.0	1300.0	5300.0
3	KP-SO-26-XX	Reference Laboratory	90.0 J+	3.7	0.1 U	210.0	230.0	1500.0	6500.0
3	KP-SO-31-XX	Reference Laboratory	88.0	28.0	0.1 U	140.0	200.0	1100.0	5700.0
3	KP-SO-04-OI	Oxford Instrument Analytical ED 2000	85.6	624.9	0.7	265.5	240.5	21200.0	4284.2
3	KP-SO-16-OI	Oxford Instrument Analytical ED 2000	93.4	616.9	0.2	272.0	235.6	21230.0	4262.2
3	KP-SO-23-OI	Oxford Instrument Analytical ED 2000	95.5	644.9	0.2	270.3	230.9	21080.0	4287.8
3	KP-SO-26-OI	Oxford Instrument Analytical ED 2000	90.6	615.7		263.6	237.4	21230.0	4346.0
3	KP-SO-31-OI	Oxford Instrument Analytical ED 2000	88.4	598.6	0.9	302.8	227.5	21290.0	4083.2

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
1	KP-SO-06-XX	Reference Laboratory	0.1 U	140.0	0.3 U	0.3 U	1.6 J	11.0
1	KP-SO-10-XX	Reference Laboratory	0.0 U	150.0	0.2 U	0.3 U	1.8 J	12.0
1	KP-SO-15-XX	Reference Laboratory	0.0 U	170.0	0.3 U	0.3 U	1.8 J	15.0
1	KP-SO-18-XX	Reference Laboratory	0.0 U	120.0	0.3 U	0.3 U	1.5 J	11.0
1	KP-SO-22-XX	Reference Laboratory	0.0 U	130.0	0.3 U	0.3 U	1.6 J	11.0
1	KP-SO-06-OI	Oxford Instrument Analytical ED 2000	0.3	145.2	1.4	1.2	22.7	19.2
1	KP-SO-10-OI	Oxford Instrument Analytical ED 2000	5.9	134.0	2.4	0.6	30.0	16.1
1	KP-SO-15-OI	Oxford Instrument Analytical ED 2000		142.6	2.7	0.2	39.2	20.1
1	KP-SO-18-OI	Oxford Instrument Analytical ED 2000		138.1	1.1	0.2	27.0	17.1
1	KP-SO-22-OI	Oxford Instrument Analytical ED 2000		139.4	5.0	0.2	29.0	24.3
2	KP-SO-07-XX	Reference Laboratory	0.0 U	87.0	0.2 U	0.3 U	1.2 J	26.0
2	KP-SO-13-XX	Reference Laboratory	0.0 U	90.0	0.3 U	0.3 U	1.2 J	24.0
2	KP-SO-20-XX	Reference Laboratory	0.0 U	79.0	0.3 U	0.3 U	1.2 J	25.0
2	KP-SO-24-XX	Reference Laboratory	0.0 U	78.0	0.3 U	0.3 U	1.1 J	22.0
2	KP-SO-27-XX	Reference Laboratory	0.0 U	87.0	0.3 U	0.3 U	1.2 J	24.0
2	KP-SO-29-XX	Reference Laboratory	0.0 U	73.0	0.3 U	0.3 U	1.1 J	22.0
2	KP-SO-32-XX	Reference Laboratory	0.0 U	88.0	0.5	0.3 U	1.2 J	24.0
2	KP-SO-07-OI	Oxford Instrument Analytical ED 2000		98.6	1.4	0.9	31.9	41.3
2	KP-SO-13-OI	Oxford Instrument Analytical ED 2000		97.9	1.2	1.3	32.3	33.3
2	KP-SO-20-OI	Oxford Instrument Analytical ED 2000		104.8	1.2	1.5	33.7	49.5
2	KP-SO-24-OI	Oxford Instrument Analytical ED 2000	5.3	106.8	0.2	0.9	34.7	20.7
2	KP-SO-27-OI	Oxford Instrument Analytical ED 2000		97.3	1.8	2.2	25.7	28.2
2	KP-SO-29-OI	Oxford Instrument Analytical ED 2000		98.4	2.4		27.2	28.9
2	KP-SO-32-OI	Oxford Instrument Analytical ED 2000		90.4	2.7	0.8	17.2	38.0
3	KP-SO-04-XX	Reference Laboratory	0.0 U	93.0	0.3 U	0.2 J	1.3 J	45.0
3	KP-SO-16-XX	Reference Laboratory	0.0 U	100.0	0.3 U	0.2 J	1.2 J	47.0
3	KP-SO-23-XX	Reference Laboratory	0.0 U	91.0	0.3 U	0.1 J	1.1 J	41.0
3	KP-SO-26-XX	Reference Laboratory	0.0 U	110.0	0.2 U	0.2 J	1.2 J	52.0
3	KP-SO-31-XX	Reference Laboratory	0.0 U	68.0	0.3 U	0.4	1.5 J	38.0
3	KP-SO-04-OI	Oxford Instrument Analytical ED 2000		80.9	1.6	0.7	40.3	55.8
3	KP-SO-16-OI	Oxford Instrument Analytical ED 2000	8.5	102.0	4.2		30.0	51.8
3	KP-SO-23-OI	Oxford Instrument Analytical ED 2000		101.4	5.1		34.1	57.9
3	KP-SO-26-OI	Oxford Instrument Analytical ED 2000		95.2	2.0		30.4	53.0
3	KP-SO-31-OI	Oxford Instrument Analytical ED 2000		103.3	1.4	0.6	32.7	51.9

Appendix D. Analytical Data Summary, Oxford ED2000 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	410.0	9.7	0.1	5.5	780.0	1700.0	18000.0
4	KP-SO-03-XX	Reference Laboratory	360.0	8.8	0.1 U	4.5	670.0	1600.0	19000.0
4	KP-SO-05-XX	Reference Laboratory	410.0	12.0	0.1 U	6.4	780.0	2000.0	24000.0
4	KP-SO-09-XX	Reference Laboratory	420.0	11.0	0.1 U	4.9	780.0	1800.0	22000.0
4	KP-SO-21-XX	Reference Laboratory	370.0	9.5	0.1 U	5.1	700.0	1700.0	19000.0
4	KP-SO-02-OI	Oxford Instrument Analytical ED 2000	471.4	2985.9	2.4	24.9	998.6	22170.0	15320.4
4	KP-SO-03-OI	Oxford Instrument Analytical ED 2000	463.7	2829.4	0.2	53.3	1008.1	22520.0	14759.9
4	KP-SO-05-OI	Oxford Instrument Analytical ED 2000	460.9	3008.5	1.5	25.4	1024.4	22360.0	15190.4
4	KP-SO-09-OI	Oxford Instrument Analytical ED 2000	454.6	2925.3	1.3	29.8	1063.0	22230.0	15099.1
4	KP-SO-21-OI	Oxford Instrument Analytical ED 2000	478.3	3011.0	0.9	68.2	1040.5	22860.0	15113.5
5	WS-SO-06-XX	Reference Laboratory	1.3 U	48.0	1.9	120.0	50.0	28000.0	110.0
5	WS-SO-08-XX	Reference Laboratory	1.3	45.0	2.0	120.0	47.0	26000.0	71.0
5	WS-SO-12-XX	Reference Laboratory	1.3 UJ	43.0	1.8	110.0	45.0	25000.0	65.0
5	WS-SO-17-XX	Reference Laboratory	1.3 UJ	47.0	1.9	120.0	49.0	28000.0	70.0
5	WS-SO-27-XX	Reference Laboratory	1.3 UJ	49.0	2.0	120.0	51.0	28000.0	72.0
5	WS-SO-30-XX	Reference Laboratory	1.2 J-	51.0	2.0	130.0	53.0	29000.0	81.0
5	WS-SO-35-XX	Reference Laboratory	1.3 UJ	49.0	2.0	130.0	51.0	28000.0	74.0
5	WS-SO-06-OI	Oxford Instrument Analytical ED 2000	3.5	74.0	1.7	124.4	56.2	53380.0	84.3
5	WS-SO-08-OI	Oxford Instrument Analytical ED 2000	4.1	62.7	1.2	126.9	59.8	53270.0	78.6
5	WS-SO-12-OI	Oxford Instrument Analytical ED 2000		74.2	2.0	126.0	63.9	52500.0	70.4
5	WS-SO-17-OI	Oxford Instrument Analytical ED 2000		60.6	0.7	133.5	54.5	52260.0	108.3
5	WS-SO-27-OI	Oxford Instrument Analytical ED 2000	2.1	63.5	1.1	79.9	65.0	42560.0	80.8
5	WS-SO-30-OI	Oxford Instrument Analytical ED 2000	4.9	77.1	4.0	83.1	51.5	42830.0	127.2
5	WS-SO-35-OI	Oxford Instrument Analytical ED 2000	2.5	68.7	1.9	76.7	58.3	42840.0	114.4
6	WS-SO-03-XX	Reference Laboratory	8.9 J-	500.0	12.0	140.0	170.0	32000.0	4300.0
6	WS-SO-05-XX	Reference Laboratory	8.6 J-	440.0	12.0	140.0	160.0	31000.0	4000.0
6	WS-SO-09-XX	Reference Laboratory	7.1 J-	480.0	12.0	130.0	160.0	30000.0	4000.0
6	WS-SO-14-XX	Reference Laboratory	8.4 J-	430.0	11.0	120.0	150.0	28000.0	3700.0
6	WS-SO-26-XX	Reference Laboratory	7.6 J-	520.0	12.0	140.0	160.0	30000.0	4000.0
6	WS-SO-31-XX	Reference Laboratory	7.2 J-	520.0	12.0	140.0	170.0	32000.0	4200.0
6	WS-SO-33-XX	Reference Laboratory	6.9 J-	450.0 J-	11.0 J-	120.0 J-	150.0 J-	28000.0 J-	3700.0 J-
6	WS-SO-03-OI	Oxford Instrument Analytical ED 2000	34.5	905.1	9.6	140.9	165.9	58370.0	3181.6
6	WS-SO-05-OI	Oxford Instrument Analytical ED 2000	36.4	883.7	9.1	130.5	181.2	59250.0	3243.2
6	WS-SO-09-OI	Oxford Instrument Analytical ED 2000	29.7	861.0	9.7	123.8	188.6	58800.0	3214.8
6	WS-SO-14-OI	Oxford Instrument Analytical ED 2000	32.5	864.8	9.6	119.9	185.9	58200.0	3280.1
6	WS-SO-26-OI	Oxford Instrument Analytical ED 2000	37.8	873.1	10.7	83.3	169.7	45820.0	3355.2
6	WS-SO-31-OI	Oxford Instrument Analytical ED 2000	28.7	921.1	10.3	84.4	179.2	44700.0	3298.1
6	WS-SO-33-OI	Oxford Instrument Analytical ED 2000	29.6	892.1	7.7	83.2	183.4	45520.0	3341.2

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
4	KP-SO-02-XX	Reference Laboratory	0.0 U	3.6	0.4 U	0.8	0.4 J	100.0
4	KP-SO-03-XX	Reference Laboratory	0.0 U	3.1	0.3 U	0.7	0.4 J	92.0
4	KP-SO-05-XX	Reference Laboratory	0.0 U	4.2	0.2 U	0.8	0.5 J	110.0
4	KP-SO-09-XX	Reference Laboratory	0.0 U	3.3	0.3 U	0.8	0.4 J	110.0
4	KP-SO-21-XX	Reference Laboratory	0.0 U	3.5	0.3 U	0.8	0.4 J	100.0
4	KP-SO-02-OI	Oxford Instrument Analytical ED 2000		47.8	4.0	0.2	32.9	137.2
4	KP-SO-03-OI	Oxford Instrument Analytical ED 2000		30.5	3.8	1.0	34.8	125.8
4	KP-SO-05-OI	Oxford Instrument Analytical ED 2000		34.4	1.8	0.7	30.8	156.6
4	KP-SO-09-OI	Oxford Instrument Analytical ED 2000		27.6	1.4	0.4	32.5	151.1
4	KP-SO-21-OI	Oxford Instrument Analytical ED 2000		53.9	2.1	1.1	27.1	158.0
5	WS-SO-06-XX	Reference Laboratory	0.1 U	61.0	1.3 U	0.9 J	56.0	230.0
5	WS-SO-08-XX	Reference Laboratory	0.1 U	58.0	1.3 U	0.9 J	52.0	220.0
5	WS-SO-12-XX	Reference Laboratory	0.1 UJ	55.0	1.3 U	0.9 J	49.0	210.0
5	WS-SO-17-XX	Reference Laboratory	0.1 UJ	59.0	1.3 U	0.9 J	56.0	230.0
5	WS-SO-27-XX	Reference Laboratory	0.1 J-	61.0	1.3 U	0.9 J	57.0	230.0
5	WS-SO-30-XX	Reference Laboratory	0.1 UJ	65.0	1.3 U	1.0 J	58.0	240.0
5	WS-SO-35-XX	Reference Laboratory	0.1 UJ	62.0	1.3 U	1.0 J	57.0	240.0
5	WS-SO-06-OI	Oxford Instrument Analytical ED 2000		51.1	0.0	0.9	103.5	245.6
5	WS-SO-08-OI	Oxford Instrument Analytical ED 2000		58.5	1.2	1.0	73.2	237.1
5	WS-SO-12-OI	Oxford Instrument Analytical ED 2000		64.7	0.5	1.7	90.8	261.1
5	WS-SO-17-OI	Oxford Instrument Analytical ED 2000		57.5		0.0	94.8	260.7
5	WS-SO-27-OI	Oxford Instrument Analytical ED 2000		52.4	1.1	0.5	75.9	252.0
5	WS-SO-30-OI	Oxford Instrument Analytical ED 2000	5.4	57.0	0.6	0.9	59.7	265.0
5	WS-SO-35-OI	Oxford Instrument Analytical ED 2000		50.7	0.8		87.8	257.0
6	WS-SO-03-XX	Reference Laboratory	0.9 J-	75.0	1.6	15.0	58.0	930.0
6	WS-SO-05-XX	Reference Laboratory	0.8 J-	71.0	1.3 U	15.0	57.0	900.0
6	WS-SO-09-XX	Reference Laboratory	0.9 J-	70.0	1.3 U	14.0	56.0	870.0
6	WS-SO-14-XX	Reference Laboratory	0.7 J-	64.0	1.3 U	13.0	50.0	820.0
6	WS-SO-26-XX	Reference Laboratory	0.8 J-	70.0	1.3 U	14.0	56.0	900.0
6	WS-SO-31-XX	Reference Laboratory	0.9 J-	72.0	1.2 U	15.0	60.0	950.0
6	WS-SO-33-XX	Reference Laboratory	0.9 J-	65.0 J-	1.3 U	13.0 J-	53.0 J-	830.0 J-
6	WS-SO-03-OI	Oxford Instrument Analytical ED 2000		79.0	1.4	13.9	97.7	905.4
6	WS-SO-05-OI	Oxford Instrument Analytical ED 2000		70.2	0.4	11.0	88.2	869.8
6	WS-SO-09-OI	Oxford Instrument Analytical ED 2000		69.1	3.2	13.4	92.0	866.3
6	WS-SO-14-OI	Oxford Instrument Analytical ED 2000		76.1	3.8	12.2	62.5	893.3
6	WS-SO-26-OI	Oxford Instrument Analytical ED 2000		73.6		8.2	75.9	901.1
6	WS-SO-31-OI	Oxford Instrument Analytical ED 2000		66.9	1.6	10.8	65.0	882.2
6	WS-SO-33-OI	Oxford Instrument Analytical ED 2000		68.7	3.1	10.3	68.7	905.2

Appendix D. Analytical Data Summary, Oxford ED2000 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	41.0 J-	1900.0	47.0	100.0	590.0	32000.0	18000.0
7	WS-SO-04-XX	Reference Laboratory	45.0 J-	2000.0	50.0	94.0	640.0	34000.0	20000.0
7	WS-SO-15-XX	Reference Laboratory	48.0 J-	2300.0	56.0	82.0	720.0	37000.0	24000.0
7	WS-SO-22-XX	Reference Laboratory	41.0 J-	1900.0	47.0	84.0	620.0	33000.0	17000.0
7	WS-SO-34-XX	Reference Laboratory	45.0 J-	2000.0	50.0	91.0	660.0	36000.0	22000.0
7	WS-SO-01-OI	Oxford Instrument Analytical ED 2000	174.0	6263.0	33.9	103.8	991.5	82320.0	11083.3
7	WS-SO-04-OI	Oxford Instrument Analytical ED 2000	177.9	6274.6	30.1	107.3	978.5	82870.0	11310.0
7	WS-SO-15-OI	Oxford Instrument Analytical ED 2000	177.9	6313.4	34.6	107.1	1000.9	80860.0	11532.1
7	WS-SO-22-OI	Oxford Instrument Analytical ED 2000	176.2	6380.5	33.8	115.8	1038.5	84150.0	11536.1
7	WS-SO-34-OI	Oxford Instrument Analytical ED 2000	226.8	6301.4	32.6	58.9	1021.3	62420.0	17939.5
8	WS-SO-02-XX	Reference Laboratory	130.0 J-	4200.0	98.0	49.0	1300.0	44000.0	35000.0
8	WS-SO-16-XX	Reference Laboratory	110.0 J-	3900.0	91.0	59.0	1300.0	42000.0	24000.0
8	WS-SO-18-XX	Reference Laboratory	130.0 J-	4100.0	95.0	63.0	1300.0	44000.0	37000.0
8	WS-SO-21-XX	Reference Laboratory	120.0 J-	3900.0	90.0	43.0	1200.0	40000.0	43000.0
8	WS-SO-24-XX	Reference Laboratory	97.0 J-	3600.0	81.0	54.0	1100.0	38000.0	27000.0
8	WS-SO-29-XX	Reference Laboratory	120.0 J-	3800.0	90.0	51.0	1200.0	40000.0	42000.0
8	WS-SO-37-XX	Reference Laboratory	120.0 J-	4100.0	95.0	63.0	1300.0	42000.0	26000.0
8	WS-SO-02-OI	Oxford Instrument Analytical ED 2000	358.0	17068.7	47.7	86.6	2763.5	123610.0	18337.4
8	WS-SO-16-OI	Oxford Instrument Analytical ED 2000	365.5	17233.1	49.6	85.2	2694.3	122680.0	18406.6
8	WS-SO-18-OI	Oxford Instrument Analytical ED 2000	658.7	17370.3	50.6	46.4	2724.0	92790.0	46619.7
8	WS-SO-21-OI	Oxford Instrument Analytical ED 2000	365.6	17250.2	45.5	88.4	2712.3	126730.0	18222.9
8	WS-SO-24-OI	Oxford Instrument Analytical ED 2000	358.0	16963.6	46.1	97.4	2695.8	124800.0	18467.6
8	WS-SO-29-OI	Oxford Instrument Analytical ED 2000	629.3	16976.2	51.9	44.7	2699.1	86890.0	44284.4
8	WS-SO-37-OI	Oxford Instrument Analytical ED 2000	635.3	16911.8	48.5	46.4	2719.7	87470.0	44831.3
9	WS-SO-13-XX	Reference Laboratory	200.0 J-	5800.0	150.0	53.0	1800.0	47000.0	45000.0
9	WS-SO-19-XX	Reference Laboratory	150.0 J-	5000.0	130.0	66.0	1500.0	39000.0	24000.0
9	WS-SO-28-XX	Reference Laboratory	120.0 J-	4200.0	100.0	54.0	1200.0	33000.0	30000.0
9	WS-SO-32-XX	Reference Laboratory	190.0 J-	5500.0	140.0	54.0	1700.0	44000.0	30000.0
9	WS-SO-36-XX	Reference Laboratory	120.0 J-	3800.0	92.0	51.0	1100.0	30000.0	45000.0
9	WS-SO-13-OI	Oxford Instrument Analytical ED 2000	1612.9	24834.6	215.2	94.6	3926.4	138890.0	18011.3
9	WS-SO-19-OI	Oxford Instrument Analytical ED 2000			0.3	13.7		18510.0	
9	WS-SO-28-OI	Oxford Instrument Analytical ED 2000	945.2	24753.2	69.3	41.4	3969.2	97470.0	61960.0
9	WS-SO-32-OI	Oxford Instrument Analytical ED 2000	976.0	25297.7	67.3	39.2	4085.8	98170.0	63414.9
9	WS-SO-36-OI	Oxford Instrument Analytical ED 2000	971.0	24868.2	72.6	41.5	4060.4	99560.0	62815.5

Appendix D. Analytical Data Summary, Oxford ED2000 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	66.0	1.3 U	69.0 J-	42.0	3000.0
7	WS-SO-04-XX	Reference Laboratory	6.5	62.0	1.3 U	76.0 J-	44.0	3100.0
7	WS-SO-15-XX	Reference Laboratory	5.8	58.0	1.3 U	90.0 J-	52.0	3400.0
7	WS-SO-22-XX	Reference Laboratory	4.8	57.0	1.3 U	72.0 J-	44.0	3000.0
7	WS-SO-34-XX	Reference Laboratory	5.4	60.0	1.3 U	78.0 J-	47.0	3200.0
7	WS-SO-01-OI	Oxford Instrument Analytical ED 2000	15.3	119.6	7.1	43.8	112.6	4583.5
7	WS-SO-04-OI	Oxford Instrument Analytical ED 2000		143.9	5.8	44.9	112.9	4760.6
7	WS-SO-15-OI	Oxford Instrument Analytical ED 2000		122.6		44.2	72.2	4809.9
7	WS-SO-22-OI	Oxford Instrument Analytical ED 2000		127.7		44.6	94.2	4734.2
7	WS-SO-34-OI	Oxford Instrument Analytical ED 2000		98.7	4.5	42.3	54.5	4705.4
8	WS-SO-02-XX	Reference Laboratory	17.0	57.0	1.3 U	150.0 J-	36.0	6000.0
8	WS-SO-16-XX	Reference Laboratory	15.0	60.0	1.1 J	150.0 J-	35.0	5700.0
8	WS-SO-18-XX	Reference Laboratory	17.0	62.0	1.9	140.0 J-	36.0	5900.0
8	WS-SO-21-XX	Reference Laboratory	14.0	51.0	1.6	150.0 J-	33.0	5500.0
8	WS-SO-24-XX	Reference Laboratory	16.0	54.0	2.1	140.0 J-	30.0	5200.0
8	WS-SO-29-XX	Reference Laboratory	15.0	55.0	1.7	140.0 J-	33.0	5500.0
8	WS-SO-37-XX	Reference Laboratory	14.0	63.0	3.0	140.0 J-	34.0	5800.0
8	WS-SO-02-OI	Oxford Instrument Analytical ED 2000		169.4	12.1	66.9	68.6	11539.1
8	WS-SO-16-OI	Oxford Instrument Analytical ED 2000		235.6	12.8	67.5	89.5	11591.0
8	WS-SO-18-OI	Oxford Instrument Analytical ED 2000		198.1		70.4	47.8	12337.3
8	WS-SO-21-OI	Oxford Instrument Analytical ED 2000		209.7		69.4	68.6	11869.8
8	WS-SO-24-OI	Oxford Instrument Analytical ED 2000		231.7		67.1	74.9	11707.8
8	WS-SO-29-OI	Oxford Instrument Analytical ED 2000		201.9	5.3	71.3	60.2	11875.1
8	WS-SO-37-OI	Oxford Instrument Analytical ED 2000		247.9	5.8	72.4	45.8	11766.6
9	WS-SO-13-XX	Reference Laboratory	11.0	75.0	3.7	170.0 J-	24.0	9000.0
9	WS-SO-19-XX	Reference Laboratory	12.0	74.0	3.7	160.0 J-	20.0	7700.0
9	WS-SO-28-XX	Reference Laboratory	11.0	59.0	2.3	130.0 J-	16.0	6100.0
9	WS-SO-32-XX	Reference Laboratory	11.0	73.0	3.7	190.0 J-	23.0	8500.0
9	WS-SO-36-XX	Reference Laboratory	13.0	55.0	1.7	120.0 J-	15.0	5700.0
9	WS-SO-13-OI	Oxford Instrument Analytical ED 2000		317.3	12.3	245.1	94.6	20334.5
9	WS-SO-19-OI	Oxford Instrument Analytical ED 2000	0.7	9.7	0.7		37.3	
9	WS-SO-28-OI	Oxford Instrument Analytical ED 2000		307.4	7.7	76.1	49.8	20711.8
9	WS-SO-32-OI	Oxford Instrument Analytical ED 2000		325.6		78.4	57.9	20903.9
9	WS-SO-36-OI	Oxford Instrument Analytical ED 2000		324.0		80.5	55.5	21042.3

Appendix D. Analytical Data Summary, Oxford ED2000 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	0.9	120.0	32.0	24000.0	63.0
10	BN-SO-10-XX	Reference Laboratory	1.3 UJ	50.0	1.2	110.0	35.0	24000.0	140.0
10	BN-SO-15-XX	Reference Laboratory	1.3 UJ	34.0	0.8	110.0	29.0	22000.0	56.0
10	BN-SO-18-XX	Reference Laboratory	1.3 U	37.0	0.9	110.0	29.0	22000.0	59.0
10	BN-SO-28-XX	Reference Laboratory	1.5	35.0	0.9	100.0	28.0	22000.0	58.0
10	BN-SO-31-XX	Reference Laboratory	1.3	41.0	1.0	140.0	33.0	26000.0	65.0
10	BN-SO-35-XX	Reference Laboratory	1.4	37.0	1.0	120.0	30.0	23000.0	60.0
10	BN-SO-01-OI	Oxford Instrument Analytical ED 2000	6.5	53.9		96.0	45.5	42290.0	75.2
10	BN-SO-10-OI	Oxford Instrument Analytical ED 2000	5.2	56.4	0.4	96.5	44.3	41200.0	75.7
10	BN-SO-15-OI	Oxford Instrument Analytical ED 2000	0.2	56.9	1.6	108.0	40.5	40820.0	61.7
10	BN-SO-18-OI	Oxford Instrument Analytical ED 2000	2.3	57.6	2.4	95.3	37.4	42180.0	60.8
10	BN-SO-28-OI	Oxford Instrument Analytical ED 2000	2.8	48.4	0.6	105.9	38.0	42030.0	86.2
10	BN-SO-31-OI	Oxford Instrument Analytical ED 2000	3.4	55.3	2.2	102.8	40.5	41220.0	62.5
10	BN-SO-35-OI	Oxford Instrument Analytical ED 2000	1.9	56.6	1.5	102.5	35.4	42620.0	64.7
11	BN-SO-02-XX	Reference Laboratory	11.0	140.0	50.0	90.0	170.0	28000.0	840.0
11	BN-SO-04-XX	Reference Laboratory	9.1	120.0	42.0	79.0	140.0	24000.0	700.0
11	BN-SO-17-XX	Reference Laboratory	9.3	110.0	39.0	79.0	140.0	23000.0	680.0
11	BN-SO-22-XX	Reference Laboratory	7.3	98.0	34.0	65.0	110.0	20000.0	590.0
11	BN-SO-27-XX	Reference Laboratory	9.6	110.0	39.0	78.0	130.0	24000.0	660.0
11	BN-SO-02-OI	Oxford Instrument Analytical ED 2000	30.7	192.9	31.4	68.9	164.3	41901.0	648.7
11	BN-SO-04-OI	Oxford Instrument Analytical ED 2000	33.2	213.4	34.7	75.3	148.9	43010.0	612.9
11	BN-SO-17-OI	Oxford Instrument Analytical ED 2000	30.1	203.8	33.0	74.7	149.3	41860.0	639.5
11	BN-SO-22-OI	Oxford Instrument Analytical ED 2000	38.2	201.6	33.8	75.8	152.5	42120.0	657.7
11	BN-SO-27-OI	Oxford Instrument Analytical ED 2000	29.5	206.7	35.1	65.1	149.8	42190.0	643.1
12	BN-SO-03-XX	Reference Laboratory	65.0	620.0	290.0	120.0	840.0	25000.0	4700.0
12	BN-SO-06-XX	Reference Laboratory	60.0	600.0	280.0	94.0	810.0	24000.0	4500.0
12	BN-SO-08-XX	Reference Laboratory	57.0	570.0	270.0	100.0	750.0	22000.0	4300.0
12	BN-SO-13-XX	Reference Laboratory	65.0	320.0	150.0	98.0	410.0	17000.0	2400.0
12	BN-SO-20-XX	Reference Laboratory	57.0	540.0	260.0	88.0	730.0	22000.0	4100.0
12	BN-SO-30-XX	Reference Laboratory	64.0	630.0	300.0	100.0	860.0	26000.0	4800.0
12	BN-SO-34-XX	Reference Laboratory	68.0	630.0	290.0	110.0	830.0	25000.0	4700.0
12	BN-SO-03-OI	Oxford Instrument Analytical ED 2000	240.8	1222.6	218.1	80.6	941.0	42780.0	3666.3
12	BN-SO-06-OI	Oxford Instrument Analytical ED 2000	243.7	1216.8	213.5	92.4	972.8	44240.0	3763.7
12	BN-SO-08-OI	Oxford Instrument Analytical ED 2000	231.8	1249.2	206.0	75.0	953.9	43670.0	3750.3
12	BN-SO-13-OI	Oxford Instrument Analytical ED 2000	242.3	1235.4	209.4	77.8	953.1	42550.0	3743.0
12	BN-SO-20-OI	Oxford Instrument Analytical ED 2000	248.3	1223.7	217.2	77.5	955.3	45500.0	3785.4
12	BN-SO-30-OI	Oxford Instrument Analytical ED 2000	240.8	1207.8	209.0	77.3	931.1	43960.0	3816.9
12	BN-SO-34-OI	Oxford Instrument Analytical ED 2000	236.6	1247.6	212.5	83.2	963.9	43140.0	3689.0

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
10	BN-SO-01-XX	Reference Laboratory	0.1	63.0	1.3 U	1.3 UJ	55.0	92.0
10	BN-SO-10-XX	Reference Laboratory	0.1	54.0	1.2 J	1.3 UJ	55.0	110.0
10	BN-SO-15-XX	Reference Laboratory	0.2	58.0	1.3 U	1.3 UJ	49.0	89.0
10	BN-SO-18-XX	Reference Laboratory	0.1	59.0	1.3	0.9 U	46.0	88.0
10	BN-SO-28-XX	Reference Laboratory	0.2	54.0	1.3 U	0.8 U	48.0	81.0
10	BN-SO-31-XX	Reference Laboratory	0.1	71.0	1.3 U	1.0 U	54.0	94.0
10	BN-SO-35-XX	Reference Laboratory	0.2	63.0	1.2 J	0.9 U	50.0	87.0
10	BN-SO-01-OI	Oxford Instrument Analytical ED 2000		60.6		1.8	76.6	102.7
10	BN-SO-10-OI	Oxford Instrument Analytical ED 2000		69.1	1.2		77.9	111.5
10	BN-SO-15-OI	Oxford Instrument Analytical ED 2000	3.9	67.6	2.3	0.9	62.6	111.9
10	BN-SO-18-OI	Oxford Instrument Analytical ED 2000	3.2	64.6	0.3	0.9	76.6	99.9
10	BN-SO-28-OI	Oxford Instrument Analytical ED 2000	4.8	62.8	2.4	0.6	78.4	126.0
10	BN-SO-31-OI	Oxford Instrument Analytical ED 2000		63.3	1.9	0.8	45.8	114.3
10	BN-SO-35-OI	Oxford Instrument Analytical ED 2000	5.1	67.2	1.3	1.1	72.9	111.7
11	BN-SO-02-XX	Reference Laboratory	0.4	54.0	4.3	7.6	60.0	470.0
11	BN-SO-04-XX	Reference Laboratory	0.4	48.0	2.9	6.5	50.0	400.0
11	BN-SO-17-XX	Reference Laboratory	0.4	47.0	2.7	6.3	49.0	390.0
11	BN-SO-22-XX	Reference Laboratory	0.4	40.0	2.8	5.4	43.0	330.0
11	BN-SO-27-XX	Reference Laboratory	0.4	46.0	3.7	6.1	52.0	380.0
11	BN-SO-02-OI	Oxford Instrument Analytical ED 2000		55.2		5.2	63.5	446.5
11	BN-SO-04-OI	Oxford Instrument Analytical ED 2000		57.9	1.0	3.5	51.5	468.1
11	BN-SO-17-OI	Oxford Instrument Analytical ED 2000		57.6	1.8	4.3	69.2	452.9
11	BN-SO-22-OI	Oxford Instrument Analytical ED 2000	8.4	59.4	0.5	5.1	84.4	450.5
11	BN-SO-27-OI	Oxford Instrument Analytical ED 2000	0.5	56.8	3.0	6.1	75.7	455.8
12	BN-SO-03-XX	Reference Laboratory	1.6	100.0	17.0	42.0	48.0	2300.0
12	BN-SO-06-XX	Reference Laboratory	2.0	92.0	15.0	41.0	48.0	2300.0
12	BN-SO-08-XX	Reference Laboratory	2.0	94.0	14.0	38.0	39.0	2200.0
12	BN-SO-13-XX	Reference Laboratory	1.6	71.0	9.2	21.0	37.0	1200.0
12	BN-SO-20-XX	Reference Laboratory	1.6	84.0	14.0	37.0	44.0	2100.0
12	BN-SO-30-XX	Reference Laboratory	1.6	99.0	17.0	44.0	50.0	2400.0
12	BN-SO-34-XX	Reference Laboratory	2.0	100.0	17.0	42.0	49.0	2300.0
12	BN-SO-03-OI	Oxford Instrument Analytical ED 2000		114.5	11.1	28.8	69.5	2712.0
12	BN-SO-06-OI	Oxford Instrument Analytical ED 2000	7.0	128.6	11.8	27.9	64.2	2718.4
12	BN-SO-08-OI	Oxford Instrument Analytical ED 2000		99.1	11.5	26.7	48.7	2767.3
12	BN-SO-13-OI	Oxford Instrument Analytical ED 2000		101.3	12.1	28.0	63.6	2757.9
12	BN-SO-20-OI	Oxford Instrument Analytical ED 2000		110.2	12.6	29.0	55.0	2757.3
12	BN-SO-30-OI	Oxford Instrument Analytical ED 2000	5.7	99.2	10.8	28.4	58.0	2820.3
12	BN-SO-34-OI	Oxford Instrument Analytical ED 2000		105.9	13.2	28.7	59.4	2684.0

Appendix D. Analytical Data Summary, Oxford ED2000 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	110.0 J-	990.0 J+	520.0	82.0	1400.0	23000.0	6900.0
13	BN-SO-16-XX	Reference Laboratory	120.0 J-	1100.0 J+	570.0	86.0	1500.0	25000.0	8100.0
13	BN-SO-21-XX	Reference Laboratory	150.0 J-	1300.0 J+	660.0	110.0	1700.0	30000.0	8900.0
13	BN-SO-25-XX	Reference Laboratory	82.0 J-	700.0 J	370.0 J-	64.0 J-	930.0 J-	16000.0 J-	5400.0 J-
13	BN-SO-33-XX	Reference Laboratory	100.0 J-	1100.0	640.0	100.0	1600.0	27000.0	8000.0
13	BN-SO-07-OI	Oxford Instrument Analytical ED 2000	465.9	2264.2	389.5	81.8	1844.8	45040.0	6402.2
13	BN-SO-16-OI	Oxford Instrument Analytical ED 2000	486.5	2276.5	389.4	62.4	1824.6	44590.0	6428.6
13	BN-SO-21-OI	Oxford Instrument Analytical ED 2000	489.4	2264.1	392.3	68.9	1845.9	44500.0	6420.8
13	BN-SO-25-OI	Oxford Instrument Analytical ED 2000	482.2	2267.2	388.0	72.5	1811.6	45010.0	6434.6
13	BN-SO-33-OI	Oxford Instrument Analytical ED 2000	458.3	2280.0	375.5	75.5	1842.9	44250.0	6460.2
14	BN-SO-05-XX	Reference Laboratory	160.0 J-	1600.0	850.0	86.0	2200.0	26000.0	12000.0
14	BN-SO-19-XX	Reference Laboratory	150.0 J-	1600.0	860.0	79.0	2200.0	26000.0	12000.0
14	BN-SO-26-XX	Reference Laboratory	150.0 J-	1700.0	900.0	82.0	2400.0	27000.0	12000.0
14	BN-SO-29-XX	Reference Laboratory	150.0 J-	1600.0	880.0	86.0	2300.0	26000.0	12000.0
14	BN-SO-32-XX	Reference Laboratory	160.0 J-	1600.0	860.0	84.0	2300.0	26000.0	12000.0
14	BN-SO-05-OI	Oxford Instrument Analytical ED 2000	779.7	3755.9	507.5	65.5	3096.0	46890.0	10106.0
14	BN-SO-19-OI	Oxford Instrument Analytical ED 2000	748.3	3767.8	496.9	55.8	3086.3	46140.0	9893.2
14	BN-SO-26-OI	Oxford Instrument Analytical ED 2000	801.7	3770.1	507.9	94.2	3045.2	72630.0	10213.3
14	BN-SO-29-OI	Oxford Instrument Analytical ED 2000	771.4	3818.8	521.9	56.8	3044.6	46810.0	9877.2
14	BN-SO-32-OI	Oxford Instrument Analytical ED 2000	785.5	3956.6	491.5	55.7	3090.3	46420.0	10171.6
15	CN-SO-01-XX	Reference Laboratory	13.0 J-	13.0	21.0	190.0	700.0	38000.0	1200.0
15	CN-SO-04-XX	Reference Laboratory	13.0 J-	11.0	21.0	200.0	680.0	37000.0	1200.0
15	CN-SO-08-XX	Reference Laboratory	15.0 J-	15.0	25.0	210.0	740.0	43000.0	1300.0
15	CN-SO-10-XX	Reference Laboratory	13.0 J-	13.0	22.0	200.0	760.0	39000.0	1200.0
15	CN-SO-11-XX	Reference Laboratory	17.0 J-	16.0	30.0	240.0	860.0	47000.0	1600.0
15	CN-SO-01-OI	Oxford Instrument Analytical ED 2000	66.2	123.9	17.9	190.4	728.8	46740.0	1096.9
15	CN-SO-04-OI	Oxford Instrument Analytical ED 2000	63.7	134.3	20.7	176.4	782.4	46110.0	1102.2
15	CN-SO-08-OI	Oxford Instrument Analytical ED 2000	66.1	133.5	19.4	181.7	717.2	46250.0	1057.6
15	CN-SO-10-OI	Oxford Instrument Analytical ED 2000	59.9	148.5	19.4	177.0	782.4	45200.0	1048.2
15	CN-SO-11-OI	Oxford Instrument Analytical ED 2000	62.7	154.4	17.4	181.8	721.8	45930.0	1043.5
16	AS-SO-02-XX	Reference Laboratory	2.6 UJ	18.0	50.0	180.0	140.0	48000.0	1600.0
16	AS-SO-06-XX	Reference Laboratory	2.4 UJ	19.0	52.0	190.0	130.0	52000.0	1600.0
16	AS-SO-10-XX	Reference Laboratory	1.9 J-	18.0	48.0	180.0	110.0	45000.0	1400.0
16	AS-SO-11-XX	Reference Laboratory	3.7 J-	22.0	63.0	230.0	150.0	52000.0	2100.0
16	AS-SO-13-XX	Reference Laboratory	2.4 UJ	20.0	57.0	200.0	150.0	52000.0	1700.0
16	AS-SO-02-OI	Oxford Instrument Analytical ED 2000	2.6	233.9	40.9	113.9	132.1	48090.0	1515.5
16	AS-SO-06-OI	Oxford Instrument Analytical ED 2000	6.5	226.3	41.7	116.6	135.9	45720.0	1440.4
16	AS-SO-10-OI	Oxford Instrument Analytical ED 2000	4.9	237.8	41.3	114.0	145.5	46870.0	1507.2
16	AS-SO-11-OI	Oxford Instrument Analytical ED 2000		212.6	0.1	121.6	126.5	48780.0	1540.5
16	AS-SO-13-OI	Oxford Instrument Analytical ED 2000	5.0	244.8	40.3	121.0	133.5	46450.0	1501.9

Appendix D. Analytical Data Summary, Oxford ED2000 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	120.0	26.0	70.0	41.0	4000.0
13	BN-SO-16-XX	Reference Laboratory	3.4	130.0	29.0	77.0	44.0	4400.0
13	BN-SO-21-XX	Reference Laboratory	3.6	160.0	35.0	88.0	52.0	5100.0
13	BN-SO-25-XX	Reference Laboratory	3.8	88.0 J-	19.0 J-	48.0 J-	28.0 J-	2900.0 J-
13	BN-SO-33-XX	Reference Laboratory	4.0	150.0	34.0	81.0	48.0	5100.0
13	BN-SO-07-OI	Oxford Instrument Analytical ED 2000		160.7	22.8	48.5	46.3	5431.7
13	BN-SO-16-OI	Oxford Instrument Analytical ED 2000		152.0	19.9	44.2	53.5	5453.9
13	BN-SO-21-OI	Oxford Instrument Analytical ED 2000		150.6	21.0	48.9	49.5	5388.1
13	BN-SO-25-OI	Oxford Instrument Analytical ED 2000		166.5	19.6	47.5	54.8	5384.0
13	BN-SO-33-OI	Oxford Instrument Analytical ED 2000		150.0	20.8	46.4	55.8	5477.9
14	BN-SO-05-XX	Reference Laboratory	5.0	160.0	48.0	110.0	39.0	6700.0
14	BN-SO-19-XX	Reference Laboratory	5.0	160.0	48.0	120.0	39.0	6700.0
14	BN-SO-26-XX	Reference Laboratory	5.4	160.0	49.0	120.0	40.0	7000.0
14	BN-SO-29-XX	Reference Laboratory	5.4	160.0	48.0	120.0	41.0	6800.0
14	BN-SO-32-XX	Reference Laboratory	5.4	160.0	48.0	120.0	39.0	6700.0
14	BN-SO-05-OI	Oxford Instrument Analytical ED 2000		215.9	40.6	65.0	61.3	8647.5
14	BN-SO-19-OI	Oxford Instrument Analytical ED 2000		184.8	37.9	61.5	62.3	8504.3
14	BN-SO-26-OI	Oxford Instrument Analytical ED 2000		195.7	37.2	63.2	79.0	8629.0
14	BN-SO-29-OI	Oxford Instrument Analytical ED 2000		192.3	31.9	65.4	63.7	8680.1
14	BN-SO-32-OI	Oxford Instrument Analytical ED 2000		199.2	29.8	63.5	54.5	8715.8
15	CN-SO-01-XX	Reference Laboratory	0.1	240.0	2.2	12.0	21.0	3100.0
15	CN-SO-04-XX	Reference Laboratory	0.1	240.0	1.5	12.0	22.0	2900.0
15	CN-SO-08-XX	Reference Laboratory	0.2	280.0	1.3 U	15.0	26.0	3200.0
15	CN-SO-10-XX	Reference Laboratory	0.1	240.0	1.9	14.0	22.0	3000.0
15	CN-SO-11-XX	Reference Laboratory	0.2	320.0	1.3 U	16.0	27.0	3500.0
15	CN-SO-01-OI	Oxford Instrument Analytical ED 2000		252.2	1.6	8.3	10.9	3685.1
15	CN-SO-04-OI	Oxford Instrument Analytical ED 2000		251.7	2.5	11.8	32.4	3582.7
15	CN-SO-08-OI	Oxford Instrument Analytical ED 2000		257.3	3.5	8.3	26.5	3559.9
15	CN-SO-10-OI	Oxford Instrument Analytical ED 2000	2.1	247.5		10.4	11.6	3530.5
15	CN-SO-11-OI	Oxford Instrument Analytical ED 2000		232.9	3.3	8.1	34.7	3577.6
16	AS-SO-02-XX	Reference Laboratory	0.8	91.0	2.6 U	4.5	42.0	3300.0
16	AS-SO-06-XX	Reference Laboratory	0.7	93.0	2.6 U	4.8	44.0	3500.0
16	AS-SO-10-XX	Reference Laboratory	0.8	84.0	1.1 U	4.4	42.0	3000.0
16	AS-SO-11-XX	Reference Laboratory	0.7	120.0	1.1 U	5.6	54.0	3800.0
16	AS-SO-13-XX	Reference Laboratory	0.8	100.0	3.0	5.2	50.0	3800.0
16	AS-SO-02-OI	Oxford Instrument Analytical ED 2000	1.6	118.9		2.8	46.6	4085.7
16	AS-SO-06-OI	Oxford Instrument Analytical ED 2000		86.6	1.5	1.3	53.7	3745.3
16	AS-SO-10-OI	Oxford Instrument Analytical ED 2000	5.5	97.9	2.0	2.3	45.1	3971.3
16	AS-SO-11-OI	Oxford Instrument Analytical ED 2000		104.2	4.7		58.9	3872.2
16	AS-SO-13-OI	Oxford Instrument Analytical ED 2000	0.7	106.6	1.8	2.2	44.8	4027.5

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
17	AS-SO-01-XX	Reference Laboratory	3.8 J-	26.0	100.0	420.0	250.0	100000.0	3200.0
17	AS-SO-04-XX	Reference Laboratory	6.4 UJ	22.0	110.0	480.0	260.0	110000.0	3300.0
17	AS-SO-07-XX	Reference Laboratory	3.6 J-	21.0	97.0	380.0	240.0	88000.0	2900.0
17	AS-SO-09-XX	Reference Laboratory	2.6 UJ	25.0 J-	100.0 J-	390.0 J-	250.0 J-	94000.0 J-	3200.0 J-
17	AS-SO-12-XX	Reference Laboratory	2.6 UJ	29.0	120.0	440.0	270.0	93000.0	3300.0
17	AS-SO-01-OI	Oxford Instrument Analytical ED 2000	5.8	563.2	74.6	153.3	313.5	61940.0	3146.2
17	AS-SO-04-OI	Oxford Instrument Analytical ED 2000	6.1	564.6	72.5	156.8	290.1	64380.0	3069.8
17	AS-SO-07-OI	Oxford Instrument Analytical ED 2000	10.8	528.4	65.7	160.1	287.2	62830.0	3165.0
17	AS-SO-09-OI	Oxford Instrument Analytical ED 2000	7.9	541.1	70.5	162.7	310.6	63230.0	3118.5
17	AS-SO-12-OI	Oxford Instrument Analytical ED 2000	4.5	532.9	69.8	152.9	293.0	63890.0	3141.5
18	SB-SO-03-XX	Reference Laboratory	1.2 UJ	8.8	0.5 U	150.0	48.0	38000.0	18.0
18	SB-SO-06-XX	Reference Laboratory	1.7 J-	8.2	0.5 U	140.0	44.0	35000.0	16.0
18	SB-SO-14-XX	Reference Laboratory	4.1 J-	8.6	0.5 U	150.0	46.0	37000.0	17.0
18	SB-SO-38-XX	Reference Laboratory	1.3 UJ	9.6	0.5 U	150.0	57.0	37000.0	18.0
18	SB-SO-41-XX	Reference Laboratory	1.3 UJ	9.1	0.5 U	160.0	58.0	40000.0	19.0
18	SB-SO-47-XX	Reference Laboratory	1.3 UJ	7.8	0.5 U	140.0	44.0	34000.0	16.0
18	SB-SO-51-XX	Reference Laboratory	1.3 UJ	9.1	0.5 U	160.0	50.0	40000.0	18.0
18	SB-SO-03-OI	Oxford Instrument Analytical ED 2000	7.3	36.0	0.5	220.3	40.5	66390.0	16.9
18	SB-SO-06-OI	Oxford Instrument Analytical ED 2000	5.8	13.6	0.1	213.1	48.7	70040.0	20.1
18	SB-SO-14-OI	Oxford Instrument Analytical ED 2000	10.2	16.0		229.4	59.9	71090.0	12.2
18	SB-SO-38-OI	Oxford Instrument Analytical ED 2000	9.5	15.5	0.6	220.7	57.9	68920.0	13.7
18	SB-SO-41-OI	Oxford Instrument Analytical ED 2000	8.1	23.4		250.2	46.3	69150.0	14.4
18	SB-SO-47-OI	Oxford Instrument Analytical ED 2000	6.3	17.2	1.0	238.3	44.2	70290.0	18.7
18	SB-SO-51-OI	Oxford Instrument Analytical ED 2000	6.1	15.0	0.0	220.8	58.7	70050.0	24.4
19	SB-SO-05-XX	Reference Laboratory	1.6 J-	9.2	0.5 U	140.0	46.0	35000.0	16.0
19	SB-SO-18-XX	Reference Laboratory	1.2 UJ	10.0	0.5 U	150.0	46.0	38000.0	17.0
19	SB-SO-30-XX	Reference Laboratory	3.2 J-	6.9	0.5 U	94.0	27.0	22000.0	10.0
19	SB-SO-40-XX	Reference Laboratory	2.2 J-	8.5	0.5 U	120.0	40.0	33000.0	15.0
19	SB-SO-53-XX	Reference Laboratory	1.2 UJ	10.0	0.5 U	140.0	44.0	37000.0	17.0
19	SB-SO-05-OI	Oxford Instrument Analytical ED 2000	19.7	19.5	1.2	214.4	45.3	68690.0	24.9
19	SB-SO-18-OI	Oxford Instrument Analytical ED 2000	15.9	18.9		229.3	59.6	68600.0	36.8
19	SB-SO-30-OI	Oxford Instrument Analytical ED 2000	13.6	18.7	0.0	220.7	50.0	68280.0	40.0
19	SB-SO-40-OI	Oxford Instrument Analytical ED 2000	14.4	24.1		234.2	43.4	69150.0	13.9
19	SB-SO-53-OI	Oxford Instrument Analytical ED 2000	14.9	19.2		219.1	35.8	67100.0	14.4

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
17	AS-SO-01-XX	Reference Laboratory	1.4	180.0	2.6 U	9.3	66.0	6900.0
17	AS-SO-04-XX	Reference Laboratory	1.3	200.0	6.2 U	12.0	72.0	7400.0
17	AS-SO-07-XX	Reference Laboratory	1.4	160.0	2.7	8.9	63.0	6300.0
17	AS-SO-09-XX	Reference Laboratory	1.4	170.0 J-	2.6 U	9.6 J-	65.0 J-	6800.0 J-
17	AS-SO-12-XX	Reference Laboratory	1.4	190.0	2.6 U	3.2	73.0	7500.0
17	AS-SO-01-OI	Oxford Instrument Analytical ED 2000		218.2	1.9	4.2	46.6	10002.5
17	AS-SO-04-OI	Oxford Instrument Analytical ED 2000		222.1	4.5	3.8	49.9	9861.4
17	AS-SO-07-OI	Oxford Instrument Analytical ED 2000	8.3	195.6	3.7	3.2	56.0	9715.6
17	AS-SO-09-OI	Oxford Instrument Analytical ED 2000	7.0	223.5	0.4	4.0	51.7	9796.8
17	AS-SO-12-OI	Oxford Instrument Analytical ED 2000		204.8		4.2	53.5	9773.8
18	SB-SO-03-XX	Reference Laboratory	62.0	210.0	1.3 U	1.3 U	67.0	90.0
18	SB-SO-06-XX	Reference Laboratory	55.0	200.0	1.3 U	1.3 U	63.0	82.0
18	SB-SO-14-XX	Reference Laboratory	55.0	210.0	1.3 U	1.3 U	66.0	95.0
18	SB-SO-38-XX	Reference Laboratory	56.0	210.0	1.3 U	1.3 U	68.0	91.0
18	SB-SO-41-XX	Reference Laboratory	54.0	230.0	1.3 U	1.3 U	71.0	96.0
18	SB-SO-47-XX	Reference Laboratory	58.0	200.0	1.3 U	1.3 U	62.0	82.0
18	SB-SO-51-XX	Reference Laboratory	54.0	230.0	1.3 U	1.3 U	74.0	93.0
18	SB-SO-03-OI	Oxford Instrument Analytical ED 2000	54.3	207.7	2.1	0.5	113.7	114.0
18	SB-SO-06-OI	Oxford Instrument Analytical ED 2000	51.5	200.1	2.6		118.5	104.0
18	SB-SO-14-OI	Oxford Instrument Analytical ED 2000	52.7	216.5	1.2	1.1	143.7	114.0
18	SB-SO-38-OI	Oxford Instrument Analytical ED 2000	61.4	194.1	0.9		114.3	111.8
18	SB-SO-41-OI	Oxford Instrument Analytical ED 2000	50.1	219.4	3.2	0.2	114.4	103.7
18	SB-SO-47-OI	Oxford Instrument Analytical ED 2000	53.4	204.1	0.1	2.3	114.1	102.5
18	SB-SO-51-OI	Oxford Instrument Analytical ED 2000	52.9	194.8	0.3	1.3	140.7	95.3
19	SB-SO-05-XX	Reference Laboratory	540.0	200.0	1.3 U	1.3 U	61.0	80.0
19	SB-SO-18-XX	Reference Laboratory	280.0	210.0	1.3 U	1.3 U	70.0	84.0
19	SB-SO-30-XX	Reference Laboratory	290.0	120.0	1.3 J+	1.3 U	43.0	50.0
19	SB-SO-40-XX	Reference Laboratory	280.0	180.0	1.3 U	1.3 U	58.0	74.0
19	SB-SO-53-XX	Reference Laboratory	270.0	200.0	1.3 U	1.3 U	64.0	81.0
19	SB-SO-05-OI	Oxford Instrument Analytical ED 2000	363.2	187.6	0.3		125.6	120.6
19	SB-SO-18-OI	Oxford Instrument Analytical ED 2000	361.0	190.6		0.6	122.1	94.3
19	SB-SO-30-OI	Oxford Instrument Analytical ED 2000	371.6	195.7	3.8	1.0	123.0	95.1
19	SB-SO-40-OI	Oxford Instrument Analytical ED 2000	361.1	195.2	2.7	1.8	125.0	104.2
19	SB-SO-53-OI	Oxford Instrument Analytical ED 2000	351.9	185.3	1.0	0.1	112.2	99.7

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
20	SB-SO-08-XX	Reference Laboratory	5.4 J-	13.0	0.5 U	120.0	39.0	32000.0	17.0
20	SB-SO-11-XX	Reference Laboratory	5.7 J-	13.0	0.5 U	140.0	46.0	36000.0	20.0
20	SB-SO-21-XX	Reference Laboratory	4.9 J	13.0	0.5 U	130.0	43.0	34000.0	18.0
20	SB-SO-39-XX	Reference Laboratory	4.7 J-	13.0	0.5 U	140.0	46.0	34000.0	19.0
20	SB-SO-42-XX	Reference Laboratory	4.6 J-	13.0	0.5 U	140.0	45.0	35000.0	18.0
20	SB-SO-08-OI	Oxford Instrument Analytical ED 2000	35.3	33.7	1.6	213.3	46.1	65310.0	14.8
20	SB-SO-11-OI	Oxford Instrument Analytical ED 2000	42.9	57.6	0.4	198.0	60.1	58240.0	29.7
20	SB-SO-21-OI	Oxford Instrument Analytical ED 2000	36.7	20.6		220.3	47.2	64410.0	23.1
20	SB-SO-39-OI	Oxford Instrument Analytical ED 2000	40.4	35.7	0.2	209.7	45.7	65030.0	24.3
20	SB-SO-42-OI	Oxford Instrument Analytical ED 2000	34.9	56.7	1.3	220.4	44.4	61850.0	17.2
21	SB-SO-22-XX	Reference Laboratory	10.0 J	18.0	0.5 U	120.0	37.0	29000.0	22.0
21	SB-SO-25-XX	Reference Laboratory	6.8 J+	18.0	0.5 U	120.0	37.0	29000.0	22.0
21	SB-SO-27-XX	Reference Laboratory	6.7 J+	18.0	0.5 U	120.0	37.0	29000.0	22.0
21	SB-SO-35-XX	Reference Laboratory	6.0 J+	17.0	0.5 U	110.0	35.0	28000.0	21.0
21	SB-SO-44-XX	Reference Laboratory	6.8 J+	18.0	0.5 U	120.0	37.0	29000.0	22.0
21	SB-SO-22-OI	Oxford Instrument Analytical ED 2000	67.8	56.1	1.7	175.9	54.9	57150.0	21.3
21	SB-SO-25-OI	Oxford Instrument Analytical ED 2000	69.9	52.5		170.4	37.6	57149.0	23.8
21	SB-SO-27-OI	Oxford Instrument Analytical ED 2000	67.6	41.5		178.3	42.5	57690.0	26.9
21	SB-SO-35-OI	Oxford Instrument Analytical ED 2000	66.6	43.7	1.7	176.9	43.3	57840.0	20.1
21	SB-SO-44-OI	Oxford Instrument Analytical ED 2000	69.6	51.9		186.6	39.2	56090.0	18.2
22	SB-SO-23-XX	Reference Laboratory	48.0 J-	37.0	0.1 U	21.0	7.0	4500.0	36.0
22	SB-SO-28-XX	Reference Laboratory	42.0 J-	36.0	0.1 U	21.0	7.0	4400.0	36.0
22	SB-SO-32-XX	Reference Laboratory	46.0 J-	40.0	0.1 U	23.0	7.6	4900.0	40.0
22	SB-SO-43-XX	Reference Laboratory	40.0 J-	35.0	0.1 U	20.0	6.7	4200.0	34.0
22	SB-SO-48-XX	Reference Laboratory	39.0 J-	36.0	0.1 U	21.0	6.9	4500.0	36.0
22	SB-SO-23-OI	Oxford Instrument Analytical ED 2000	134.0	129.3	0.3	74.6	5.7	24750.0	46.7
22	SB-SO-28-OI	Oxford Instrument Analytical ED 2000	183.9	132.1		96.2	21.0	24490.0	38.5
22	SB-SO-32-OI	Oxford Instrument Analytical ED 2000	180.4	126.1	0.8	86.3	19.5	24360.0	35.9
22	SB-SO-43-OI	Oxford Instrument Analytical ED 2000	141.4	134.3		73.8	18.3	24590.0	46.4
22	SB-SO-48-OI	Oxford Instrument Analytical ED 2000	198.3	132.2	2.2	86.2	10.4	24410.0	31.3
23	SB-SO-02-XX	Reference Laboratory	44.0 J-	23.0 J-	0.5 U	130.0	43.0	35000.0	22.0 J-
23	SB-SO-07-XX	Reference Laboratory	45.0 J	22.0	0.5 U	120.0	38.0	35000.0	23.0
23	SB-SO-10-XX	Reference Laboratory	62.0 J	26.0	0.5 U	140.0	44.0	41000.0	27.0
23	SB-SO-26-XX	Reference Laboratory	61.0 J	30.0	0.5 U	160.0	50.0	46000.0	31.0
23	SB-SO-50-XX	Reference Laboratory	57.0 J	27.0	0.5 U	140.0	46.0	42000.0	28.0
23	SB-SO-02-OI	Oxford Instrument Analytical ED 2000	262.9	39.7	1.6	224.9	61.3	75760.0	28.4
23	SB-SO-07-OI	Oxford Instrument Analytical ED 2000	250.4	40.9		207.3	48.8	72940.0	30.5
23	SB-SO-10-OI	Oxford Instrument Analytical ED 2000	254.2	40.6		207.7	48.5	72930.0	25.1
23	SB-SO-26-OI	Oxford Instrument Analytical ED 2000	254.2	56.3	0.9	212.8	46.9	70140.0	28.2
23	SB-SO-50-OI	Oxford Instrument Analytical ED 2000	251.2	48.1	1.1	227.5	52.3	72860.0	22.7

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn	
20	SB-SO-08-XX	Reference Laboratory	730.0	180.0	1.3 U	1.3 U	57.0	70.0	
20	SB-SO-11-XX	Reference Laboratory	810.0	200.0	1.3 U	1.3 U	66.0	84.0	
20	SB-SO-21-XX	Reference Laboratory	740.0	190.0	1.3 U	1.3 U	58.0	75.0	
20	SB-SO-39-XX	Reference Laboratory	790.0	200.0	1.3 U	1.3 U	62.0	77.0	
20	SB-SO-42-XX	Reference Laboratory	740.0	200.0	1.3 U	1.3 U	65.0	78.0	
20	SB-SO-08-OI	Oxford Instrument Analytical ED 2000	1034.4	197.9	1.3		45.7	93.1	
20	SB-SO-11-OI	Oxford Instrument Analytical ED 2000	937.1	177.1	2.7	1.4	82.1	80.2	
20	SB-SO-21-OI	Oxford Instrument Analytical ED 2000	965.4	175.8	4.9		69.9	87.4	
20	SB-SO-39-OI	Oxford Instrument Analytical ED 2000	1007.1	183.5	2.8	1.5	82.0	93.0	
20	SB-SO-42-OI	Oxford Instrument Analytical ED 2000	1028.1	197.2	1.5	0.4	67.0	83.7	
21	SB-SO-22-XX	Reference Laboratory	3300.0	160.0	1.3 U	1.3 U	52.0	64.0	J-
21	SB-SO-25-XX	Reference Laboratory	3000.0	160.0	1.3 U	1.3 U	54.0	63.0	
21	SB-SO-27-XX	Reference Laboratory	3100.0	170.0	1.3 U	1.3 U	54.0	65.0	
21	SB-SO-35-XX	Reference Laboratory	3100.0	160.0	1.3 U	1.3 U	50.0	62.0	
21	SB-SO-44-XX	Reference Laboratory	3000.0	170.0	1.3 U	1.3 U	53.0	64.0	
21	SB-SO-22-OI	Oxford Instrument Analytical ED 2000	2328.2	174.5	2.6	1.3	71.8	74.9	
21	SB-SO-25-OI	Oxford Instrument Analytical ED 2000	2285.3	159.0			65.3	84.4	
21	SB-SO-27-OI	Oxford Instrument Analytical ED 2000	2323.4	170.3	0.7		62.5	82.4	
21	SB-SO-35-OI	Oxford Instrument Analytical ED 2000	2390.0	161.1	2.1	0.4	46.9	88.9	
21	SB-SO-44-OI	Oxford Instrument Analytical ED 2000	2260.0	165.4	0.3	0.5	78.6	85.6	
22	SB-SO-23-XX	Reference Laboratory	8500.0	26.0	0.2 J	0.3 UJ	13.0	8.4	
22	SB-SO-28-XX	Reference Laboratory	8800.0	26.0	0.3 U	0.3 UJ	13.0	7.8	
22	SB-SO-32-XX	Reference Laboratory	8900.0	28.0	0.4	0.1 UJ	14.0	8.5	
22	SB-SO-43-XX	Reference Laboratory	7600.0	24.0	0.3 U	0.3 UJ	13.0	8.3	
22	SB-SO-48-XX	Reference Laboratory	8200.0	25.0	0.3 U	0.1 UJ	13.0	7.8	
22	SB-SO-23-OI	Oxford Instrument Analytical ED 2000	7042.3	38.6	0.2			4.7	
22	SB-SO-28-OI	Oxford Instrument Analytical ED 2000	7317.5	36.7		0.1		7.2	
22	SB-SO-32-OI	Oxford Instrument Analytical ED 2000	7973.4	43.1	1.7			30.2	
22	SB-SO-43-OI	Oxford Instrument Analytical ED 2000	7597.2	45.4	4.1	1.0		24.6	
22	SB-SO-48-OI	Oxford Instrument Analytical ED 2000	7886.8	38.9	3.4	1.0		1.8	
23	SB-SO-02-XX	Reference Laboratory	130.0 J+	180.0	1.2 U	1.2 UJ	59.0	88.0	
23	SB-SO-07-XX	Reference Laboratory	270.0	170.0	1.4	1.6	53.0	86.0	
23	SB-SO-10-XX	Reference Laboratory	220.0	200.0	2.8	1.8	59.0	100.0	
23	SB-SO-26-XX	Reference Laboratory	260.0	220.0	3.4	1.8	68.0	110.0	
23	SB-SO-50-XX	Reference Laboratory	200.0	200.0	2.9	1.8	61.0	100.0	
23	SB-SO-02-OI	Oxford Instrument Analytical ED 2000	132.1	197.3	3.4		115.6	128.4	
23	SB-SO-07-OI	Oxford Instrument Analytical ED 2000	130.4	180.6		0.6	118.8	121.3	
23	SB-SO-10-OI	Oxford Instrument Analytical ED 2000	115.1	192.0	2.5	1.3	108.5	134.2	
23	SB-SO-26-OI	Oxford Instrument Analytical ED 2000	136.6	192.6	2.0	0.9	116.9	116.5	
23	SB-SO-50-OI	Oxford Instrument Analytical ED 2000	137.7	197.0	0.8		94.9	107.6	

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
24	SB-SO-01-XX	Reference Laboratory	180.0 J	65.0	0.5 U	140.0	46.0	47000.0	30.0
24	SB-SO-16-XX	Reference Laboratory	170.0 J	64.0	0.5 U	140.0	45.0	47000.0	30.0
24	SB-SO-24-XX	Reference Laboratory	180.0 J	66.0	0.5 U	150.0	49.0	49000.0	32.0
24	SB-SO-45-XX	Reference Laboratory	180.0 J	63.0	0.5 U	140.0	45.0	47000.0	30.0
24	SB-SO-52-XX	Reference Laboratory	150.0 J	62.0	0.5 U	140.0	47.0	46000.0	29.0
24	SB-SO-01-OI	Oxford Instrument Analytical ED 2000	658.0	71.4		210.4	44.1	77950.0	31.6
24	SB-SO-16-OI	Oxford Instrument Analytical ED 2000	620.5	62.8	1.9	213.2	44.0	81100.0	25.7
24	SB-SO-24-OI	Oxford Instrument Analytical ED 2000	639.1	66.7		215.3	47.4	81380.0	25.6
24	SB-SO-45-OI	Oxford Instrument Analytical ED 2000	617.7	88.3		202.0	55.4	79520.0	26.6
24	SB-SO-52-OI	Oxford Instrument Analytical ED 2000	620.4	89.9	0.0	214.7	60.4	79290.0	21.5
25	SB-SO-13-XX	Reference Laboratory	430.0 J	160.0	1.0 U	140.0	46.0	61000.0	36.0
25	SB-SO-19-XX	Reference Laboratory	310.0 J	100.0	0.5 U	100.0	32.0	42000.0	25.0
25	SB-SO-33-XX	Reference Laboratory	350.0 J	110.0	0.5 U	100.0	33.0	45000.0	28.0
25	SB-SO-37-XX	Reference Laboratory	340.0 J	130.0	1.0 U	120.0	39.0	51000.0	31.0
25	SB-SO-55-XX	Reference Laboratory	340.0 J	120.0	0.5 U	120.0	37.0	49000.0	29.0
25	SB-SO-13-OI	Oxford Instrument Analytical ED 2000	1252.9	146.2	0.7	181.3	50.4	91850.0	27.4
25	SB-SO-19-OI	Oxford Instrument Analytical ED 2000	1357.7	143.9		172.0	52.2	93070.0	32.4
25	SB-SO-33-OI	Oxford Instrument Analytical ED 2000	1345.4	158.3		171.8	45.7	93530.0	30.9
25	SB-SO-37-OI	Oxford Instrument Analytical ED 2000	1331.8	151.4		174.9	49.0	94500.0	38.5
25	SB-SO-55-OI	Oxford Instrument Analytical ED 2000	1319.1	153.9	1.1	179.9	46.2	90560.0	36.2
26	SB-SO-12-XX	Reference Laboratory	620.0 J	190.0	1.0 U	100.0	33.0	55000.0	43.0
26	SB-SO-15-XX	Reference Laboratory	600.0 J-	170.0 J-	1.0 U	91.0 J-	30.0 J-	51000.0 J-	40.0 J-
26	SB-SO-17-XX	Reference Laboratory	800.0 J+	210.0	1.0 U	110.0	37.0	61000.0	48.0
26	SB-SO-46-XX	Reference Laboratory	740.0 J+	190.0	1.0 U	120.0	35.0	57000.0	47.0
26	SB-SO-54-XX	Reference Laboratory	280.0	31.0	0.2 U	25.0	5.8	8600.0	5.2 J-
26	SB-SO-12-OI	Oxford Instrument Analytical ED 2000	2219.2	201.1		183.9	50.3	108480.0	52.4
26	SB-SO-15-OI	Oxford Instrument Analytical ED 2000	2192.0	217.4		169.8	39.2	107370.0	46.9
26	SB-SO-17-OI	Oxford Instrument Analytical ED 2000	2189.3	224.6		166.7	43.3	105190.0	52.2
26	SB-SO-46-OI	Oxford Instrument Analytical ED 2000	2178.1	225.5		178.1	47.2	104380.0	54.7
26	SB-SO-54-OI	Oxford Instrument Analytical ED 2000	2176.4	231.9	1.1	177.7	50.2	104310.0	43.6
27	KP-SE-08-XX	Reference Laboratory	6.2	2.6	0.1 U	88.0	3.8	840.0	300.0 J-
27	KP-SE-11-XX	Reference Laboratory	5.6	2.6	0.1 U	96.0	4.1	940.0	310.0 J-
27	KP-SE-17-XX	Reference Laboratory	4.9	2.6	0.1 U	98.0	4.1	940.0	300.0 J-
27	KP-SE-25-XX	Reference Laboratory	6.0	2.8	0.1 U	99.0	4.3	960.0	310.0 J-
27	KP-SE-30-XX	Reference Laboratory	5.7	2.8	0.1 U	83.0	3.6	830.0	300.0 J-
27	KP-SE-08-OI	Oxford Instrument Analytical ED 2000	2.1	40.7		210.8	7.8	21030.0	360.6
27	KP-SE-11-OI	Oxford Instrument Analytical ED 2000	5.2	46.8	0.4	212.3	1.0	20980.0	349.1
27	KP-SE-17-OI	Oxford Instrument Analytical ED 2000	5.7	49.0		198.0	5.7	20860.0	327.6
27	KP-SE-25-OI	Oxford Instrument Analytical ED 2000	6.7	44.1	0.9	208.8	6.0	20970.0	325.5
27	KP-SE-30-OI	Oxford Instrument Analytical ED 2000	4.7	49.5		223.0	0.3	21170.0	327.7

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
24	SB-SO-01-XX	Reference Laboratory	400.0	190.0	1.8	2.3	65.0	95.0
24	SB-SO-16-XX	Reference Laboratory	480.0	190.0	1.9	2.2	65.0	97.0
24	SB-SO-24-XX	Reference Laboratory	420.0	200.0	2.5	2.3	67.0	95.0
24	SB-SO-45-XX	Reference Laboratory	450.0	190.0	2.8	2.1 J-	63.0	93.0
24	SB-SO-52-XX	Reference Laboratory	430.0	190.0	1.8	2.2	64.0	90.0
24	SB-SO-01-OI	Oxford Instrument Analytical ED 2000	213.8	167.6			132.3	109.0
24	SB-SO-16-OI	Oxford Instrument Analytical ED 2000	225.3	191.1	3.4		97.2	98.4
24	SB-SO-24-OI	Oxford Instrument Analytical ED 2000	218.3	171.5	0.8	0.2	97.6	117.9
24	SB-SO-45-OI	Oxford Instrument Analytical ED 2000	235.7	168.6	1.9	0.4	75.2	103.0
24	SB-SO-52-OI	Oxford Instrument Analytical ED 2000	230.6	183.4	1.1	0.0	131.2	105.5
25	SB-SO-13-XX	Reference Laboratory	850.0	180.0	4.4	2.2 UJ	74.0	70.0
25	SB-SO-19-XX	Reference Laboratory	740.0	120.0	2.5	1.8	51.0	51.0
25	SB-SO-33-XX	Reference Laboratory	870.0	130.0	3.0	2.0 J	52.0	56.0
25	SB-SO-37-XX	Reference Laboratory	790.0	150.0	2.5 U	2.0 UJ	63.0	58.0
25	SB-SO-55-XX	Reference Laboratory	900.0	140.0	2.5	2.2 J	61.0	60.0
25	SB-SO-13-OI	Oxford Instrument Analytical ED 2000	401.0	160.1	1.5		120.4	82.8
25	SB-SO-19-OI	Oxford Instrument Analytical ED 2000	387.4	166.2	1.1	0.0	109.8	64.0
25	SB-SO-33-OI	Oxford Instrument Analytical ED 2000	392.4	164.7	3.5	0.3	137.2	87.9
25	SB-SO-37-OI	Oxford Instrument Analytical ED 2000	407.6	164.0	1.8	1.2	115.3	68.9
25	SB-SO-55-OI	Oxford Instrument Analytical ED 2000	393.4	155.2	1.0	1.8	96.7	72.4
26	SB-SO-12-XX	Reference Laboratory	1400.0	110.0	2.5 U	2.1 UJ	59.0	42.0
26	SB-SO-15-XX	Reference Laboratory	1100.0	100.0 J-	3.4	1.6 UJ	52.0 J-	36.0 J-
26	SB-SO-17-XX	Reference Laboratory	1200.0	120.0	2.8	2.3 UJ	60.0	42.0
26	SB-SO-46-XX	Reference Laboratory	670.0	120.0	2.6	2.2 UJ	57.0	41.0
26	SB-SO-54-XX	Reference Laboratory	560.0	20.0	0.5 U	0.5 UJ	11.0	6.0
26	SB-SO-12-OI	Oxford Instrument Analytical ED 2000	558.4	132.9	1.8	2.8	83.9	62.9
26	SB-SO-15-OI	Oxford Instrument Analytical ED 2000	534.2	119.9	1.4		93.2	84.8
26	SB-SO-17-OI	Oxford Instrument Analytical ED 2000	531.5	128.1	0.4	0.6	111.0	67.5
26	SB-SO-46-OI	Oxford Instrument Analytical ED 2000	525.9	127.3	1.4	0.1	97.9	69.8
26	SB-SO-54-OI	Oxford Instrument Analytical ED 2000	560.7	117.0	0.1		84.3	69.9
27	KP-SE-08-XX	Reference Laboratory	0.1 U	42.0	0.3 U	0.3 UJ	3.7	4.8
27	KP-SE-11-XX	Reference Laboratory	0.1 U	46.0	0.4	0.3 UJ	4.0	5.7
27	KP-SE-17-XX	Reference Laboratory	0.1 U	47.0	0.3 U	0.3 UJ	4.0	4.7
27	KP-SE-25-XX	Reference Laboratory	0.1 U	47.0	0.3 U	0.3 UJ	4.1	4.8
27	KP-SE-30-XX	Reference Laboratory	0.1 U	39.0	0.2 U	0.3 UJ	3.6	4.5
27	KP-SE-08-OI	Oxford Instrument Analytical ED 2000		59.4	1.2	0.7	37.4	15.6
27	KP-SE-11-OI	Oxford Instrument Analytical ED 2000	5.5	59.9	0.2	0.0	33.0	11.8
27	KP-SE-17-OI	Oxford Instrument Analytical ED 2000	3.3	58.7	2.2	0.5	38.9	7.5
27	KP-SE-25-OI	Oxford Instrument Analytical ED 2000	2.0	58.8	1.3	0.1	30.2	2.8
27	KP-SE-30-OI	Oxford Instrument Analytical ED 2000		63.1	1.9	1.1	40.3	7.4

Appendix D. Analytical Data Summary, Oxford ED2000 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	1.5	0.1 U	34.0	2.2	480.0	310.0 J-
28	KP-SE-12-XX	Reference Laboratory	3.1	1.5	0.1 U	42.0	2.5	510.0	320.0 J-
28	KP-SE-14-XX	Reference Laboratory	11.0 J-	1.9	0.1 U	46.0 J-	2.7 J+	520.0 J-	680.0 J-
28	KP-SE-19-XX	Reference Laboratory	3.0	1.5	0.1 U	44.0	2.3	510.0	330.0
28	KP-SE-28-XX	Reference Laboratory	3.3	1.6	0.1 U	45.0	2.3	520.0	320.0
28	KP-SE-01-OI	Oxford Instrument Analytical ED 2000	5.6	37.2	0.7	93.9	4.9	19990.0	329.8
28	KP-SE-12-OI	Oxford Instrument Analytical ED 2000	3.8	51.5		112.5	1.6	20080.0	339.9
28	KP-SE-14-OI	Oxford Instrument Analytical ED 2000		44.1		99.4	1.4	20040.0	358.2
28	KP-SE-19-OI	Oxford Instrument Analytical ED 2000	2.1	42.1		99.1		19920.0	314.4
28	KP-SE-28-OI	Oxford Instrument Analytical ED 2000	4.3	41.3		102.5	5.6	20040.0	326.1
29	TL-SE-04-XX	Reference Laboratory	1.2 U	9.8	0.5 U	62.0	1900.0	42000.0	32.0
29	TL-SE-10-XX	Reference Laboratory	1.2 U	10.0	0.5 U	64.0	2000.0	43000.0	35.0
29	TL-SE-12-XX	Reference Laboratory	1.2 U	10.0	0.5 U	66.0	2100.0	44000.0	34.0
29	TL-SE-15-XX	Reference Laboratory	1.2 U	8.8	0.5 U	54.0	1800.0	36000.0	28.0
29	TL-SE-20-XX	Reference Laboratory	1.2 U	10.0	0.5 U	64.0	2000.0	42000.0	32.0
29	TL-SE-24-XX	Reference Laboratory	1.2 U	11.0	0.5 U	67.0	2100.0	43000.0	37.0
29	TL-SE-26-XX	Reference Laboratory	1.2 U	9.9	0.5 U	62.0	2000.0	40000.0	34.0
29	TL-SE-04-OI	Oxford Instrument Analytical ED 2000		29.3	1.3	90.4	1975.7	83270.0	37.3
29	TL-SE-10-OI	Oxford Instrument Analytical ED 2000	5.3	12.6	0.1	75.1	1988.7	83230.0	53.6
29	TL-SE-12-OI	Oxford Instrument Analytical ED 2000		26.4	1.8	89.2	2023.3	80650.0	45.8
29	TL-SE-15-OI	Oxford Instrument Analytical ED 2000	1.1	35.2	2.2	74.0	2027.1	83220.0	27.0
29	TL-SE-20-OI	Oxford Instrument Analytical ED 2000	3.4	21.0	0.8	70.9	1973.1	83270.0	35.9
29	TL-SE-24-OI	Oxford Instrument Analytical ED 2000		17.6		81.8	2021.3	82930.0	46.9
29	TL-SE-26-OI	Oxford Instrument Analytical ED 2000	1.7	17.4	0.0	72.8	1971.2	81600.0	43.6
30	TL-SE-03-XX	Reference Laboratory	2.5 U	9.3	1.0 U	91.0	1600.0	63000.0	12.0
30	TL-SE-19-XX	Reference Laboratory	2.5 U	9.6	1.0 U	96.0	1700.0	66000.0	13.0
30	TL-SE-23-XX	Reference Laboratory	2.5 U	9.1	1.0 U	92.0	1600.0	64000.0	12.0
30	TL-SE-25-XX	Reference Laboratory	2.5 U	9.9	1.0 U	91.0	1600.0	62000.0	11.0
30	TL-SE-31-XX	Reference Laboratory	2.5 U	10.0	1.0 U	110.0	1800.0	74000.0	13.0
30	TL-SE-03-OI	Oxford Instrument Analytical ED 2000	2.4	36.0		105.8	1647.9	118620.0	
30	TL-SE-19-OI	Oxford Instrument Analytical ED 2000		0.2		13.7	1633.6	18510.0	26.6
30	TL-SE-23-OI	Oxford Instrument Analytical ED 2000	0.2	19.4	0.0	95.9	1659.9	119440.0	20.6
30	TL-SE-25-OI	Oxford Instrument Analytical ED 2000		15.1	0.5	105.1	1655.3	122170.0	23.4
30	TL-SE-31-OI	Oxford Instrument Analytical ED 2000		46.6		98.4	1634.6	118700.0	13.0

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
28	KP-SE-01-XX	Reference Laboratory	0.1 U	16.0	0.3 U	0.3 UJ	2.3 J	5.8
28	KP-SE-12-XX	Reference Laboratory	0.1 U	20.0	0.3 U	0.3 UJ	2.4 J	7.7
28	KP-SE-14-XX	Reference Laboratory	0.1 U	23.0 J-	0.3 U	0.3 UJ	2.5 J	7.1
28	KP-SE-19-XX	Reference Laboratory	0.0 U	22.0	0.3 U	0.3 U	2.3 J	7.3
28	KP-SE-28-XX	Reference Laboratory	0.1 U	22.0	0.3 U	0.3 U	2.4 J	6.3
28	KP-SE-01-OI	Oxford Instrument Analytical ED 2000	3.7	27.2	2.2		29.8	15.2
28	KP-SE-12-OI	Oxford Instrument Analytical ED 2000	7.5	38.1	2.4	0.9	37.5	9.5
28	KP-SE-14-OI	Oxford Instrument Analytical ED 2000		24.9		0.7	26.5	0.6
28	KP-SE-19-OI	Oxford Instrument Analytical ED 2000		26.0	1.1	1.6	17.9	11.1
28	KP-SE-28-OI	Oxford Instrument Analytical ED 2000		32.3	2.8		30.7	5.6
29	TL-SE-04-XX	Reference Laboratory	0.3 J-	71.0	1.2 U	1.3	95.0	160.0
29	TL-SE-10-XX	Reference Laboratory	0.2 J-	72.0	1.2 U	1.2 U	95.0	160.0
29	TL-SE-12-XX	Reference Laboratory	0.2 J-	75.0	1.2 U	1.2 U	100.0	170.0
29	TL-SE-15-XX	Reference Laboratory	0.3 J-	63.0	1.2 U	1.0 U	84.0	140.0
29	TL-SE-20-XX	Reference Laboratory	0.3 J-	74.0	1.2 U	1.2 U	100.0	160.0
29	TL-SE-24-XX	Reference Laboratory	0.3 J-	77.0	1.2 U	1.3 U	100.0	170.0
29	TL-SE-26-XX	Reference Laboratory	0.2 J-	70.0	1.2 U	1.2 U	96.0	160.0
29	TL-SE-04-OI	Oxford Instrument Analytical ED 2000	3.5	93.3	1.6	1.7	76.1	165.7
29	TL-SE-10-OI	Oxford Instrument Analytical ED 2000	9.3	87.2	0.7	2.4	90.9	218.0
29	TL-SE-12-OI	Oxford Instrument Analytical ED 2000	10.1	96.7	3.7	3.8	61.1	203.6
29	TL-SE-15-OI	Oxford Instrument Analytical ED 2000	6.9	88.9	3.2	1.9	55.7	196.4
29	TL-SE-20-OI	Oxford Instrument Analytical ED 2000	8.7	92.4	1.3	3.4	83.7	189.4
29	TL-SE-24-OI	Oxford Instrument Analytical ED 2000	4.9	98.5	1.9	2.1	46.2	183.2
29	TL-SE-26-OI	Oxford Instrument Analytical ED 2000		95.6		3.7	95.7	177.0
30	TL-SE-03-XX	Reference Laboratory	0.3 J-	110.0	2.5 U	0.9 U	140.0	200.0
30	TL-SE-19-XX	Reference Laboratory	0.3 J-	120.0	2.5 U	1.1 U	150.0	210.0
30	TL-SE-23-XX	Reference Laboratory	0.4 J-	110.0	2.5 U	1.3 U	150.0	200.0
30	TL-SE-25-XX	Reference Laboratory	0.4 J-	110.0	2.5 U	0.9 U	150.0	200.0
30	TL-SE-31-XX	Reference Laboratory	0.6 J-	130.0	2.5 U	1.2 U	170.0	230.0
30	TL-SE-03-OI	Oxford Instrument Analytical ED 2000		139.7	1.8	1.8	114.8	218.9
30	TL-SE-19-OI	Oxford Instrument Analytical ED 2000	2.6	131.5	0.3	2.5	37.3	235.4
30	TL-SE-23-OI	Oxford Instrument Analytical ED 2000	1.6	135.5	1.7	3.2	108.5	240.2
30	TL-SE-25-OI	Oxford Instrument Analytical ED 2000		163.4		2.1	95.5	233.0
30	TL-SE-31-OI	Oxford Instrument Analytical ED 2000	3.7	136.4	0.6	2.0	91.7	232.3

Appendix D. Analytical Data Summary, Oxford ED2000 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	9.3	0.5 U	110.0	1400.0	19000.0	48.0 J-
31	TL-SE-11-XX	Reference Laboratory	1.2 UJ	15.0	0.5 U	140.0	1600.0	28000.0	54.0 J-
31	TL-SE-14-XX	Reference Laboratory	1.2 UJ	10.0	0.3 J	110.0	1500.0	18000.0	50.0 J-
31	TL-SE-18-XX	Reference Laboratory	1.2 UJ	9.9	0.5 U	150.0	1300.0	24000.0	46.0 J-
31	TL-SE-22-XX	Reference Laboratory	1.2 UJ	11.0	0.5 U	150.0	1700.0	26000.0	54.0 J-
31	TL-SE-27-XX	Reference Laboratory	1.2 UJ	10.0	0.3 J	130.0	1500.0	19000.0	51.0 J-
31	TL-SE-29-XX	Reference Laboratory	1.2 UJ	11.0	0.2 J	140.0	1600.0	23000.0	51.0 J-
31	TL-SE-01-OI	Oxford Instrument Analytical ED 2000	0.3	51.5	0.1	160.9	1671.8	70690.0	58.8
31	TL-SE-11-OI	Oxford Instrument Analytical ED 2000		25.4		162.9	1661.6	73470.0	62.4
31	TL-SE-14-OI	Oxford Instrument Analytical ED 2000		50.8		146.4	1734.2	71320.0	46.2
31	TL-SE-18-OI	Oxford Instrument Analytical ED 2000	3.7	46.9	0.1	154.5	1715.7	76820.0	59.5
31	TL-SE-22-OI	Oxford Instrument Analytical ED 2000	1.4	31.4	1.5	156.2	1681.0	77070.0	52.2
31	TL-SE-27-OI	Oxford Instrument Analytical ED 2000	1.9	34.2	0.7	168.8	1703.6	75080.0	51.1
31	TL-SE-29-OI	Oxford Instrument Analytical ED 2000		28.1	2.9	172.3	1704.2	75700.0	62.9
32	LV-SE-02-XX	Reference Laboratory	1.3 UJ	28.0	0.5 U	72.0	33.0	23000.0	20.0 J-
32	LV-SE-10-XX	Reference Laboratory	1.3 UJ	34.0	0.5 U	84.0	42.0	28000.0	25.0 J-
32	LV-SE-22-XX	Reference Laboratory	1.3 UJ	30.0	0.5 U	69.0	33.0	23000.0	22.0 J-
32	LV-SE-25-XX	Reference Laboratory	1.3 UJ	31.0	0.5 U	74.0	36.0	25000.0	23.0 J-
32	LV-SE-31-XX	Reference Laboratory	1.3 UJ	32.0	0.5 U	78.0	36.0	25000.0	49.0 J-
32	LV-SE-35-XX	Reference Laboratory	1.3 UJ	31.0 J-	0.5 U	74.0 J-	35.0	24000.0 J-	22.0 J-
32	LV-SE-50-XX	Reference Laboratory	2.5 U	29.0	1.0 U	74.0	34.0	24000.0	24.0 J-
32	LV-SE-02-OI	Oxford Instrument Analytical ED 2000	1.7	41.0	0.7	89.5	38.9	57320.0	33.5
32	LV-SE-10-OI	Oxford Instrument Analytical ED 2000	2.5	47.5		95.7	37.3	55750.0	38.6
32	LV-SE-22-OI	Oxford Instrument Analytical ED 2000	6.8	43.5		86.8	40.6	56620.0	38.2
32	LV-SE-25-OI	Oxford Instrument Analytical ED 2000	0.4	47.2		83.9	41.4	57560.0	37.4
32	LV-SE-31-OI	Oxford Instrument Analytical ED 2000	2.1	31.7	1.3	90.2	43.7	56340.0	40.1
32	LV-SE-35-OI	Oxford Instrument Analytical ED 2000	3.5	71.6		91.1	44.8	55180.0	36.0
32	LV-SE-50-OI	Oxford Instrument Analytical ED 2000	2.4	47.9		86.3	37.1	56140.0	29.2
33	LV-SE-12-XX	Reference Laboratory	2.6 U	190.0	1.0 U	55.0	34.0	72000.0	19.0 J-
33	LV-SE-26-XX	Reference Laboratory	2.6 U	220.0	1.0 U	64.0	39.0	83000.0	25.0 J-
33	LV-SE-33-XX	Reference Laboratory	2.6 U	170.0	1.0 U	52.0	31.0	66000.0	21.0 J-
33	LV-SE-39-XX	Reference Laboratory	2.6 U	190.0	1.0 U	58.0	35.0	74000.0	22.0 J-
33	LV-SE-42-XX	Reference Laboratory	2.7 U	170.0	1.1 U	50.0	30.0	65000.0	22.0 J-
33	LV-SE-12-OI	Oxford Instrument Analytical ED 2000	4.5	218.3	0.8	84.3	43.0	140770.0	22.6
33	LV-SE-26-OI	Oxford Instrument Analytical ED 2000	2.9	211.4	0.8	86.0	46.6	146680.0	26.4
33	LV-SE-33-OI	Oxford Instrument Analytical ED 2000	5.4	199.2	0.4	78.8	37.9	143070.0	19.9
33	LV-SE-39-OI	Oxford Instrument Analytical ED 2000	1.6	226.1	0.3	102.3	44.5	143440.0	47.9
33	LV-SE-42-OI	Oxford Instrument Analytical ED 2000	3.2	228.4	1.0	83.5	47.7	136600.0	34.7

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
31	TL-SE-01-XX	Reference Laboratory	0.1 U	180.0	1.2 U	5.7 J-	75.0	130.0
31	TL-SE-11-XX	Reference Laboratory	0.0 U	210.0	1.2 U	5.5 J-	85.0	140.0
31	TL-SE-14-XX	Reference Laboratory	0.1 U	180.0	1.2 U	5.7 J-	73.0	140.0
31	TL-SE-18-XX	Reference Laboratory	0.0 U	190.0	1.2 U	6.3 J-	70.0	120.0
31	TL-SE-22-XX	Reference Laboratory	0.1 U	210.0	1.2 U	6.5 J-	80.0	150.0
31	TL-SE-27-XX	Reference Laboratory	0.0 U	200.0	1.2 U	7.8 J-	67.0	140.0
31	TL-SE-29-XX	Reference Laboratory	0.1 U	200.0	1.2 U	5.9 J-	80.0	140.0
31	TL-SE-01-OI	Oxford Instrument Analytical ED 2000		213.1	2.0	3.7	96.0	155.9
31	TL-SE-11-OI	Oxford Instrument Analytical ED 2000	3.0	221.1	1.1	4.1	98.2	149.9
31	TL-SE-14-OI	Oxford Instrument Analytical ED 2000		193.0	1.4	5.7	60.1	160.3
31	TL-SE-18-OI	Oxford Instrument Analytical ED 2000		220.1	1.0	3.2	82.3	158.9
31	TL-SE-22-OI	Oxford Instrument Analytical ED 2000		216.6	1.6	5.0	81.3	194.1
31	TL-SE-27-OI	Oxford Instrument Analytical ED 2000	8.5	206.3		5.6	68.3	159.7
31	TL-SE-29-OI	Oxford Instrument Analytical ED 2000		223.5		3.8	79.5	162.5
32	LV-SE-02-XX	Reference Laboratory	0.0 U	160.0	3.8	1.3 UJ	53.0	65.0
32	LV-SE-10-XX	Reference Laboratory	0.0 U	200.0	4.7	1.3 UJ	66.0	77.0
32	LV-SE-22-XX	Reference Laboratory	1.1	170.0	5.2	1.3 UJ	51.0	66.0
32	LV-SE-25-XX	Reference Laboratory	1.0	170.0	5.1	1.3 UJ	56.0	70.0
32	LV-SE-31-XX	Reference Laboratory	1.0	180.0	5.1	1.3 UJ	58.0	70.0
32	LV-SE-35-XX	Reference Laboratory	1.4	170.0 J-	5.0	1.3 UJ	55.0 J-	67.0 J-
32	LV-SE-50-XX	Reference Laboratory	1.2	170.0	3.3	2.5 U	57.0	65.0
32	LV-SE-02-OI	Oxford Instrument Analytical ED 2000		143.8	6.0		77.4	94.3
32	LV-SE-10-OI	Oxford Instrument Analytical ED 2000	2.1	147.3	5.9		59.3	98.5
32	LV-SE-22-OI	Oxford Instrument Analytical ED 2000		143.4	3.2		109.5	95.1
32	LV-SE-25-OI	Oxford Instrument Analytical ED 2000		136.1	4.9		87.2	89.9
32	LV-SE-31-OI	Oxford Instrument Analytical ED 2000	9.4	145.7	4.1	0.6	70.0	83.1
32	LV-SE-35-OI	Oxford Instrument Analytical ED 2000	2.8	144.6	5.2	0.6	75.7	113.6
32	LV-SE-50-OI	Oxford Instrument Analytical ED 2000	3.0	131.1	5.1		84.4	95.9
33	LV-SE-12-XX	Reference Laboratory	5.6	71.0	3.0	2.6 U	72.0	66.0
33	LV-SE-26-XX	Reference Laboratory	6.0	83.0	6.1	2.6 U	86.0	75.0
33	LV-SE-33-XX	Reference Laboratory	6.8	66.0	2.8	2.6 U	67.0	59.0
33	LV-SE-39-XX	Reference Laboratory	8.0	74.0	5.1	2.6 U	74.0	66.0
33	LV-SE-42-XX	Reference Laboratory	4.3	67.0	3.4	2.7 U	64.0	57.0
33	LV-SE-12-OI	Oxford Instrument Analytical ED 2000	8.4	101.4	5.7	0.3	132.7	104.6
33	LV-SE-26-OI	Oxford Instrument Analytical ED 2000	6.2	104.3	4.6	0.3	124.5	82.3
33	LV-SE-33-OI	Oxford Instrument Analytical ED 2000		106.6	2.4	0.4	139.1	90.6
33	LV-SE-39-OI	Oxford Instrument Analytical ED 2000	3.5	103.3	4.3	0.4	103.7	86.5
33	LV-SE-42-OI	Oxford Instrument Analytical ED 2000	3.2	114.7	3.6	1.1	114.4	111.3

Appendix D. Analytical Data Summary, Oxford ED2000 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	450.0	2.7 U	48.0	34.0	150000.0	14.0 J-
34	LV-SE-19-XX	Reference Laboratory	6.7 U	500.0	2.7 U	55.0	37.0	160000.0	17.0 J-
34	LV-SE-27-XX	Reference Laboratory	6.7 U	530.0	2.7 U	56.0	39.0	180000.0	16.0 J-
34	LV-SE-36-XX	Reference Laboratory	6.7 U	550.0	2.7 U	60.0	40.0	180000.0	21.0 J-
34	LV-SE-38-XX	Reference Laboratory	6.7 U	480.0	2.7 U	52.0	36.0	160000.0	15.0 J-
34	LV-SE-09-OI	Oxford Instrument Analytical ED 2000	0.7	494.0	0.5	85.5	67.3	340860.0	21.6
34	LV-SE-19-OI	Oxford Instrument Analytical ED 2000	5.3	494.9		94.0	55.2	339190.0	28.8
34	LV-SE-27-OI	Oxford Instrument Analytical ED 2000	4.1	486.4	1.3	102.2	62.5	339390.0	17.0
34	LV-SE-36-OI	Oxford Instrument Analytical ED 2000	1.1	492.1	0.2	104.7	48.0	342120.0	16.2
34	LV-SE-38-OI	Oxford Instrument Analytical ED 2000	4.6	490.2	0.6	86.9	45.2	340830.0	19.0
35	LV-SE-07-XX	Reference Laboratory	6.7 UJ	780.0	2.7 U	57.0	48.0	200000.0	11.0
35	LV-SE-18-XX	Reference Laboratory	6.7 UJ	800.0	2.7 U	61.0	49.0	210000.0	11.0
35	LV-SE-23-XX	Reference Laboratory	6.6 UJ	660.0	2.6 U	53.0	40.0	170000.0	7.7
35	LV-SE-45-XX	Reference Laboratory	6.7 UJ	650.0	2.7 U	50.0	40.0	170000.0	7.6
35	LV-SE-48-XX	Reference Laboratory	6.6 UJ	680.0	2.6 U	52.0	42.0	180000.0	8.9
35	LV-SE-07-OI	Oxford Instrument Analytical ED 2000	5.2	751.6	0.9	98.0	69.0	464910.0	5.7
35	LV-SE-18-OI	Oxford Instrument Analytical ED 2000	2.9	731.7	1.1	107.4	61.5	451600.0	9.4
35	LV-SE-23-OI	Oxford Instrument Analytical ED 2000	5.2	717.7	0.4	100.5	60.8	456600.0	13.6
35	LV-SE-45-OI	Oxford Instrument Analytical ED 2000		742.2	1.1	99.6	69.4	449880.0	
35	LV-SE-48-OI	Oxford Instrument Analytical ED 2000	0.5	733.3		139.2	69.8	447660.0	
36	LV-SE-01-XX	Reference Laboratory	1.5 UJ	6.1	0.8	4.4	18.0	1100.0	17.0
36	LV-SE-14-XX	Reference Laboratory	1.5 UJ	5.0	0.7	4.2	16.0	980.0	14.0
36	LV-SE-21-XX	Reference Laboratory	1.5 UJ	6.5	0.8	4.4	19.0	970.0	18.0
36	LV-SE-24-XX	Reference Laboratory	1.5 UJ	4.9	0.7	3.9	15.0	840.0	14.0
36	LV-SE-32-XX	Reference Laboratory	1.4 UJ	5.6	0.9	4.4	16.0	860.0	14.0
36	LV-SE-01-OI	Oxford Instrument Analytical ED 2000		8.6		16.9	15.7	19400.0	15.0
36	LV-SE-14-OI	Oxford Instrument Analytical ED 2000		1.1	0.6	17.1	14.0	19390.0	13.9
36	LV-SE-21-OI	Oxford Instrument Analytical ED 2000	2.3		0.5	15.9	18.3	19380.0	20.3
36	LV-SE-24-OI	Oxford Instrument Analytical ED 2000	0.9	1.7		16.0	16.3	19430.0	18.2
36	LV-SE-32-OI	Oxford Instrument Analytical ED 2000	1.4			15.9	11.7	19410.0	16.3
37	LV-SE-08-XX	Reference Laboratory	1.3 UJ	30.0	0.5 U	54.0	23.0	23000.0	55.0
37	LV-SE-16-XX	Reference Laboratory	1.3 UJ	29.0	0.5 U	53.0	22.0	22000.0	53.0
37	LV-SE-28-XX	Reference Laboratory	1.3 UJ	31.0	0.5 U	59.0	25.0	25000.0	59.0
37	LV-SE-30-XX	Reference Laboratory	1.3 UJ	30.0	0.5 U	58.0	25.0	24000.0	58.0
37	LV-SE-47-XX	Reference Laboratory	1.3 UJ	31.0	0.5 U	56.0	23.0	23000.0	57.0
37	LV-SE-08-OI	Oxford Instrument Analytical ED 2000	11.1	60.4	0.0	86.7	28.9	59160.0	84.0
37	LV-SE-16-OI	Oxford Instrument Analytical ED 2000	10.9	60.2	1.9	86.2	34.9	55510.0	89.9
37	LV-SE-28-OI	Oxford Instrument Analytical ED 2000	8.2	63.8	1.3	85.2	33.7	58660.0	81.6
37	LV-SE-30-OI	Oxford Instrument Analytical ED 2000	10.3	49.0		86.1	38.5	59350.0	75.3
37	LV-SE-47-OI	Oxford Instrument Analytical ED 2000	11.2	52.2	0.5	86.1	30.2	58020.0	83.6

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
34	LV-SE-09-XX	Reference Laboratory	6.0	55.0	6.7 U	6.7 U	100.0	51.0 J
34	LV-SE-19-XX	Reference Laboratory	7.2	65.0	5.9 J	6.7 U	110.0	55.0 J
34	LV-SE-27-XX	Reference Laboratory	11.0	64.0	6.7 U	6.7 U	120.0	58.0 J
34	LV-SE-36-XX	Reference Laboratory	8.5	70.0	11.0	6.7 U	120.0	60.0 J
34	LV-SE-38-XX	Reference Laboratory	7.9	75.0	6.7 U	6.7 U	100.0	54.0 J
34	LV-SE-09-OI	Oxford Instrument Analytical ED 2000	7.0	141.2	3.6		135.0	87.0
34	LV-SE-19-OI	Oxford Instrument Analytical ED 2000	2.2	134.0	0.0		166.8	98.2
34	LV-SE-27-OI	Oxford Instrument Analytical ED 2000	4.4	133.2	2.8	0.2	141.3	91.2
34	LV-SE-36-OI	Oxford Instrument Analytical ED 2000		130.3	0.6		134.6	84.8
34	LV-SE-38-OI	Oxford Instrument Analytical ED 2000	13.7	136.4	6.4	1.5	166.7	89.5
35	LV-SE-07-XX	Reference Laboratory	5.5	58.0	10.0	6.7 U	130.0	24.0 J
35	LV-SE-18-XX	Reference Laboratory	5.4	60.0	12.0	6.7 U	140.0	52.0 J
35	LV-SE-23-XX	Reference Laboratory	5.0	50.0 J	9.6	6.6 U	120.0	18.0 J
35	LV-SE-45-XX	Reference Laboratory	5.6	50.0 J	8.2	6.7 U	120.0	19.0 J
35	LV-SE-48-XX	Reference Laboratory	7.3	50.0 J	7.6	6.6 U	120.0	30.0 J
35	LV-SE-07-OI	Oxford Instrument Analytical ED 2000		133.3	4.5	0.7	193.4	66.1
35	LV-SE-18-OI	Oxford Instrument Analytical ED 2000	2.8	151.7	7.5	0.0	142.1	64.9
35	LV-SE-23-OI	Oxford Instrument Analytical ED 2000	2.3	135.1	4.2		210.5	56.4
35	LV-SE-45-OI	Oxford Instrument Analytical ED 2000	4.1	174.7	4.1		240.1	85.0
35	LV-SE-48-OI	Oxford Instrument Analytical ED 2000	4.3	151.3	2.2	0.3	184.6	68.1
36	LV-SE-01-XX	Reference Laboratory	0.1 U	49.0	1.5 U	1.5 U	1.6 J	14.0 J
36	LV-SE-14-XX	Reference Laboratory	0.1 U	46.0	1.5 U	1.5 U	1.4 J	12.0 J
36	LV-SE-21-XX	Reference Laboratory	0.0 U	49.0	1.5 U	1.5 U	1.6 J	14.0 J
36	LV-SE-24-XX	Reference Laboratory	0.1 U	44.0	1.5 U	1.5 U	1.4 J	12.0 J
36	LV-SE-32-XX	Reference Laboratory	0.1 U	47.0	1.4 U	1.4 U	1.3 J	19.0
36	LV-SE-01-OI	Oxford Instrument Analytical ED 2000	0.8	45.1	2.7	0.3	34.0	16.7
36	LV-SE-14-OI	Oxford Instrument Analytical ED 2000	3.2	46.2	1.8		35.9	19.1
36	LV-SE-21-OI	Oxford Instrument Analytical ED 2000		39.1	0.3	0.7	37.0	18.2
36	LV-SE-24-OI	Oxford Instrument Analytical ED 2000	1.3	48.0		0.2	38.6	18.5
36	LV-SE-32-OI	Oxford Instrument Analytical ED 2000		47.0	1.0		31.9	20.1
37	LV-SE-08-XX	Reference Laboratory	5.2	110.0	4.8	1.3 U	44.0	61.0
37	LV-SE-16-XX	Reference Laboratory	5.4	110.0	5.0	1.3 U	42.0	59.0
37	LV-SE-28-XX	Reference Laboratory	5.4	120.0	5.8	1.3 U	48.0	65.0
37	LV-SE-30-XX	Reference Laboratory	6.3	120.0	5.6	1.3 U	48.0	66.0
37	LV-SE-47-XX	Reference Laboratory	4.9	120.0	4.2	1.3 U	45.0	65.0
37	LV-SE-08-OI	Oxford Instrument Analytical ED 2000		126.0	6.8	0.6	72.8	84.7
37	LV-SE-16-OI	Oxford Instrument Analytical ED 2000		122.8	3.8	0.8	41.2	95.0
37	LV-SE-28-OI	Oxford Instrument Analytical ED 2000	1.1	129.7	6.2	1.3	59.0	92.8
37	LV-SE-30-OI	Oxford Instrument Analytical ED 2000	6.5	137.2	4.3	0.3	72.9	79.2
37	LV-SE-47-OI	Oxford Instrument Analytical ED 2000	7.4	132.0	5.1		49.9	103.3

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
38	LV-SE-11-XX	Reference Laboratory	1.4 UJ	150.0	6.6	120.0	270.0	42000.0	7.2
38	LV-SE-29-XX	Reference Laboratory	1.4 UJ	150.0	6.3	120.0	260.0	42000.0	7.2 J+
38	LV-SE-44-XX	Reference Laboratory	1.4 U	140.0	6.1	120.0	250.0	40000.0	7.8
38	LV-SE-46-XX	Reference Laboratory	0.9 U	110.0	5.0	92.0	200.0	32000.0	5.9
38	LV-SE-52-XX	Reference Laboratory	1.4 U	160.0	6.8	130.0	280.0	44000.0	7.8
38	LV-SE-11-OI	Oxford Instrument Analytical ED 2000		153.6	5.3	133.3	271.0	57390.0	22.5
38	LV-SE-29-OI	Oxford Instrument Analytical ED 2000		160.3	5.9	121.0	265.8	54730.0	15.5
38	LV-SE-44-OI	Oxford Instrument Analytical ED 2000	3.5	162.4	7.0	123.9	284.5	52330.0	18.1
38	LV-SE-46-OI	Oxford Instrument Analytical ED 2000		171.0	4.8	123.2	272.4	54660.0	7.6
38	LV-SE-52-OI	Oxford Instrument Analytical ED 2000	2.3	155.4	8.0	127.1	276.7	51000.0	26.2
39	RF-SE-07-XX	Reference Laboratory	1.3 U	12.0	0.5 U	92.0	81.0	17000.0	24.0
39	RF-SE-12-XX	Reference Laboratory	1.2 U	14.0	0.5 U	100.0	110.0	20000.0	25.0
39	RF-SE-23-XX	Reference Laboratory	0.3 U	0.3 U	0.1 U	0.3 U	0.2 U	3.9 J	0.3 U
39	RF-SE-36-XX	Reference Laboratory	1.2 U	12.0	0.5 U	91.0	82.0	17000.0	22.0
39	RF-SE-42-XX	Reference Laboratory	1.3 UJ	14.0	0.6	110.0	95.0	19000.0	28.0
39	RF-SE-45-XX	Reference Laboratory	1.3 UJ	15.0	0.5 U	110.0	100.0	21000.0	33.0
39	RF-SE-53-XX	Reference Laboratory	1.3 UJ	14.0	0.6 U	110.0	95.0	19000.0	28.0
39	RF-SE-07-OI	Oxford Instrument Analytical ED 2000	1.5	20.3	0.6	125.4	116.6	48420.0	42.3
39	RF-SE-12-OI	Oxford Instrument Analytical ED 2000	0.8	24.6	0.7	104.7	106.5	47030.0	43.9
39	RF-SE-23-OI	Oxford Instrument Analytical ED 2000	1.7	19.6	0.3	37.7	109.5	24450.0	50.7
39	RF-SE-36-OI	Oxford Instrument Analytical ED 2000	0.1	22.2	2.5	119.0	110.3	48460.0	45.4
39	RF-SE-42-OI	Oxford Instrument Analytical ED 2000	4.1	27.2	2.6	126.5	99.3	46700.0	40.5
39	RF-SE-45-OI	Oxford Instrument Analytical ED 2000		32.0	2.0	115.7	117.6	47550.0	44.2
39	RF-SE-53-OI	Oxford Instrument Analytical ED 2000	0.0	21.0	0.4	120.9	115.5	46950.0	51.3
40	RF-SE-03-XX	Reference Laboratory	1.2 UJ	27.0	1.3	93.0	200.0	17000.0	88.0
40	RF-SE-28-XX	Reference Laboratory	1.2 UJ	31.0	1.5	100.0	220.0	18000.0	99.0
40	RF-SE-38-XX	Reference Laboratory	1.2 UJ	27.0	1.2	90.0	190.0	16000.0	83.0
40	RF-SE-49-XX	Reference Laboratory	1.2 UJ	31.0	1.5	100.0	220.0	18000.0	97.0
40	RF-SE-55-XX	Reference Laboratory	1.2 UJ	24.0	1.1	91.0	180.0	15000.0	75.0
40	RF-SE-03-OI	Oxford Instrument Analytical ED 2000	4.3	46.2	2.0	111.0	235.8	44980.0	92.3
40	RF-SE-28-OI	Oxford Instrument Analytical ED 2000	1.0	50.6	2.0	102.5	238.1	44870.0	100.3
40	RF-SE-38-OI	Oxford Instrument Analytical ED 2000	2.8	49.9	2.1	118.2	233.2	44630.0	108.8
40	RF-SE-49-OI	Oxford Instrument Analytical ED 2000	1.9	57.5	2.6	101.6	227.5	44250.0	92.4
40	RF-SE-55-OI	Oxford Instrument Analytical ED 2000	3.5	47.1	0.2	119.8	242.3	44690.0	107.0

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
38	LV-SE-11-XX	Reference Laboratory	2.8	870.0	1.3 U	1.4 U	35.0	200.0
38	LV-SE-29-XX	Reference Laboratory	1.5 J-	860.0	1.2 U	1.4 U	35.0	200.0
38	LV-SE-44-XX	Reference Laboratory	1.5	830.0	1.4 U	1.4 U	34.0	190.0
38	LV-SE-46-XX	Reference Laboratory	1.4	660.0	0.9 U	0.9 U	27.0	150.0
38	LV-SE-52-XX	Reference Laboratory	21.0	910.0	1.4 U	1.4 U	38.0	210.0
38	LV-SE-11-OI	Oxford Instrument Analytical ED 2000	1.4	889.8	2.4		63.6	238.2
38	LV-SE-29-OI	Oxford Instrument Analytical ED 2000	3.0	852.1	0.5	0.6	75.5	240.0
38	LV-SE-44-OI	Oxford Instrument Analytical ED 2000	0.6	823.3	2.5		63.4	226.9
38	LV-SE-46-OI	Oxford Instrument Analytical ED 2000		856.5	1.4		46.6	224.6
38	LV-SE-52-OI	Oxford Instrument Analytical ED 2000		835.9	0.9		53.0	215.1
39	RF-SE-07-XX	Reference Laboratory	0.1 U	180.0	1.3 U	1.3 U	34.0	130.0
39	RF-SE-12-XX	Reference Laboratory	0.1 U	210.0	1.2 U	1.2 U	38.0	140.0
39	RF-SE-23-XX	Reference Laboratory	2.4	2.0 U	0.3 U	0.4	2.5 U	0.6 U
39	RF-SE-36-XX	Reference Laboratory	0.1 U	180.0	1.0 U	1.2 U	34.0	120.0
39	RF-SE-42-XX	Reference Laboratory	0.1 U	210.0	1.3 U	1.3 U	40.0	140.0
39	RF-SE-45-XX	Reference Laboratory	0.1 U	220.0	1.3 U	1.3 U	43.0	150.0
39	RF-SE-53-XX	Reference Laboratory	0.1 U	210.0	1.3 U	1.3 U	40.0	140.0
39	RF-SE-07-OI	Oxford Instrument Analytical ED 2000		185.6	1.3	0.9	59.1	172.4
39	RF-SE-12-OI	Oxford Instrument Analytical ED 2000		172.5	2.9	0.8	66.4	153.8
39	RF-SE-23-OI	Oxford Instrument Analytical ED 2000	1.6	175.4	2.3	2.5	47.8	150.9
39	RF-SE-36-OI	Oxford Instrument Analytical ED 2000		195.0	1.5	1.7	58.8	161.4
39	RF-SE-42-OI	Oxford Instrument Analytical ED 2000	8.8	176.6	3.1	0.5	66.5	159.5
39	RF-SE-45-OI	Oxford Instrument Analytical ED 2000		181.4	6.2	0.7	82.8	172.7
39	RF-SE-53-OI	Oxford Instrument Analytical ED 2000	0.0	176.6	0.4	0.7	54.7	179.7
40	RF-SE-03-XX	Reference Laboratory	0.5	150.0	1.2 U	1.2 U	40.0	300.0
40	RF-SE-28-XX	Reference Laboratory	0.6	160.0	1.2 U	1.2 U	44.0	320.0
40	RF-SE-38-XX	Reference Laboratory	0.4	140.0	1.2 U	1.2 U	39.0	300.0
40	RF-SE-49-XX	Reference Laboratory	0.4	170.0	1.2 U	1.2 U	43.0	330.0
40	RF-SE-55-XX	Reference Laboratory	0.4	140.0	1.2 U	1.2 U	35.0	280.0
40	RF-SE-03-OI	Oxford Instrument Analytical ED 2000		147.0	2.9	1.8	69.9	343.9
40	RF-SE-28-OI	Oxford Instrument Analytical ED 2000	1.9	129.6	1.0	0.5	72.7	344.4
40	RF-SE-38-OI	Oxford Instrument Analytical ED 2000	2.3	135.5	2.0	1.8	85.7	357.9
40	RF-SE-49-OI	Oxford Instrument Analytical ED 2000	0.3	135.7	2.4	3.0	55.4	375.3
40	RF-SE-55-OI	Oxford Instrument Analytical ED 2000	11.5	134.2	2.2	0.0	44.0	335.7

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
41	RF-SE-06-XX	Reference Laboratory	1.3 UJ	70.0	3.6	90.0	490.0	20000.0	230.0
41	RF-SE-13-XX	Reference Laboratory	1.3 UJ	76.0	3.7	92.0	530.0	21000.0	230.0
41	RF-SE-27-XX	Reference Laboratory	1.3 UJ	64.0	3.1	78.0	440.0	18000.0	200.0
41	RF-SE-31-XX	Reference Laboratory	1.3 UJ	39.0	1.8	63.0	250.0	12000.0	120.0
41	RF-SE-58-XX	Reference Laboratory	1.3 UJ	71.0	3.6	89.0	500.0	21000.0	230.0
41	RF-SE-06-OI	Oxford Instrument Analytical ED 2000	3.9	100.9	2.7	95.8	542.9	46580.0	243.5
41	RF-SE-13-OI	Oxford Instrument Analytical ED 2000	4.2	111.3	4.1	97.2	537.3	46500.0	237.4
41	RF-SE-27-OI	Oxford Instrument Analytical ED 2000	4.1	122.4	3.8	92.0	540.1	46340.0	226.6
41	RF-SE-31-OI	Oxford Instrument Analytical ED 2000	4.3	110.8	1.9	94.0	521.5	45460.0	217.1
41	RF-SE-58-OI	Oxford Instrument Analytical ED 2000		115.4	2.3	102.1	537.1	46260.0	242.9
42	RF-SE-02-XX	Reference Laboratory	1.3 UJ	110.0	5.4	93.0	740.0	24000.0	330.0
42	RF-SE-22-XX	Reference Laboratory	1.3 UJ	99.0	4.7	84.0	670.0	22000.0	300.0
42	RF-SE-25-XX	Reference Laboratory	1.3 UJ	88.0	4.0	78.0	580.0	19000.0	270.0
42	RF-SE-30-XX	Reference Laboratory	1.3 UJ	89.0	4.3	78.0	610.0	21000.0	290.0
42	RF-SE-57-XX	Reference Laboratory	1.3 UJ	89.0	4.5	79.0	610.0	21000.0	300.0
42	RF-SE-02-OI	Oxford Instrument Analytical ED 2000	4.6	129.5	3.7	97.2	705.1	48620.0	314.7
42	RF-SE-22-OI	Oxford Instrument Analytical ED 2000	8.4	150.3	2.2	50.5	717.7	31760.0	312.1
42	RF-SE-25-OI	Oxford Instrument Analytical ED 2000	7.6	152.8	3.5	89.3	703.3	48040.0	289.2
42	RF-SE-30-OI	Oxford Instrument Analytical ED 2000	4.2	166.1	4.1	88.0	713.4	46570.0	296.4
42	RF-SE-57-OI	Oxford Instrument Analytical ED 2000	8.2	131.4	5.3	86.5	720.1	48400.0	313.8
43	RF-SE-15-XX	Reference Laboratory	1.3 UJ	120.0	6.2	72.0	820.0	23000.0	390.0
43	RF-SE-24-XX	Reference Laboratory	1.3 UJ	130.0 J+	6.5 J+	74.0 J+	860.0 J+	24000.0 J+	410.0 J+
43	RF-SE-32-XX	Reference Laboratory	1.3 UJ	120.0	5.1	64.0	770.0	20000.0	330.0
43	RF-SE-43-XX	Reference Laboratory	1.3 UJ	130.0	5.7	68.0	840.0	22000.0	350.0
43	RF-SE-59-XX	Reference Laboratory	1.3 UJ	140.0	5.9	73.0	890.0	23000.0	380.0
43	RF-SE-15-OI	Oxford Instrument Analytical ED 2000	9.3	209.8	6.4	116.6	983.2	49510.0	418.7
43	RF-SE-24-OI	Oxford Instrument Analytical ED 2000	6.7	192.6	7.7	78.6	965.8	51360.0	413.2
43	RF-SE-32-OI	Oxford Instrument Analytical ED 2000	10.8	197.3	6.0	90.1	932.8	49800.0	392.0
43	RF-SE-43-OI	Oxford Instrument Analytical ED 2000	3.4	192.1	7.5	84.4	960.4	50410.0	397.7
43	RF-SE-59-OI	Oxford Instrument Analytical ED 2000	10.4	182.2	6.7	96.5	947.4	50550.0	432.7
44	RF-SE-05-XX	Reference Laboratory	4.1 J+	160.0	9.1	69.0	1000.0	26000.0	450.0
44	RF-SE-26-XX	Reference Laboratory	2.2 J+	140.0	8.4	64.0	990.0	23000.0	440.0
44	RF-SE-39-XX	Reference Laboratory	2.9 J+	160.0	9.3	73.0	1100.0	26000.0	490.0
44	RF-SE-44-XX	Reference Laboratory	2.7 J+	140.0	8.2	64.0	970.0	24000.0	420.0
44	RF-SE-56-XX	Reference Laboratory	3.5 J+	180.0	9.6	75.0	1200.0	27000.0	490.0
44	RF-SE-05-OI	Oxford Instrument Analytical ED 2000	8.8	216.1	7.3	87.4	1159.9	51340.0	467.5
44	RF-SE-26-OI	Oxford Instrument Analytical ED 2000	6.1	220.8	6.4	83.4	1136.0	51710.0	445.0
44	RF-SE-39-OI	Oxford Instrument Analytical ED 2000	7.0	235.8	7.3	79.3	1155.4	52140.0	448.9
44	RF-SE-44-OI	Oxford Instrument Analytical ED 2000	4.7	216.8	9.2	96.1	1179.8	52130.0	433.9
44	RF-SE-56-OI	Oxford Instrument Analytical ED 2000	11.0	240.4	9.8	89.3	1168.7	53460.0	439.5

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
41	RF-SE-06-XX	Reference Laboratory	1.1	150.0	1.3 U	1.3 U	44.0	740.0
41	RF-SE-13-XX	Reference Laboratory	1.2	160.0	1.3 U	1.3	45.0	790.0
41	RF-SE-27-XX	Reference Laboratory	1.2	130.0	1.3 U	1.3 U	39.0	670.0
41	RF-SE-31-XX	Reference Laboratory	1.1	86.0	1.3 U	1.3 U	28.0	420.0
41	RF-SE-58-XX	Reference Laboratory	1.2	150.0	1.3 U	1.3 U	46.0	770.0
41	RF-SE-06-OI	Oxford Instrument Analytical ED 2000		151.8	5.4	3.4	70.5	792.5
41	RF-SE-13-OI	Oxford Instrument Analytical ED 2000		136.4	1.7	2.4	75.8	791.5
41	RF-SE-27-OI	Oxford Instrument Analytical ED 2000		135.1	1.9	4.7	71.1	785.2
41	RF-SE-31-OI	Oxford Instrument Analytical ED 2000		136.8	2.0	3.1	70.0	761.6
41	RF-SE-58-OI	Oxford Instrument Analytical ED 2000		130.0	2.1	3.6	63.6	790.2
42	RF-SE-02-XX	Reference Laboratory	1.6	180.0	1.3 U	2.7	50.0	1100.0
42	RF-SE-22-XX	Reference Laboratory	1.7	160.0	1.3 U	2.3	44.0	990.0
42	RF-SE-25-XX	Reference Laboratory	1.5	140.0	1.5	1.7	40.0	890.0
42	RF-SE-30-XX	Reference Laboratory	1.5	150.0	1.3 U	1.9	44.0	960.0
42	RF-SE-57-XX	Reference Laboratory	1.5	150.0	2.0	2.2	44.0	1000.0
42	RF-SE-02-OI	Oxford Instrument Analytical ED 2000		142.6	1.9	4.4	66.3	1006.3
42	RF-SE-22-OI	Oxford Instrument Analytical ED 2000	1.6	145.2	1.9	5.2	48.6	1005.6
42	RF-SE-25-OI	Oxford Instrument Analytical ED 2000	1.7	148.2	1.6	4.6	69.3	1010.9
42	RF-SE-30-OI	Oxford Instrument Analytical ED 2000		138.9	4.7	6.0	77.1	1045.4
42	RF-SE-57-OI	Oxford Instrument Analytical ED 2000	7.6	144.8	4.5	3.7	74.6	1034.5
43	RF-SE-15-XX	Reference Laboratory	2.6	160.0	1.4	3.6	45.0	1300.0
43	RF-SE-24-XX	Reference Laboratory	2.3	170.0 J+	1.3 U	3.8 J+	46.0 J+	1400.0 J-
43	RF-SE-32-XX	Reference Laboratory	2.8	140.0	1.3 U	4.2	36.0	1100.0
43	RF-SE-43-XX	Reference Laboratory	2.7	150.0	1.3 U	4.0	40.0	1200.0
43	RF-SE-59-XX	Reference Laboratory	0.1 U	160.0	1.3 U	4.5	42.0	1300.0
43	RF-SE-15-OI	Oxford Instrument Analytical ED 2000	0.6	154.3	5.9	5.3	73.6	1361.1
43	RF-SE-24-OI	Oxford Instrument Analytical ED 2000		146.9	2.5	5.9	97.5	1347.2
43	RF-SE-32-OI	Oxford Instrument Analytical ED 2000	17.8	158.3	1.9	6.4	68.3	1365.8
43	RF-SE-43-OI	Oxford Instrument Analytical ED 2000	2.3	161.6	2.6	6.2	72.6	1318.2
43	RF-SE-59-OI	Oxford Instrument Analytical ED 2000		161.3	2.5	5.9	64.9	1384.3
44	RF-SE-05-XX	Reference Laboratory	2.6	150.0	3.1	7.4 J-	48.0	1800.0
44	RF-SE-26-XX	Reference Laboratory	2.5	140.0	2.8	7.2 J-	42.0	1700.0
44	RF-SE-39-XX	Reference Laboratory	2.2	150.0	2.6	8.2 J-	49.0	1900.0
44	RF-SE-44-XX	Reference Laboratory	2.3	140.0	2.4	7.2 J-	44.0	1600.0
44	RF-SE-56-XX	Reference Laboratory	2.2	160.0	1.8	8.3 J-	51.0	1900.0
44	RF-SE-05-OI	Oxford Instrument Analytical ED 2000	13.4	142.3	3.8	8.1	67.7	1716.7
44	RF-SE-26-OI	Oxford Instrument Analytical ED 2000		135.2	3.4	7.2	85.8	1809.6
44	RF-SE-39-OI	Oxford Instrument Analytical ED 2000	11.8	131.5	0.6	9.1	85.1	1768.3
44	RF-SE-44-OI	Oxford Instrument Analytical ED 2000		140.6	2.4	5.4	85.1	1724.8
44	RF-SE-56-OI	Oxford Instrument Analytical ED 2000	11.2	139.3	3.0	5.4	89.1	1796.4

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
45	RF-SE-04-XX	Reference Laboratory	3.2 J+	230.0	12.0	42.0	1500.0	27000.0	730.0
45	RF-SE-14-XX	Reference Laboratory	4.4 J+	260.0	12.0	47.0	1700.0	30000.0	800.0
45	RF-SE-19-XX	Reference Laboratory	3.7 J+	250.0	13.0	48.0	1700.0	30000.0	800.0
45	RF-SE-34-XX	Reference Laboratory	2.9 J+	210.0	10.0	39.0	1400.0	24000.0	660.0
45	RF-SE-52-XX	Reference Laboratory	3.4 J+	220.0	11.0	42.0	1500.0	26000.0	720.0
45	RF-SE-04-OI	Oxford Instrument Analytical ED 2000	9.2	342.9	10.3	73.4	1608.9	54830.0	677.1
45	RF-SE-14-OI	Oxford Instrument Analytical ED 2000	9.0	346.8	10.4	51.4	1587.0	54920.0	688.4
45	RF-SE-19-OI	Oxford Instrument Analytical ED 2000	9.0	338.8	9.8	69.7	1620.4	55290.0	679.8
45	RF-SE-34-OI	Oxford Instrument Analytical ED 2000	12.3	338.5	9.7	60.2	1650.1	55250.0	709.1
45	RF-SE-52-OI	Oxford Instrument Analytical ED 2000	13.7	339.0	12.2	50.9	1640.8	55440.0	694.5
46	BN-SO-11-XX	Reference Laboratory	4.0 J-	2900.0	720.0	820.0	120.0	23000.0	56.0
46	BN-SO-14-XX	Reference Laboratory	3.5 J-	2800.0	690.0	800.0	120.0	22000.0	51.0
46	BN-SO-23-XX	Reference Laboratory	1.2 UJ	2800.0	700.0	800.0	120.0	23000.0	52.0
46	BN-SO-11-OI	Oxford Instrument Analytical ED 2000	6.5	3235.5	508.5	430.3	127.7	38940.0	63.3
46	BN-SO-14-OI	Oxford Instrument Analytical ED 2000	3.2	3231.5	532.5	439.9	157.0	39550.0	79.5
46	BN-SO-23-OI	Oxford Instrument Analytical ED 2000	2.7	3221.1	537.7	415.1	137.4	38650.0	61.0
47	BN-SO-09-XX	Reference Laboratory	750.0 J-	97.0	2700.0	2900.0	100.0	22000.0	4700.0
47	BN-SO-12-XX	Reference Laboratory	750.0 J-	89.0	2600.0	2800.0	96.0	21000.0	4500.0
47	BN-SO-24-XX	Reference Laboratory	810.0 J-	97.0	2900.0	3000.0	100.0	23000.0	4900.0
47	BN-SO-09-OI	Oxford Instrument Analytical ED 2000	1713.5	733.8	1618.2	1567.5	107.1	42410.0	4126.8
47	BN-SO-12-OI	Oxford Instrument Analytical ED 2000	1534.4	957.9	1548.2	1620.3	159.7	40760.0	4642.8
47	BN-SO-24-OI	Oxford Instrument Analytical ED 2000	1643.9	669.7	1538.9	1617.5	112.8	42800.0	4074.6
48	SB-SO-09-XX	Reference Laboratory	1.3 UJ	8.9	0.5 U	130.0	120.0	35000.0	19.0
48	SB-SO-20-XX	Reference Laboratory	1.3 UJ	11.0	0.5 U	170.0	150.0	44000.0	24.0
48	SB-SO-31-XX	Reference Laboratory	1.3 UJ	8.0 J-	0.5 U	140.0	130.0	38000.0	21.0
48	SB-SO-09-OI	Oxford Instrument Analytical ED 2000	8.0	19.7	1.0	219.8	130.6	70440.0	9.3
48	SB-SO-20-OI	Oxford Instrument Analytical ED 2000	9.5	9.6	0.1	240.3	110.0	72670.0	14.4
48	SB-SO-31-OI	Oxford Instrument Analytical ED 2000	5.3	11.2		210.7	108.4	72080.0	19.2
49	SB-SO-29-XX	Reference Laboratory	1.2 U	9.4	0.5 U	140.0	130.0	41000.0	19.0
49	SB-SO-36-XX	Reference Laboratory	1.2 U	7.8	0.5 U	120.0	100.0	33000.0	15.0
49	SB-SO-56-XX	Reference Laboratory	1.2 U	9.5	0.5 U	150.0	140.0	42000.0	20.0
49	SB-SO-29-OI	Oxford Instrument Analytical ED 2000	5.3		0.1	239.1	151.7	72470.0	26.9
49	SB-SO-36-OI	Oxford Instrument Analytical ED 2000	3.4	18.3	1.4	228.1	160.7	73250.0	24.9
49	SB-SO-56-OI	Oxford Instrument Analytical ED 2000	7.8	9.7	0.2	225.7	141.3	71990.0	33.8

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
45	RF-SE-04-XX	Reference Laboratory	4.2	130.0	2.8	12.0 J-	46.0	2400.0
45	RF-SE-14-XX	Reference Laboratory	4.7	140.0	3.0	13.0 J-	51.0	2600.0
45	RF-SE-19-XX	Reference Laboratory	3.9	140.0	4.1	14.0 J-	52.0	2700.0
45	RF-SE-34-XX	Reference Laboratory	4.5	120.0	1.9	10.0 J-	42.0	2200.0
45	RF-SE-52-XX	Reference Laboratory	4.1	130.0	2.0	11.0 J-	47.0	2300.0
45	RF-SE-04-OI	Oxford Instrument Analytical ED 2000	0.2	148.8	3.3	10.7	103.5	2329.1
45	RF-SE-14-OI	Oxford Instrument Analytical ED 2000	7.8	138.9	2.5	8.4	74.8	2343.8
45	RF-SE-19-OI	Oxford Instrument Analytical ED 2000	15.0	138.1	0.8	10.1	72.0	2311.1
45	RF-SE-34-OI	Oxford Instrument Analytical ED 2000	8.7	126.9	2.2	8.8	65.5	2389.6
45	RF-SE-52-OI	Oxford Instrument Analytical ED 2000	8.0	144.0	3.9	8.6	79.9	2322.1
46	BN-SO-11-XX	Reference Laboratory	24.0 J-	2900.0	140.0	140.0 J-	150.0	3900.0
46	BN-SO-14-XX	Reference Laboratory	26.0	2800.0	130.0	140.0 J-	150.0	3800.0
46	BN-SO-23-XX	Reference Laboratory	31.0	2800.0	130.0	130.0 J-	150.0	3800.0
46	BN-SO-11-OI	Oxford Instrument Analytical ED 2000	26.5	2888.6	96.0	87.9	121.0	4647.0
46	BN-SO-14-OI	Oxford Instrument Analytical ED 2000	21.3	2895.5	92.9	90.8	119.5	4559.5
46	BN-SO-23-OI	Oxford Instrument Analytical ED 2000	33.2	2819.1	100.5	96.5	126.3	4642.6
47	BN-SO-09-XX	Reference Laboratory	0.4	1500.0	290.0	100.0 J-	340.0	81.0
47	BN-SO-12-XX	Reference Laboratory	0.3	1400.0	290.0	210.0 J-	310.0	74.0
47	BN-SO-24-XX	Reference Laboratory	0.4	1600.0	300.0	140.0 J-	350.0	81.0
47	BN-SO-09-OI	Oxford Instrument Analytical ED 2000		1459.2	209.6	171.7	267.3	93.2
47	BN-SO-12-OI	Oxford Instrument Analytical ED 2000		1878.4	257.7	166.0	243.8	167.9
47	BN-SO-24-OI	Oxford Instrument Analytical ED 2000		1429.0	207.0	165.5	268.1	126.9
48	SB-SO-09-XX	Reference Laboratory	30.0	2900.0	26.0	160.0 J-	120.0	3600.0
48	SB-SO-20-XX	Reference Laboratory	10.0	3700.0	30.0	140.0 J-	160.0	4500.0
48	SB-SO-31-XX	Reference Laboratory	32.0	3200.0 J-	28.0 J-	160.0 J-	140.0	3900.0 J-
48	SB-SO-09-OI	Oxford Instrument Analytical ED 2000	43.0	3248.3	19.7	308.7	216.3	4268.1
48	SB-SO-20-OI	Oxford Instrument Analytical ED 2000	33.8	3202.3	20.2	293.6	223.2	4300.9
48	SB-SO-31-OI	Oxford Instrument Analytical ED 2000	33.4	3169.4	20.2	304.6	206.2	4260.1
49	SB-SO-29-XX	Reference Laboratory	7.9 J	200.0	160.0	1.2 UJ	400.0	3900.0
49	SB-SO-36-XX	Reference Laboratory	36.0	160.0	130.0	1.2 UJ	320.0	3200.0
49	SB-SO-56-XX	Reference Laboratory	9.0	210.0	160.0	1.2 UJ	410.0	4100.0
49	SB-SO-29-OI	Oxford Instrument Analytical ED 2000	51.8	215.9	104.4		473.7	4359.3
49	SB-SO-36-OI	Oxford Instrument Analytical ED 2000	47.2	209.6	98.3	0.6	445.6	4279.2
49	SB-SO-56-OI	Oxford Instrument Analytical ED 2000	29.2	208.2	104.2	0.0	428.4	4217.8

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
50	SB-SO-04-XX	Reference Laboratory	940.0	13.0	2800.0	2800.0	100.0	38000.0	21.0
50	SB-SO-34-XX	Reference Laboratory	980.0	12.0	2500.0	2500.0	91.0	34000.0	18.0
50	SB-SO-49-XX	Reference Laboratory	700.0	12.0	2500.0	2400.0	89.0	33000.0	18.0
50	SB-SO-04-OI	Oxford Instrument Analytical ED 2000	2840.8	0.7	2609.2	2687.1	104.9	66020.0	26.7
50	SB-SO-34-OI	Oxford Instrument Analytical ED 2000	2769.4		2560.6	2666.4	73.3	64740.0	32.3
50	SB-SO-49-OI	Oxford Instrument Analytical ED 2000	2771.2	9.5	2577.6	2645.2	83.0	68740.0	24.9
51	WS-SO-07-XX	Reference Laboratory	3.8	53.0	1.9	640.0	4400.0	25000.0	1700.0
51	WS-SO-11-XX	Reference Laboratory	1.2 U	46.0	1.4	570.0	3900.0	19000.0	1500.0
51	WS-SO-25-XX	Reference Laboratory	1.2 U	59.0	3.1	730.0	4900.0	24000.0	1900.0
51	WS-SO-07-OI	Oxford Instrument Analytical ED 2000	4.5	324.4	4.4	704.5	4304.9	55080.0	1800.8
51	WS-SO-11-OI	Oxford Instrument Analytical ED 2000	2.1	304.9	1.9	690.1	4328.2	55110.0	1848.5
51	WS-SO-25-OI	Oxford Instrument Analytical ED 2000	2.8	315.1	0.2	401.2	4460.6	41300.0	1906.3
52	WS-SO-10-XX	Reference Laboratory	1.3 U	83.0	1.8	67.0	76.0	19000.0	1900.0
52	WS-SO-20-XX	Reference Laboratory	1.3 U	100.0	1.9	81.0	90.0	23000.0	2300.0
52	WS-SO-23-XX	Reference Laboratory	1.3 U	110.0	2.1	82.0	96.0	23000.0	2500.0
52	WS-SO-10-OI	Oxford Instrument Analytical ED 2000	5.0	441.3	3.1	113.0	133.7	55940.0	2286.2
52	WS-SO-20-OI	Oxford Instrument Analytical ED 2000	1.4	425.9	1.9	98.8	125.3	55170.0	2397.2
52	WS-SO-23-OI	Oxford Instrument Analytical ED 2000	1.7	430.3	2.2	112.9	123.9	55130.0	2312.8
53	AS-SO-03-XX	Reference Laboratory	1.2 U	14.0	1300.0	33.0	6200.0	15000.0	160.0
53	AS-SO-05-XX	Reference Laboratory	1.2 U	9.3	900.0	23.0	4500.0	11000.0	110.0
53	AS-SO-08-XX	Reference Laboratory	1.2 U	10.0	930.0	24.0	4600.0	11000.0	120.0
53	AS-SO-03-OI	Oxford Instrument Analytical ED 2000	4.8	28.3	895.3	52.7	4414.7	34450.0	181.0
53	AS-SO-05-OI	Oxford Instrument Analytical ED 2000	7.6	40.0	869.5	50.3	4292.8	33500.0	154.4
53	AS-SO-08-OI	Oxford Instrument Analytical ED 2000	6.1	43.0	882.2	60.4	4184.1	33910.0	154.2
54	LV-SO-03-XX	Reference Laboratory	1.6	42.0	590.0	600.0	130.0	24000.0	94.0
54	LV-SO-40-XX	Reference Laboratory	2.7	42.0	580.0	590.0	130.0	24000.0	92.0
54	LV-SO-49-XX	Reference Laboratory	7.4	43.0	600.0	610.0	130.0	25000.0	98.0
54	LV-SO-03-OI	Oxford Instrument Analytical ED 2000	19.7	69.2	550.3	637.4	146.4	68020.0	134.0
54	LV-SO-40-OI	Oxford Instrument Analytical ED 2000	22.5	82.6	560.5	674.5	144.5	67730.0	103.3
54	LV-SO-49-OI	Oxford Instrument Analytical ED 2000	12.6	91.7	533.1	677.3	138.9	69260.0	117.1
55	LV-SO-04-XX	Reference Laboratory	860.0	120.0	2400.0	2300.0	98.0	22000.0	4000.0
55	LV-SO-34-XX	Reference Laboratory	870.0 J-	110.0 J-	2300.0 J-	2200.0 J-	87.0	20000.0 J-	3700.0 J-
55	LV-SO-37-XX	Reference Laboratory	590.0	84.0	1700.0	1600.0	66.0	16000.0	2800.0
55	LV-SO-04-OI	Oxford Instrument Analytical ED 2000	2249.8	700.9	2254.8	2169.5	117.1	66090.0	4103.5
55	LV-SO-34-OI	Oxford Instrument Analytical ED 2000	2200.1	744.2	2242.9	2390.5	123.8	66320.0	3982.0
55	LV-SO-37-OI	Oxford Instrument Analytical ED 2000	2156.3	768.8	2184.4	2515.7	151.4	67770.0	4052.7

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
50	SB-SO-04-XX	Reference Laboratory	40.0	3300.0	390.0	1.3 UJ	58.0	86.0
50	SB-SO-34-XX	Reference Laboratory	36.0	3000.0	360.0	1.3 UJ	52.0	77.0
50	SB-SO-49-XX	Reference Laboratory	36.0	2800.0	330.0	1.2 UJ	52.0	72.0
50	SB-SO-04-OI	Oxford Instrument Analytical ED 2000	50.1	3088.3	247.4	0.8	110.6	99.4
50	SB-SO-34-OI	Oxford Instrument Analytical ED 2000	51.4	2983.5	251.0		79.1	88.1
50	SB-SO-49-OI	Oxford Instrument Analytical ED 2000	42.7	3000.1	254.2		93.4	81.1
51	WS-SO-07-XX	Reference Laboratory	0.3	260.0	1.2 U	400.0 J-	48.0	180.0
51	WS-SO-11-XX	Reference Laboratory	0.3	240.0	1.2 U	340.0 J-	43.0	160.0
51	WS-SO-25-XX	Reference Laboratory	0.3	300.0	1.2 U	450.0 J-	54.0	200.0
51	WS-SO-07-OI	Oxford Instrument Analytical ED 2000		332.2		363.8	84.6	199.6
51	WS-SO-11-OI	Oxford Instrument Analytical ED 2000		329.5		361.4	103.4	194.7
51	WS-SO-25-OI	Oxford Instrument Analytical ED 2000		307.0	1.4	306.9	52.2	193.1
52	WS-SO-10-XX	Reference Laboratory	0.1 U	290.0	280.0	1.3 UJ	260.0	1900.0
52	WS-SO-20-XX	Reference Laboratory	0.1 U	350.0	340.0	1.3 UJ	320.0	2300.0
52	WS-SO-23-XX	Reference Laboratory	0.1 U	380.0	360.0	1.3 UJ	330.0	2500.0
52	WS-SO-10-OI	Oxford Instrument Analytical ED 2000		404.4	243.4		449.2	3045.9
52	WS-SO-20-OI	Oxford Instrument Analytical ED 2000		411.0	246.4		447.8	3116.2
52	WS-SO-23-OI	Oxford Instrument Analytical ED 2000		395.9	243.0		417.3	3017.2
53	AS-SO-03-XX	Reference Laboratory	3.7 J-	520.0	200.0	480.0 J-	29.0	350.0
53	AS-SO-05-XX	Reference Laboratory	2.5 J-	370.0	140.0	330.0 J-	23.0	250.0
53	AS-SO-08-XX	Reference Laboratory	2.5 J-	380.0	140.0	280.0 J-	23.0	260.0
53	AS-SO-03-OI	Oxford Instrument Analytical ED 2000	8.5	472.8	116.3	311.7	45.6	282.4
53	AS-SO-05-OI	Oxford Instrument Analytical ED 2000		429.9	113.9	302.9	53.0	256.9
53	AS-SO-08-OI	Oxford Instrument Analytical ED 2000		426.5	116.2	301.4	39.4	290.7
54	LV-SO-03-XX	Reference Laboratory	48.0 J-	2000.0	120.0	210.0 J-	120.0	3700.0
54	LV-SO-40-XX	Reference Laboratory	46.0 J-	1900.0	120.0	210.0 J-	120.0	3700.0
54	LV-SO-49-XX	Reference Laboratory	52.0 J-	2000.0	120.0	220.0 J-	120.0	3800.0
54	LV-SO-03-OI	Oxford Instrument Analytical ED 2000	56.0	2199.3	90.9	177.7	171.0	4743.4
54	LV-SO-40-OI	Oxford Instrument Analytical ED 2000	40.9	2248.6	91.9	178.8	160.6	4833.5
54	LV-SO-49-OI	Oxford Instrument Analytical ED 2000	52.6	2235.0	88.1	172.0	131.3	4819.4
55	LV-SO-04-XX	Reference Laboratory	130.0 J-	2000.0	230.0	1.2 UJ	260.0	53.0
55	LV-SO-34-XX	Reference Laboratory	130.0 J-	1900.0 J-	220.0 J-	1.2 UJ	230.0 J-	48.0 J-
55	LV-SO-37-XX	Reference Laboratory	130.0 J-	1400.0	170.0	1.2 U	180.0	37.0
55	LV-SO-04-OI	Oxford Instrument Analytical ED 2000	120.2	2268.3	187.4	0.4	279.3	91.2
55	LV-SO-34-OI	Oxford Instrument Analytical ED 2000	136.5	2306.9	180.2		298.3	95.4
55	LV-SO-37-OI	Oxford Instrument Analytical ED 2000	129.7	2447.0	189.9		335.3	98.7

Appendix D. Analytical Data Summary, Oxford ED2000 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	22.0	87.0	63.0	17.0	72.0	15000.0	130.0
56	CN-SO-06-XX	Reference Laboratory	20.0	91.0	64.0	18.0	74.0	16000.0	130.0
56	CN-SO-07-XX	Reference Laboratory	20.0	90.0	63.0	19.0	72.0	17000.0	130.0
56	CN-SO-03-OI	Oxford Instrument Analytical ED 2000	64.7	152.0	57.1	46.0	105.1	40660.0	156.7
56	CN-SO-06-OI	Oxford Instrument Analytical ED 2000	66.1	146.4	51.4	42.4	103.9	40690.0	177.5
56	CN-SO-07-OI	Oxford Instrument Analytical ED 2000	64.9	146.7	52.0	48.0	106.7	41270.0	169.2
57	CN-SO-02-XX	Reference Laboratory	230.0	19.0	820.0	290.0	140.0	22000.0	490.0
57	CN-SO-05-XX	Reference Laboratory	130.0	6.2	630.0	26.0	160.0	23000.0	25.0
57	CN-SO-09-XX	Reference Laboratory	120.0	6.0	580.0	21.0	140.0	19000.0	23.0
57	CN-SO-02-OI	Oxford Instrument Analytical ED 2000	353.5	16.2	418.8	33.7	188.0	40540.0	57.4
57	CN-SO-05-OI	Oxford Instrument Analytical ED 2000	350.6	19.1	448.9	27.7	171.3	39970.0	37.7
57	CN-SO-09-OI	Oxford Instrument Analytical ED 2000	335.6	27.3	428.0	30.9	189.0	39340.0	34.0
58	LV-SE-06-XX	Reference Laboratory	30.0	23.0	160.0	540.0	30.0	18000.0	1600.0
58	LV-SE-13-XX	Reference Laboratory	31.0	24.0	160.0	540.0	30.0	18000.0	1600.0
58	LV-SE-41-XX	Reference Laboratory	30.0	21.0	150.0	480.0	26.0	16000.0	1500.0
58	LV-SE-06-OI	Oxford Instrument Analytical ED 2000	157.6	271.7	177.3	538.3	42.1	56000.0	1843.0
58	LV-SE-13-OI	Oxford Instrument Analytical ED 2000	141.8	255.6	165.5	522.8	49.5	56230.0	1830.6
58	LV-SE-41-OI	Oxford Instrument Analytical ED 2000	141.0	274.5	164.0	572.6	43.2	54990.0	1834.6
59	LV-SE-05-XX	Reference Laboratory	92.0	20.0	440.0	840.0	39.0	16000.0	14.0
59	LV-SE-20-XX	Reference Laboratory	140.0 J+	31.0	680.0	1400.0	60.0	22000.0	21.0
59	LV-SE-43-XX	Reference Laboratory	160.0 J+	24.0	550.0	1100.0	47.0	19000.0	17.0
59	LV-SE-05-OI	Oxford Instrument Analytical ED 2000	504.8	16.9	550.0	958.8	55.8	55690.0	37.7
59	LV-SE-20-OI	Oxford Instrument Analytical ED 2000	499.8	7.6	553.9	1040.9	69.7	55900.0	47.5
59	LV-SE-43-OI	Oxford Instrument Analytical ED 2000	487.9	34.8	543.1	1036.6	61.2	55540.0	32.3
60	LV-SE-15-XX	Reference Laboratory	290.0 J+	32.0	1300.0	83.0	2300.0	22000.0	18.0
60	LV-SE-17-XX	Reference Laboratory	280.0 J+	31.0	1300.0	79.0	2200.0	21000.0	17.0 J-
60	LV-SE-51-XX	Reference Laboratory	210.0 J+	26.0	1100.0	72.0	2000.0	19000.0	15.0
60	LV-SE-15-OI	Oxford Instrument Analytical ED 2000	776.5	45.0	1086.8	84.7	1980.8	54390.0	41.0
60	LV-SE-17-OI	Oxford Instrument Analytical ED 2000	788.3	39.5	1098.1	88.6	2006.2	55080.0	38.8
60	LV-SE-51-OI	Oxford Instrument Analytical ED 2000	735.2	47.2	1048.8	91.6	2069.5	53940.0	24.9
61	TL-SE-05-XX	Reference Laboratory	100.0 J+	34.0	0.3 J	40.0	4900.0	24000.0	1200.0
61	TL-SE-09-XX	Reference Laboratory	100.0 J+	33.0	0.2 J	39.0	4800.0	23000.0	1200.0
61	TL-SE-13-XX	Reference Laboratory	95.0 J+	31.0	0.5 J	36.0 J+	4400.0 J+	22000.0 J+	1100.0 J+
61	TL-SE-05-OI	Oxford Instrument Analytical ED 2000	662.3	157.8		62.7	3874.4	60550.0	1116.0
61	TL-SE-09-OI	Oxford Instrument Analytical ED 2000	648.5	165.3		64.3	3894.7	61530.0	1142.5
61	TL-SE-13-OI	Oxford Instrument Analytical ED 2000	646.3	159.9		56.8	3879.4	61090.0	1099.4

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
56	CN-SO-03-XX	Reference Laboratory	34.0 J-	74.0	36.0	90.0	30.0	58.0
56	CN-SO-06-XX	Reference Laboratory	40.0 J-	76.0	38.0	94.0	32.0	59.0
56	CN-SO-07-XX	Reference Laboratory	36.0 J-	75.0	37.0	91.0	33.0	58.0
56	CN-SO-03-OI	Oxford Instrument Analytical ED 2000	45.9	113.6	30.8	68.7	51.8	100.1
56	CN-SO-06-OI	Oxford Instrument Analytical ED 2000	51.6	107.3	32.0	68.0	49.6	94.3
56	CN-SO-07-OI	Oxford Instrument Analytical ED 2000	32.5	108.8	31.1	68.8	58.1	88.9
57	CN-SO-02-XX	Reference Laboratory	270.0 J-	530.0	190.0	68.0	160.0	1900.0
57	CN-SO-05-XX	Reference Laboratory	280.0 J-	360.0	190.0	78.0	160.0	2200.0
57	CN-SO-09-XX	Reference Laboratory	260.0 J-	330.0	170.0	74.0	140.0	2100.0
57	CN-SO-02-OI	Oxford Instrument Analytical ED 2000	293.9	369.4	123.2	45.9	144.3	2652.8
57	CN-SO-05-OI	Oxford Instrument Analytical ED 2000	262.2	365.2	120.3	49.2	145.3	2610.1
57	CN-SO-09-OI	Oxford Instrument Analytical ED 2000	276.3	363.5	115.5	46.4	153.7	2518.4
58	LV-SE-06-XX	Reference Laboratory	610.0 J-	360.0	160.0	110.0	480.0	52.0
58	LV-SE-13-XX	Reference Laboratory	640.0 J-	360.0	160.0	110.0	470.0	51.0
58	LV-SE-41-XX	Reference Laboratory	610.0 J-	320.0	150.0	99.0	420.0	46.0
58	LV-SE-06-OI	Oxford Instrument Analytical ED 2000	617.5	432.1	130.9	104.1	601.7	89.0
58	LV-SE-13-OI	Oxford Instrument Analytical ED 2000	604.8	411.6	125.0	100.4	569.9	88.5
58	LV-SE-41-OI	Oxford Instrument Analytical ED 2000	621.8	420.5	122.0	97.9	655.9	71.6
59	LV-SE-05-XX	Reference Laboratory	2.6 J-	400.0	340.0	49.0	340.0	1800.0
59	LV-SE-20-XX	Reference Laboratory	2.8	660.0	500.0	75.0 J-	530.0	2800.0
59	LV-SE-43-XX	Reference Laboratory	2.8	530.0	420.0	60.0 J-	430.0	2300.0
59	LV-SE-05-OI	Oxford Instrument Analytical ED 2000	2.0	511.6	293.6	57.3	489.3	2531.5
59	LV-SE-20-OI	Oxford Instrument Analytical ED 2000		511.7	283.2	55.9	488.6	2580.9
59	LV-SE-43-OI	Oxford Instrument Analytical ED 2000	6.5	535.2	286.8	54.3	543.2	2619.8
60	LV-SE-15-XX	Reference Laboratory	500.0	230.0	92.0	300.0 J-	180.0	62.0
60	LV-SE-17-XX	Reference Laboratory	490.0	220.0	89.0	200.0 J-	170.0	58.0
60	LV-SE-51-XX	Reference Laboratory	470.0	200.0	76.0	250.0 J-	160.0	54.0
60	LV-SE-15-OI	Oxford Instrument Analytical ED 2000	479.6	210.4	54.5	353.3	177.9	65.3
60	LV-SE-17-OI	Oxford Instrument Analytical ED 2000	483.0	204.7	57.6	364.3	180.9	83.0
60	LV-SE-51-OI	Oxford Instrument Analytical ED 2000	472.5	213.6	52.0	345.2	195.6	76.5
61	TL-SE-05-XX	Reference Laboratory	980.0	54.0	130.0	180.0 J-	66.0	100.0
61	TL-SE-09-XX	Reference Laboratory	820.0	53.0	130.0	170.0 J-	63.0	100.0
61	TL-SE-13-XX	Reference Laboratory	990.0	49.0	120.0	160.0 J	59.0 J+	96.0
61	TL-SE-05-OI	Oxford Instrument Analytical ED 2000	726.2	79.5	82.3	126.4	59.4	126.5
61	TL-SE-09-OI	Oxford Instrument Analytical ED 2000	750.1	61.9	84.1	125.5	68.2	99.1
61	TL-SE-13-OI	Oxford Instrument Analytical ED 2000	736.2	71.7	84.0	122.3	39.1	104.4

Appendix D. Analytical Data Summary, Oxford ED2000 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	86.0	350.0	34.0	2000.0	22000.0	1700.0
62	TL-SE-17-XX	Reference Laboratory	1.2 U	85.0	340.0	33.0	2100.0	21000.0	1700.0
62	TL-SE-28-XX	Reference Laboratory	1.2 U	89.0	360.0	34.0	2100.0	22000.0	1700.0
62	TL-SE-06-OI	Oxford Instrument Analytical ED 2000		318.7	332.3	62.0	2192.7	59820.0	1748.3
62	TL-SE-17-OI	Oxford Instrument Analytical ED 2000	1.0	289.1	341.1	65.2	2221.5	59180.0	1747.3
62	TL-SE-28-OI	Oxford Instrument Analytical ED 2000		338.6	323.9	69.4	2330.4	77260.0	1735.1
63	TL-SE-07-XX	Reference Laboratory	30.0	11.0	48.0	66.0	2200.0	37000.0	13.0
63	TL-SE-21-XX	Reference Laboratory	33.0	13.0	51.0	73.0	2300.0	44000.0	15.0
63	TL-SE-30-XX	Reference Laboratory	31.0	11.0	47.0	64.0	2200.0	36000.0	14.0
63	TL-SE-07-OI	Oxford Instrument Analytical ED 2000	219.1	14.5	40.9	104.2	2415.1	113930.0	14.3
63	TL-SE-21-OI	Oxford Instrument Analytical ED 2000	232.8	25.5	42.3	87.9	2421.4	118970.0	19.2
63	TL-SE-30-OI	Oxford Instrument Analytical ED 2000	219.6	48.2	39.8	82.1	2391.5	110890.0	17.6
64	TL-SE-02-XX	Reference Laboratory	77.0	15.0	160.0	64.0	3100.0	32000.0	12.0
64	TL-SE-08-XX	Reference Laboratory	66.0	10.0	180.0	74.0	3200.0	45000.0	11.0
64	TL-SE-16-XX	Reference Laboratory	73.0	15.0	170.0	69.0	3100.0	38000.0	13.0
64	TL-SE-02-OI	Oxford Instrument Analytical ED 2000	603.1	38.8	139.3	108.0	3238.1	120700.0	15.7
64	TL-SE-08-OI	Oxford Instrument Analytical ED 2000	605.0	14.9	137.8	101.4	3247.2	118120.0	8.9
64	TL-SE-16-OI	Oxford Instrument Analytical ED 2000	589.5	29.1	141.5	77.8	3260.3	123390.0	14.8
65	RF-SE-01-XX	Reference Laboratory	12.0	230.0	40.0	280.0	63.0	14000.0	22.0
65	RF-SE-09-XX	Reference Laboratory	10.0	260.0	45.0	310.0	71.0	16000.0	26.0
65	RF-SE-11-XX	Reference Laboratory	11.0	240.0	43.0	300.0	72.0	15000.0	25.0
65	RF-SE-17-XX	Reference Laboratory	11.0	250.0	43.0	300.0	67.0	15000.0	26.0
65	RF-SE-29-XX	Reference Laboratory	13.0	280.0	49.0	330.0	75.0	17000.0	26.0
65	RF-SE-37-XX	Reference Laboratory	11.0	260.0	45.0	320.0	72.0	16000.0	27.0
65	RF-SE-50-XX	Reference Laboratory	8.9	230.0	40.0	280.0	65.0	14000.0	23.0
65	RF-SE-01-OI	Oxford Instrument Analytical ED 2000	41.6	308.9	45.0	327.7	80.0	41740.0	45.3
65	RF-SE-09-OI	Oxford Instrument Analytical ED 2000	43.7	316.6	47.2	318.3	76.6	42800.0	40.9
65	RF-SE-11-OI	Oxford Instrument Analytical ED 2000	95.4	331.9	46.6	342.3	98.3	73730.0	50.6
65	RF-SE-17-OI	Oxford Instrument Analytical ED 2000	74.1	290.1	45.8	343.5	84.9	59910.0	41.8
65	RF-SE-29-OI	Oxford Instrument Analytical ED 2000	38.7	308.2	47.0	325.4	99.1	42030.0	43.4
65	RF-SE-37-OI	Oxford Instrument Analytical ED 2000	39.7	300.7	47.4	309.6	87.8	41500.0	46.5
65	RF-SE-50-OI	Oxford Instrument Analytical ED 2000	43.7	306.7	48.2	336.2	85.0	41950.0	29.9

Appendix D. Analytical Data Summary, Oxford ED2000 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	44.0	45.0	56.0	78.0	83.0
62	TL-SE-17-XX	Reference Laboratory	2.6	43.0	44.0	56.0	78.0	81.0
62	TL-SE-28-XX	Reference Laboratory	2.8	44.0	45.0	57.0	81.0	83.0
62	TL-SE-06-OI	Oxford Instrument Analytical ED 2000	10.9	74.3	33.6	48.3	73.1	120.9
62	TL-SE-17-OI	Oxford Instrument Analytical ED 2000	4.2	69.5	31.8	47.4	84.0	118.3
62	TL-SE-28-OI	Oxford Instrument Analytical ED 2000	2.4	57.4	33.1	50.9	93.3	108.3
63	TL-SE-07-XX	Reference Laboratory	40.0	94.0	120.0	63.0	110.0	160.0
63	TL-SE-21-XX	Reference Laboratory	120.0	100.0	140.0	67.0	120.0	170.0
63	TL-SE-30-XX	Reference Laboratory	100.0	93.0	120.0	62.0	100.0	160.0
63	TL-SE-07-OI	Oxford Instrument Analytical ED 2000	39.2	137.5	106.3	51.0	48.5	184.2
63	TL-SE-21-OI	Oxford Instrument Analytical ED 2000	28.1	136.8	102.4	55.2	108.6	228.7
63	TL-SE-30-OI	Oxford Instrument Analytical ED 2000	34.2	132.2	99.1	52.0	117.2	196.6
64	TL-SE-02-XX	Reference Laboratory	400.0	99.0	44.0	120.0	110.0	160.0
64	TL-SE-08-XX	Reference Laboratory	350.0	100.0	39.0	130.0	120.0	170.0
64	TL-SE-16-XX	Reference Laboratory	420.0	100.0	44.0	120.0	110.0	160.0
64	TL-SE-02-OI	Oxford Instrument Analytical ED 2000	122.2	149.0	38.0	93.4	135.7	226.7
64	TL-SE-08-OI	Oxford Instrument Analytical ED 2000	132.1	143.2	37.4	94.2	87.6	185.6
64	TL-SE-16-OI	Oxford Instrument Analytical ED 2000	127.3	127.0	41.3	95.1	141.8	219.9
65	RF-SE-01-XX	Reference Laboratory	47.0	200.0	21.0	37.0	29.0	1700.0
65	RF-SE-09-XX	Reference Laboratory	45.0	220.0	23.0	42.0	32.0	1900.0
65	RF-SE-11-XX	Reference Laboratory	52.0	210.0	20.0	40.0	29.0	1800.0
65	RF-SE-17-XX	Reference Laboratory	20.0	210.0	22.0	40.0	30.0	1800.0
65	RF-SE-29-XX	Reference Laboratory	20.0	240.0	26.0	44.0	35.0	2100.0
65	RF-SE-37-XX	Reference Laboratory	22.0	220.0	23.0	44.0	32.0	1900.0
65	RF-SE-50-XX	Reference Laboratory	19.0	200.0	20.0	38.0	29.0	1700.0
65	RF-SE-01-OI	Oxford Instrument Analytical ED 2000	31.2	232.9	15.0	37.7	55.2	2198.0
65	RF-SE-09-OI	Oxford Instrument Analytical ED 2000	23.1	228.1	15.7	43.0	68.4	2216.3
65	RF-SE-11-OI	Oxford Instrument Analytical ED 2000	16.4	237.8	19.9	35.9	60.7	2209.0
65	RF-SE-17-OI	Oxford Instrument Analytical ED 2000	4.8	224.2	15.6	40.3	61.9	2148.0
65	RF-SE-29-OI	Oxford Instrument Analytical ED 2000	24.5	223.6	17.1	38.9	66.2	2138.2
65	RF-SE-37-OI	Oxford Instrument Analytical ED 2000	34.5	208.9	12.5	40.2	67.8	2154.9
65	RF-SE-50-OI	Oxford Instrument Analytical ED 2000	33.9	219.8	15.1	39.9	66.3	2162.9

Appendix D. Analytical Data Summary, Oxford ED2000 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	14.0	460.0	67.0	510.0	1800.0	18000.0	580.0
66	RF-SE-10-XX	Reference Laboratory	12.0	400.0	58.0	440.0	1500.0	16000.0	510.0
66	RF-SE-33-XX	Reference Laboratory	13.0	440.0	64.0	490.0	1700.0	18000.0	570.0
66	RF-SE-08-OI	Oxford Instrument Analytical ED 2000	60.4	613.8	69.3	497.8	1977.6	45260.0	574.6
66	RF-SE-10-OI	Oxford Instrument Analytical ED 2000	62.9	621.5	68.9	513.7	2022.9	44750.0	594.1
66	RF-SE-33-OI	Oxford Instrument Analytical ED 2000	63.3	610.1	72.3	520.6	1939.8	44880.0	566.4
67	RF-SE-16-XX	Reference Laboratory	85.0 J-	72.0 J-	310.0 J-	820.0 J-	73.0 J-	16000.0 J-	24.0 J-
67	RF-SE-41-XX	Reference Laboratory	100.0	82.0	360.0	950.0	85.0	18000.0	25.0
67	RF-SE-48-XX	Reference Laboratory	100.0	87.0	380.0	1000.0	90.0	19000.0	27.0
67	RF-SE-16-OI	Oxford Instrument Analytical ED 2000	293.6	103.6	343.8	891.7	88.6	43130.0	38.6
67	RF-SE-41-OI	Oxford Instrument Analytical ED 2000	299.8	101.4	342.5	881.3	85.9	43810.0	36.6
67	RF-SE-48-OI	Oxford Instrument Analytical ED 2000	310.4	99.6	354.5	851.8	82.1	42480.0	50.4
68	RF-SE-18-XX	Reference Laboratory	320.0	810.0	770.0	950.0	78.0	16000.0	860.0
68	RF-SE-35-XX	Reference Laboratory	300.0	740.0	700.0	860.0	70.0	15000.0	780.0
68	RF-SE-54-XX	Reference Laboratory	320.0	880.0	840.0	1000.0	86.0	18000.0	920.0
68	RF-SE-18-OI	Oxford Instrument Analytical ED 2000	835.4	1000.1	723.5	873.6	90.0	41660.0	783.4
68	RF-SE-35-OI	Oxford Instrument Analytical ED 2000	876.3	1057.4	761.2	912.1	100.8	42010.0	781.3
68	RF-SE-54-OI	Oxford Instrument Analytical ED 2000	852.6	970.8	721.2	895.9	84.2	41150.0	784.8
69	RF-SE-20-XX	Reference Laboratory	550.0	1300.0	540.0	94.0	93.0	20000.0	28.0
69	RF-SE-46-XX	Reference Laboratory	270.0	590.0	240.0	44.0	40.0	8900.0	13.0
69	RF-SE-51-XX	Reference Laboratory	480.0	1100.0	450.0	77.0	77.0	17000.0	23.0
69	RF-SE-20-OI	Oxford Instrument Analytical ED 2000	5606.5	996.5	287.3	96.0	59.3	237540.0	58.5
69	RF-SE-46-OI	Oxford Instrument Analytical ED 2000	1165.0	1259.3	476.1	90.2	86.9	43920.0	44.8
69	RF-SE-51-OI	Oxford Instrument Analytical ED 2000	1132.4	1322.6	467.9	106.0	89.6	46080.0	33.0
70	RF-SE-21-XX	Reference Laboratory	1.3 U	62.0	1700.0	76.0	1000.0	16000.0	2100.0
70	RF-SE-40-XX	Reference Laboratory	1.3 U	70.0	1900.0	85.0	1100.0	18000.0	2400.0
70	RF-SE-47-XX	Reference Laboratory	1.3 U	72.0	1900.0	90.0	1200.0	19000.0	2400.0
70	RF-SE-21-OI	Oxford Instrument Analytical ED 2000	0.2	378.5	758.8	138.0	1312.8	59140.0	2273.3
70	RF-SE-40-OI	Oxford Instrument Analytical ED 2000	7.8	359.3	1781.6	96.1	1244.5	45590.0	2258.6
70	RF-SE-47-OI	Oxford Instrument Analytical ED 2000	8.3	380.9	1790.1	93.3	1218.7	45670.0	2186.2

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
66	RF-SE-08-XX	Reference Laboratory	29.0	250.0	42.0	0.4 U	120.0	120.0
66	RF-SE-10-XX	Reference Laboratory	27.0	220.0	39.0	0.3 U	100.0	110.0
66	RF-SE-33-XX	Reference Laboratory	28.0	240.0	41.0	0.3 U	120.0	130.0
66	RF-SE-08-OI	Oxford Instrument Analytical ED 2000	30.9	252.2	29.5		177.0	140.5
66	RF-SE-10-OI	Oxford Instrument Analytical ED 2000	35.9	252.9	31.4	0.1	166.7	151.2
66	RF-SE-33-OI	Oxford Instrument Analytical ED 2000	31.9	268.1	28.6	3.8	169.8	121.4
67	RF-SE-16-XX	Reference Laboratory	260.0	1700.0 J-	1.2 U	130.0 J-	32.0 J-	760.0 J-
67	RF-SE-41-XX	Reference Laboratory	230.0	1900.0	1.2 U	140.0	39.0	830.0
67	RF-SE-48-XX	Reference Laboratory	250.0	2000.0	2.2	150.0	40.0	880.0
67	RF-SE-16-OI	Oxford Instrument Analytical ED 2000	299.8	1800.8	2.5	130.5	67.8	910.7
67	RF-SE-41-OI	Oxford Instrument Analytical ED 2000	295.6	1804.6	3.6	130.5	56.3	889.6
67	RF-SE-48-OI	Oxford Instrument Analytical ED 2000	310.4	1739.7	4.4	134.5	65.5	896.1
68	RF-SE-18-XX	Reference Laboratory	600.0	390.0	140.0	140.0	390.0	120.0
68	RF-SE-35-XX	Reference Laboratory	650.0	350.0	140.0	150.0	340.0	110.0
68	RF-SE-54-XX	Reference Laboratory	670.0	420.0	160.0	180.0	410.0	120.0
68	RF-SE-18-OI	Oxford Instrument Analytical ED 2000	795.7	386.0	104.2	176.2	459.1	132.9
68	RF-SE-35-OI	Oxford Instrument Analytical ED 2000	838.8	368.5	106.8	184.0	485.1	167.2
68	RF-SE-54-OI	Oxford Instrument Analytical ED 2000	797.1	379.3	104.8	174.2	420.9	139.4
69	RF-SE-20-XX	Reference Laboratory	0.5	1400.0	380.0	59.0	36.0	1400.0
69	RF-SE-46-XX	Reference Laboratory	0.5	650.0	170.0	26.0	16.0	650.0
69	RF-SE-51-XX	Reference Laboratory	0.5	1200.0	320.0	48.0	30.0	1200.0
69	RF-SE-20-OI	Oxford Instrument Analytical ED 2000	2.2	1191.9	249.3	28.6	60.1	1407.3
69	RF-SE-46-OI	Oxford Instrument Analytical ED 2000		1245.9	239.6	45.1	64.4	1377.9
69	RF-SE-51-OI	Oxford Instrument Analytical ED 2000		1281.9	241.3	44.6	57.1	1407.1
70	RF-SE-21-XX	Reference Laboratory	320.0	220.0	440.0	120.0	130.0	100.0
70	RF-SE-40-XX	Reference Laboratory	280.0	250.0	480.0	100.0	150.0	120.0
70	RF-SE-47-XX	Reference Laboratory	320.0	250.0	510.0	120.0	150.0	120.0
70	RF-SE-21-OI	Oxford Instrument Analytical ED 2000	395.1	241.7	351.8	114.4	244.6	157.8
70	RF-SE-40-OI	Oxford Instrument Analytical ED 2000	397.8	221.4	349.9	270.5	189.9	141.3
70	RF-SE-47-OI	Oxford Instrument Analytical ED 2000	378.3	223.2	340.6	271.9	182.4	120.4

Appendix D. Analytical Data Summary, Oxford ED2000 and Reference Laboratory (Continued)

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

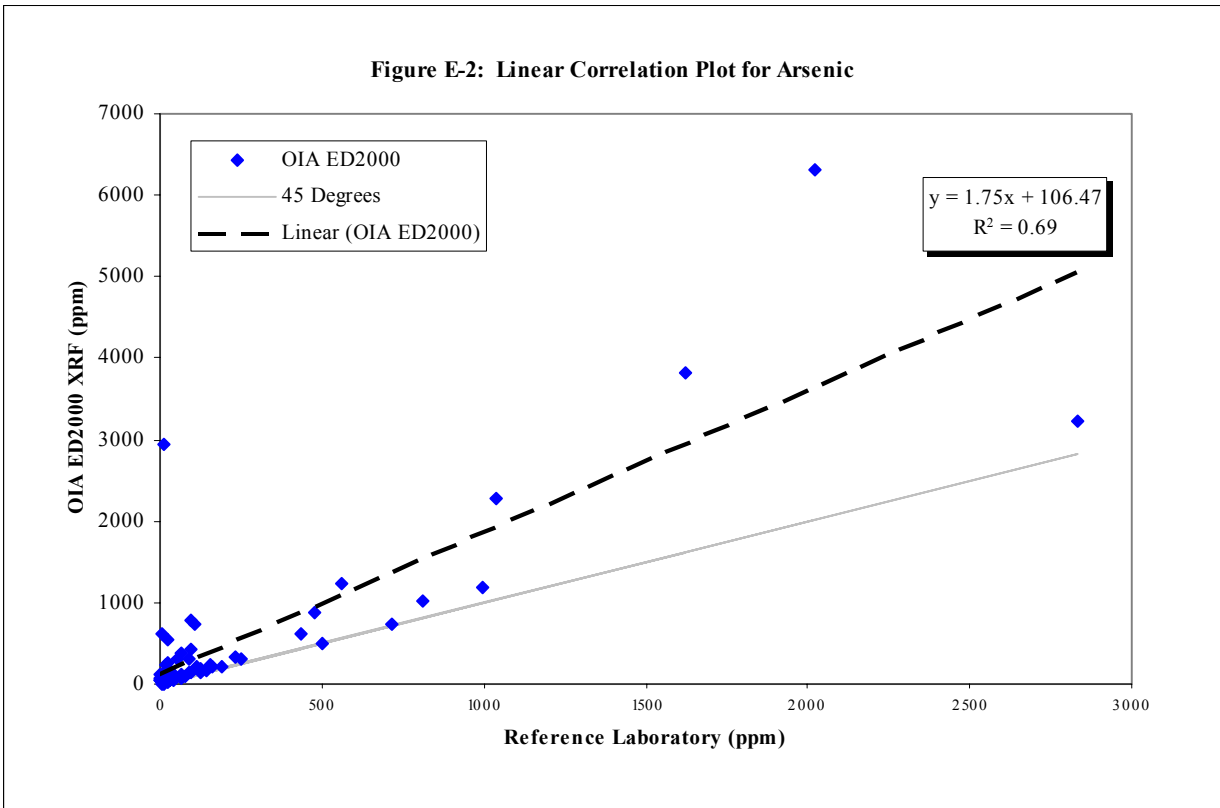
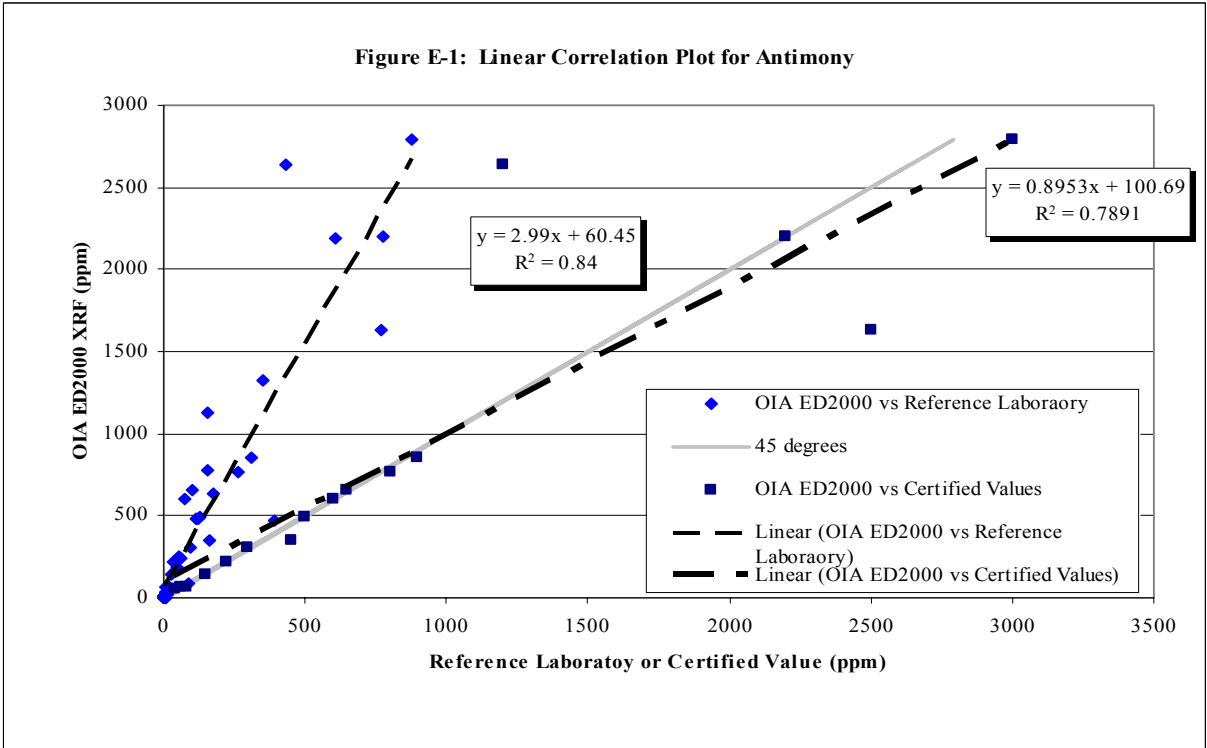


Figure E-3: Linear Correlation Plot for Cadmium

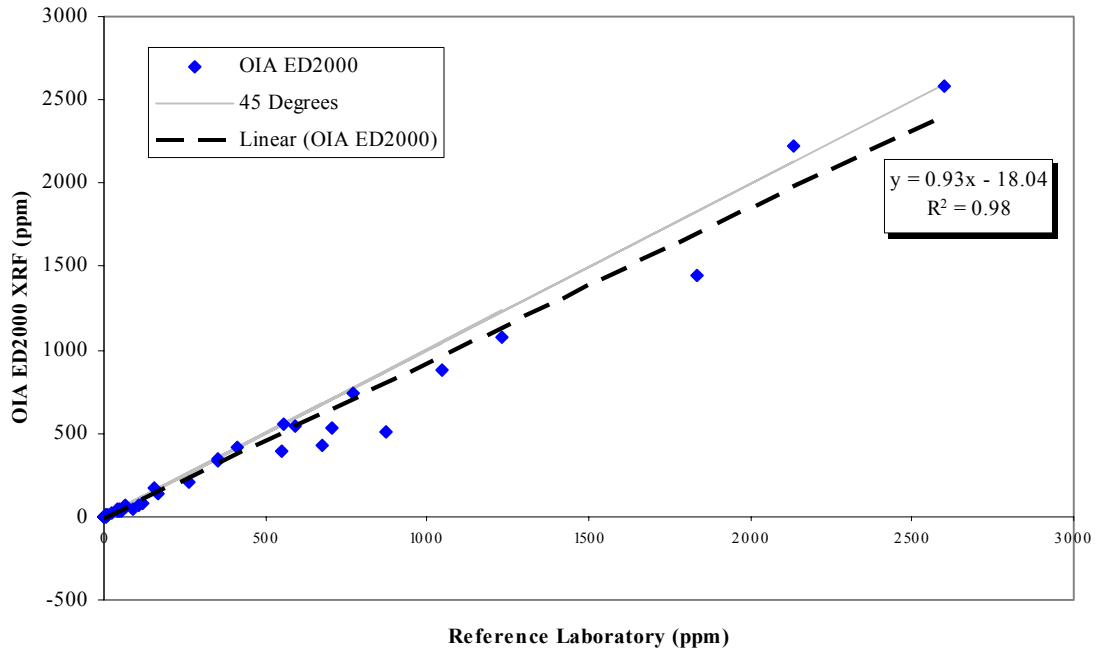


Figure E-4: Linear Correlation Plot for Chromium

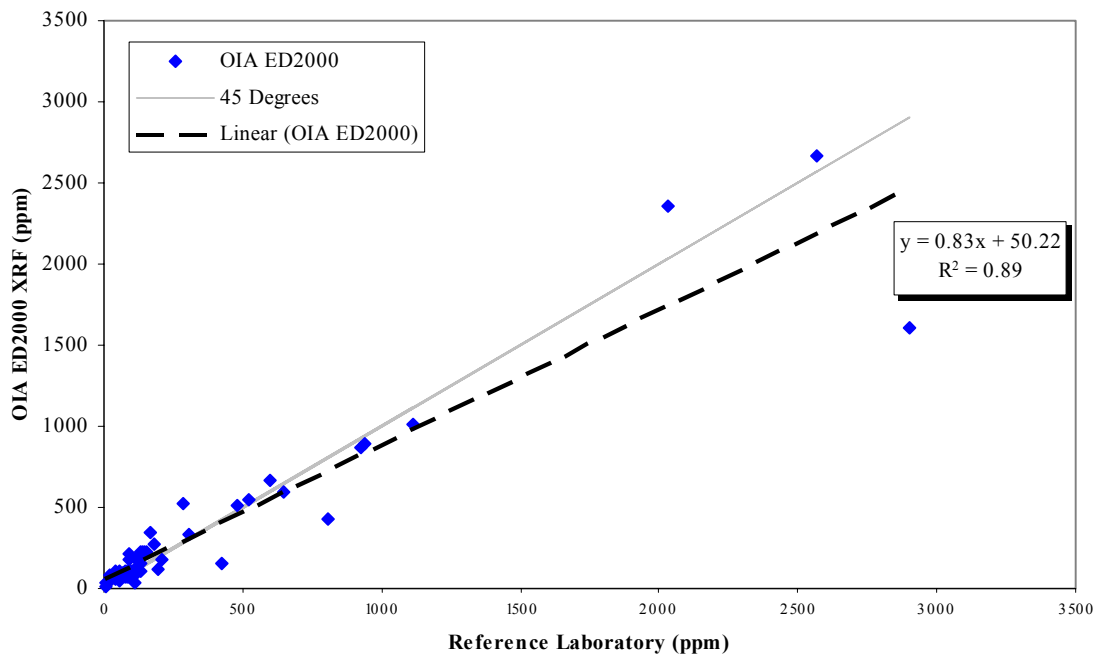


Figure E-5: Linear Correlation Plot for Copper

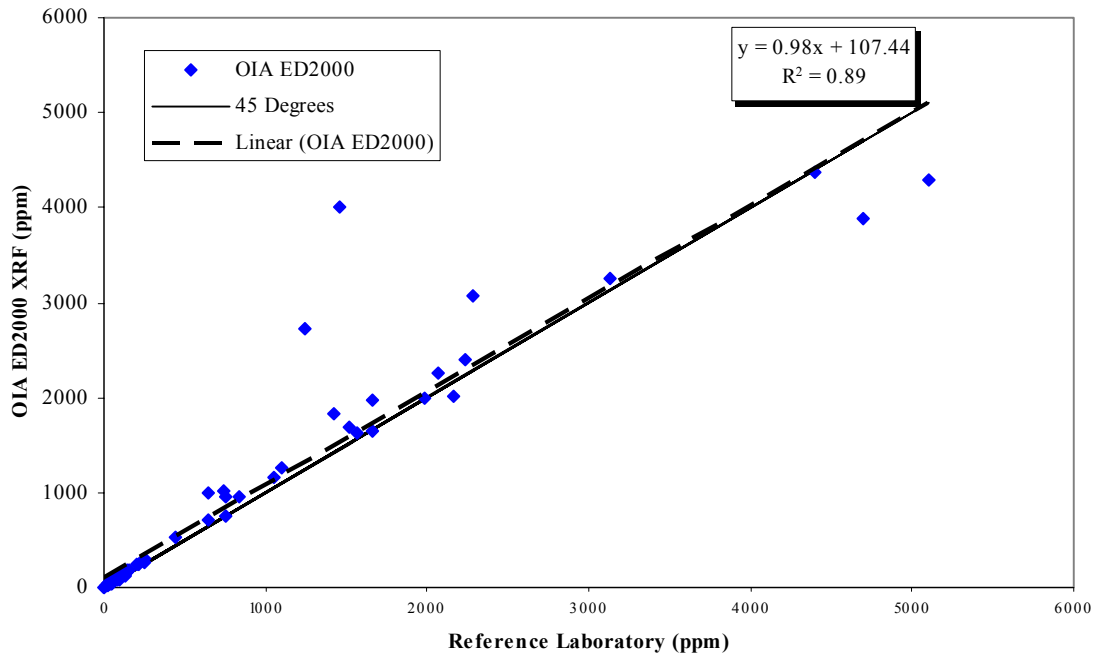


Figure E-6: Linear Correlation Plot for Iron

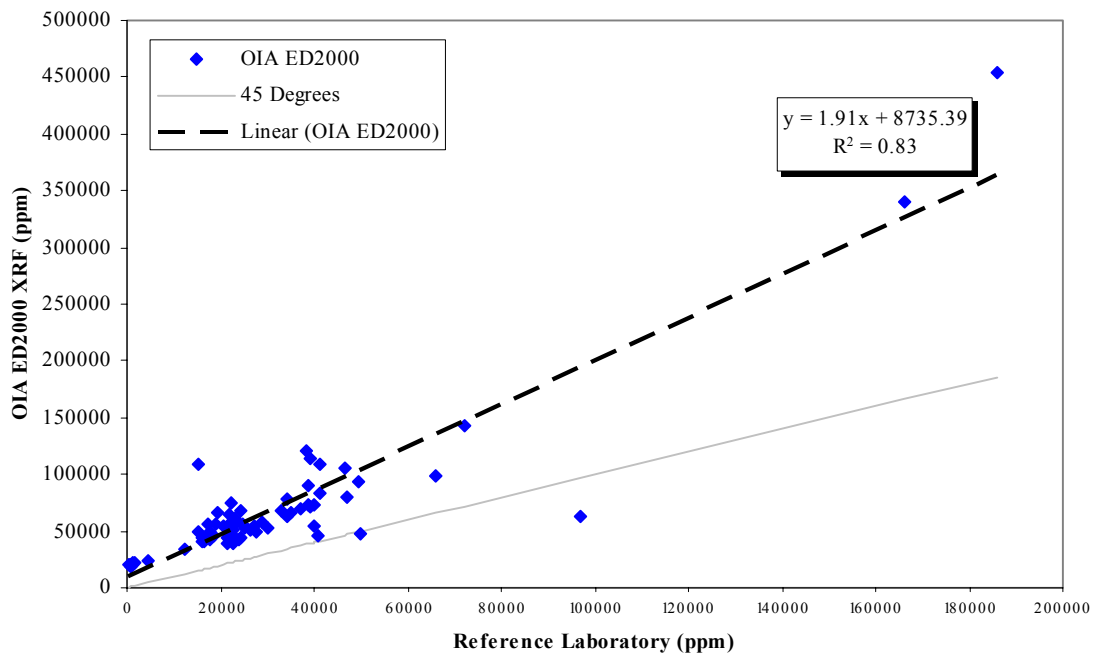


Figure E-7: Linear Correlation Plot for Lead

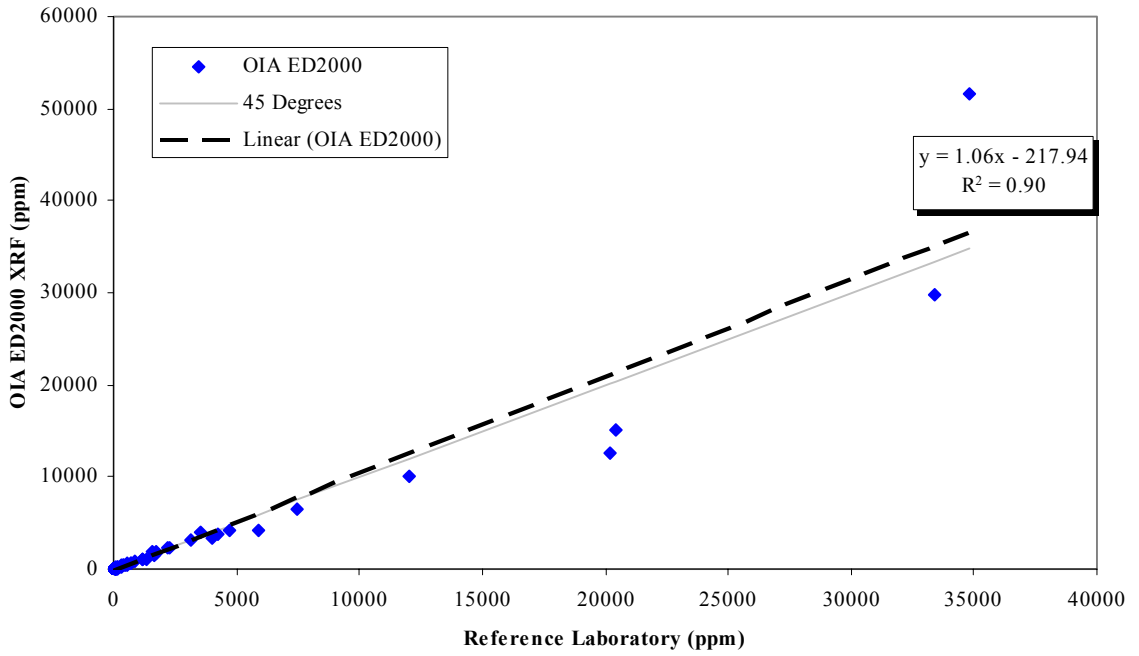


Figure E-8: Linear Correlation Plot for Mercury

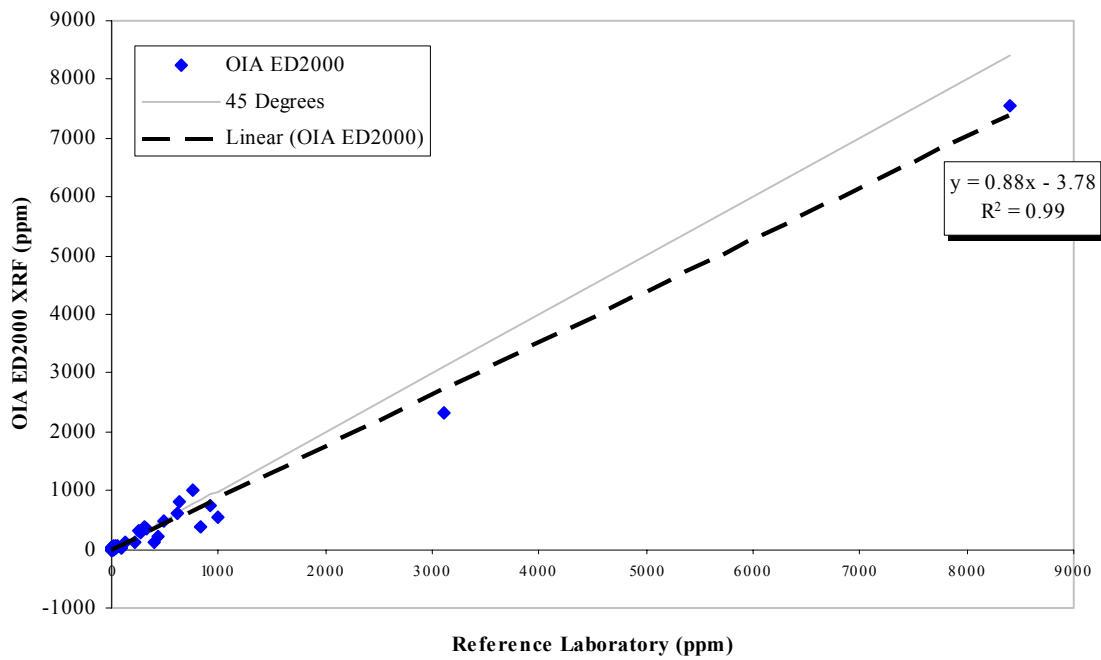


Figure E-9: Linear Correlation Plot for Nickel

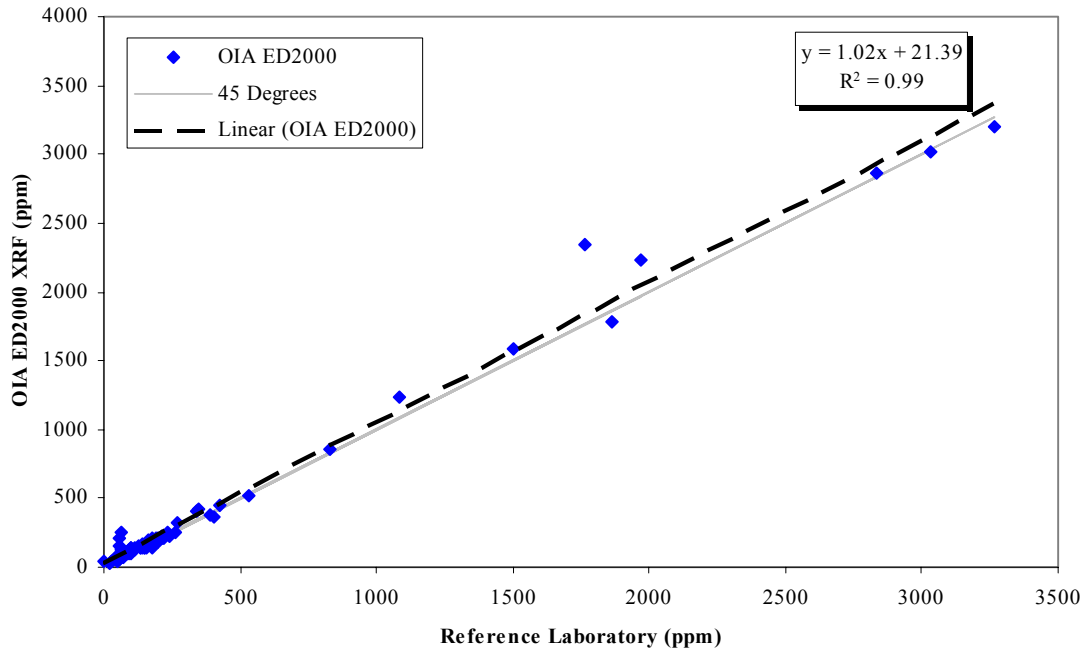


Figure E-10: Linear Correlation Plot for Selenium

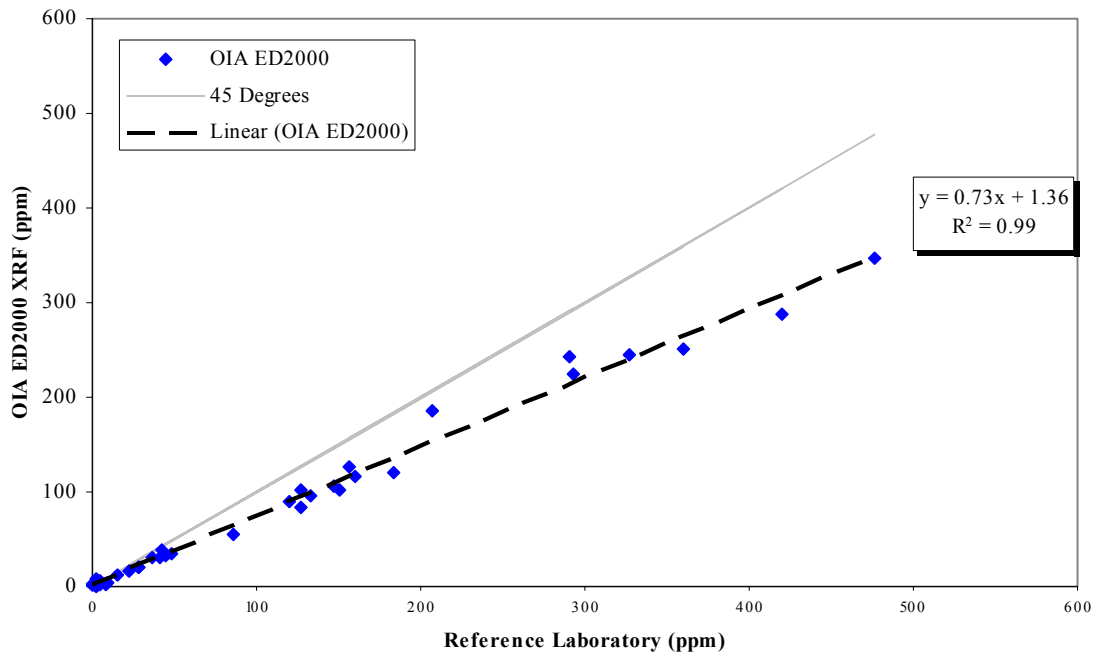


Figure E-11: Linear Correlation Plot for Silver

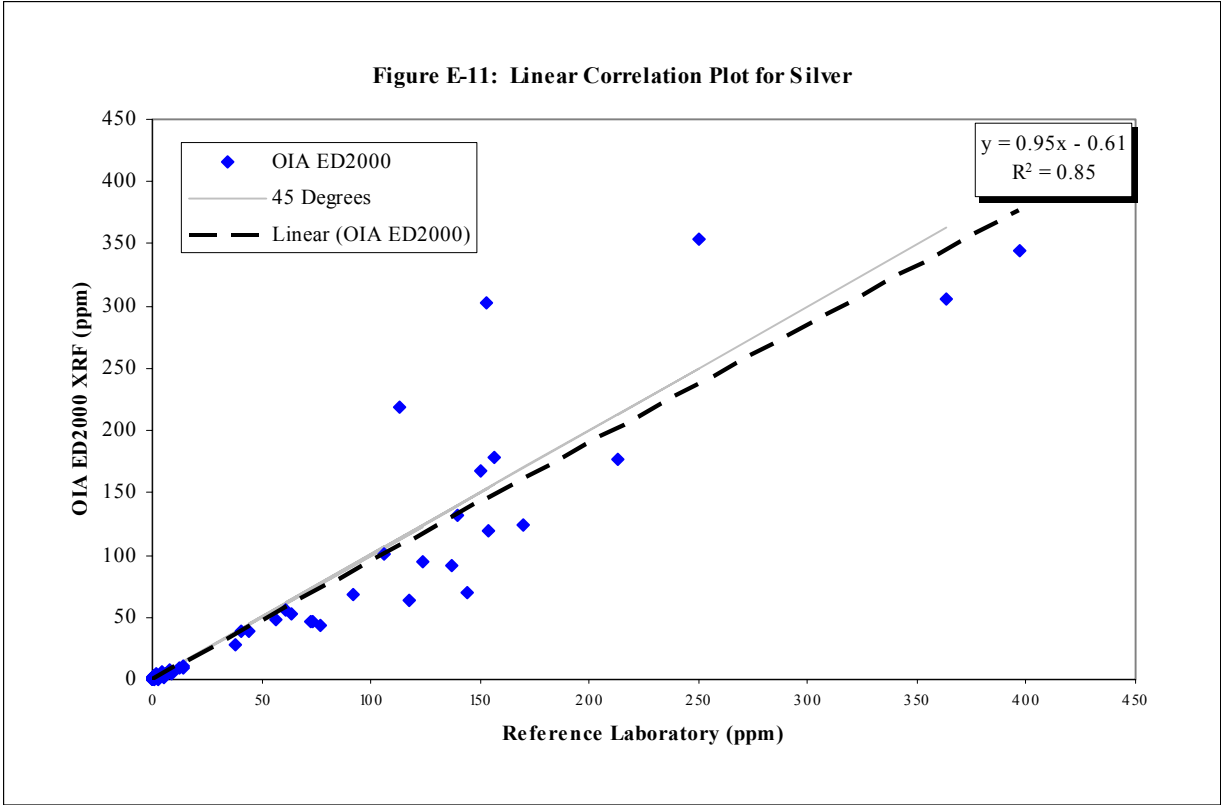


Figure E-12: Linear Correlation Plot for Vanadium

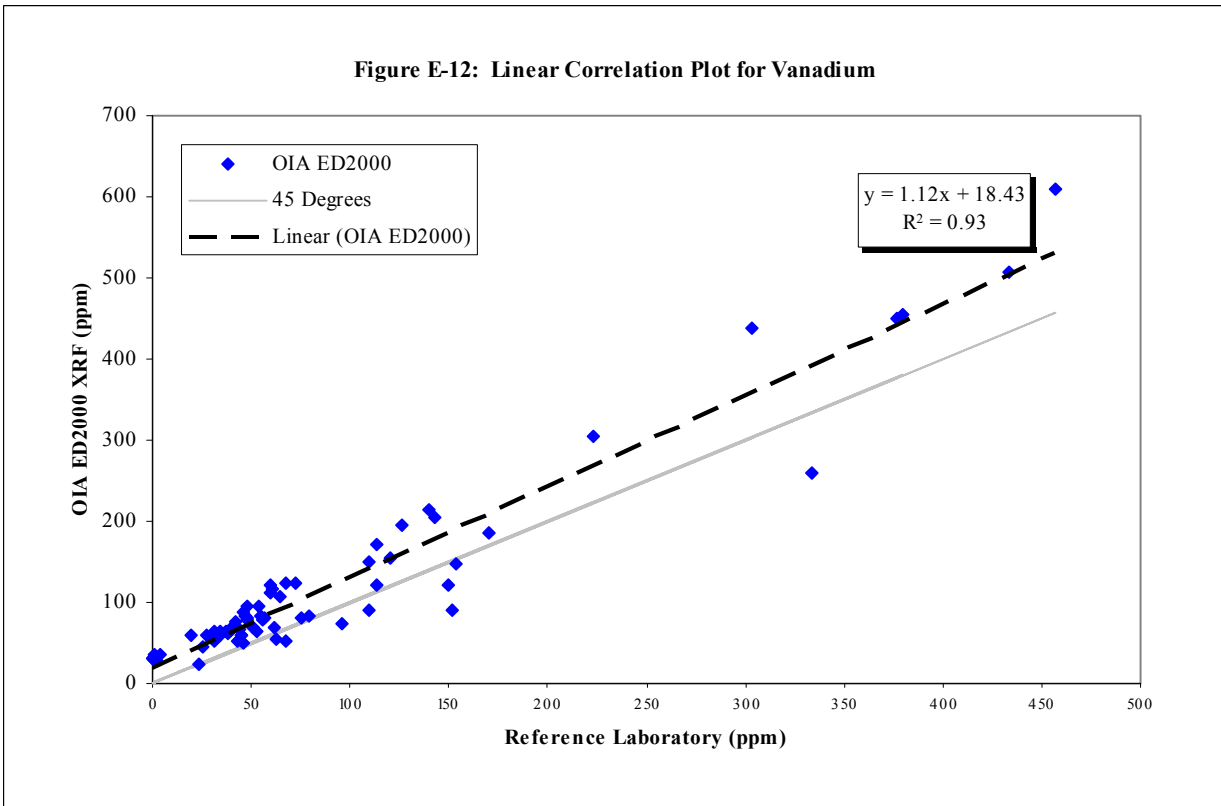
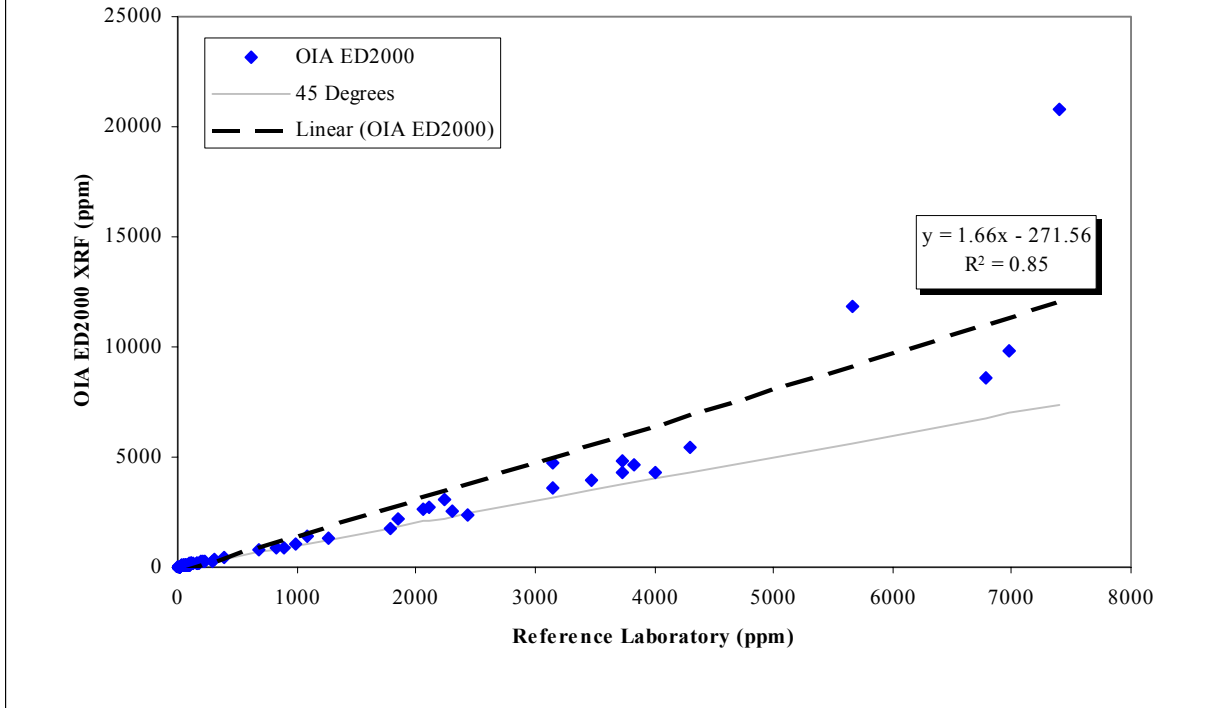
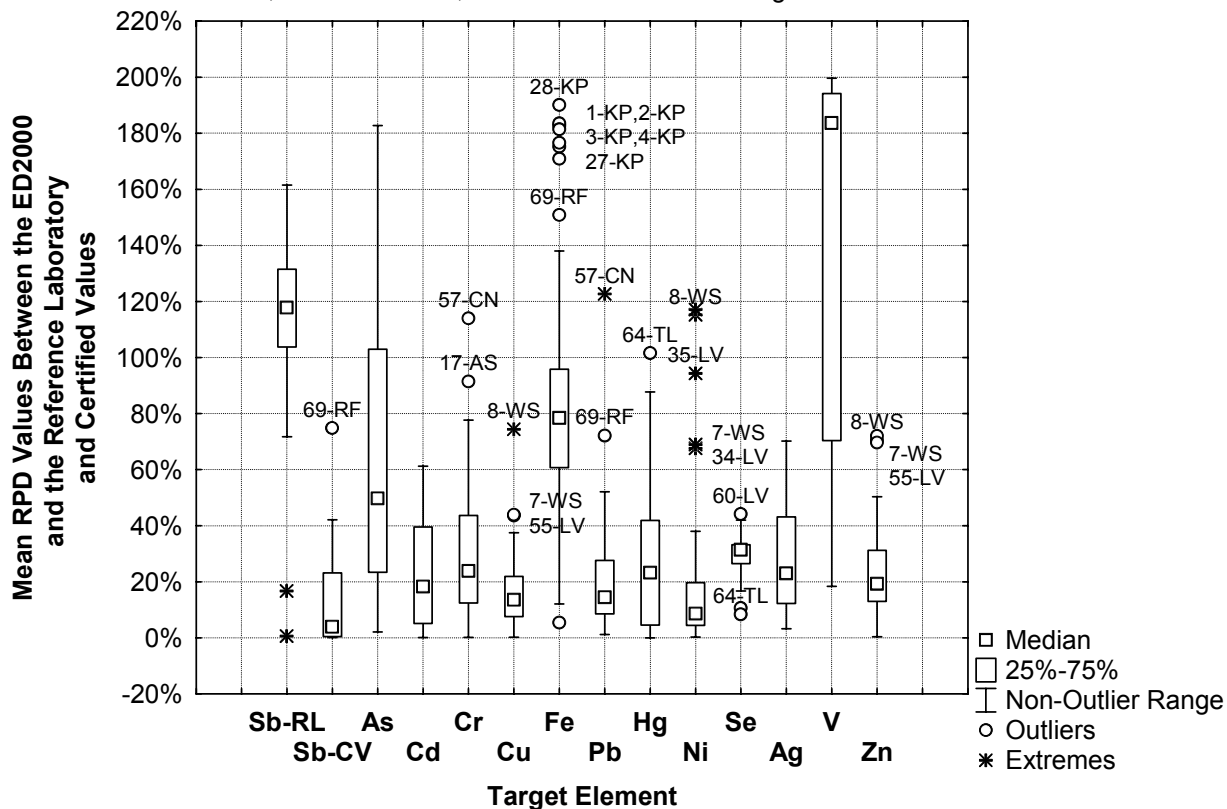


Figure E-13: Linear Correlation Plot for Zinc



**Box Plot for Relative Percent Difference (RPD)
Oxford ED2000**

Median; Box: 25%-75%; Whisker: Non-Outlier Range



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.

Figure E-14. Box and Whiskers Plot for Mean RPD Values Showing Outliers and Extremes for Target Elements, Oxford ED2000 Data Set.

Table E-1. Evaluation of Accuracy - Relative Percent Differences versus Reference Laboratory Data Calculated for the Oxford ED2000

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
			Ref Lab	ERA Spike							
Soil	Level 1	Number	9	1	15	7	28	16	5	16	7
		Minimum	0.6%	20.3%	16.9%	16.8%	8.3%	1.0%	138.0%	1.2%	0.0%
		Maximum	161.6%	20.3%	182.8%	61.3%	114.0%	43.9%	182.7%	122.6%	41.9%
		Mean	99.3%	20.3%	82.3%	37.8%	39.6%	17.7%	168.7%	23.0%	13.1%
		Median	118.3%	20.3%	55.2%	39.5%	40.5%	14.7%	175.1%	17.1%	4.6%
	Level 2	Number	5	1	4	7	4	7	13	4	7
		Minimum	73.7%	26.0%	60.0%	4.3%	7.7%	0.2%	53.1%	8.6%	2.7%
		Maximum	133.9%	26.0%	80.7%	53.1%	61.2%	74.4%	110.1%	19.4%	70.7%
		Mean	112.2%	26.0%	72.6%	27.1%	23.4%	32.8%	73.9%	13.6%	40.0%
		Median	115.5%	26.0%	74.8%	28.8%	12.4%	29.6%	62.5%	13.1%	49.4%
	Level 3	Number	4	3	3	2	2	2	13	8	2
		Minimum	71.7%	0.1%	13.1%	0.7%	3.8%	0.8%	54.9%	1.6%	10.5%
		Maximum	113.1%	42.1%	125.1%	54.2%	57.7%	17.1%	80.4%	32.1%	28.9%
		Mean	96.4%	16.4%	80.4%	27.4%	30.7%	8.9%	63.9%	13.5%	19.7%
		Median	100.4%	7.1%	103.0%	27.4%	30.7%	8.9%	61.0%	13.1%	19.7%
	Level 4	Number	--	--	--	--	--	--	7	4	--
		Minimum	--	--	--	--	--	--	5.4%	11.2%	--
		Maximum	--	--	--	--	--	--	90.0%	45.7%	--
		Mean	--	--	--	--	--	--	48.5%	26.1%	--
		Median	--	--	--	--	--	--	51.4%	23.8%	--
	All Soil	Number	18	5	22	16	34	25	38	32	16
		Minimum	0.6%	0.1%	13.1%	0.7%	3.8%	0.2%	5.4%	1.2%	0.0%
		Maximum	161.6%	42.1%	182.8%	61.3%	114.0%	74.4%	182.7%	122.6%	70.7%
		Mean	102.2%	19.1%	80.3%	31.8%	37.2%	21.2%	78.3%	19.8%	25.7%
		Median	114.9%	20.3%	68.7%	35.6%	39.7%	17.1%	62.3%	14.7%	20.9%

**Table E-1. Evaluation of Accuracy - Relative Percent Differences versus Reference Laboratory Data Calculated for the Oxford ED2000
(Continued)**

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	24	4	3	13	20
		Minimum	1.1%	16.7%	42.9%	11.8%	0.4%
		Maximum	117.0%	33.2%	54.6%	69.0%	72.0%
		Mean	22.1%	27.9%	47.0%	42.4%	25.5%
		Median	9.5%	30.9%	43.4%	41.4%	19.3%
	Level 2	Number	5	5	3	4	6
		Minimum	4.6%	28.2%	28.9%	3.7%	12.4%
		Maximum	19.1%	42.0%	59.7%	42.4%	40.2%
		Mean	11.5%	34.5%	42.7%	22.9%	24.3%
		Median	10.5%	32.3%	39.3%	22.7%	24.3%
	Level 3	Number	6	4	6	4	8
		Minimum	0.3%	10.6%	11.2%	17.6%	6.7%
		Maximum	28.0%	35.7%	70.2%	36.4%	70.5%
		Mean	8.3%	25.4%	32.9%	27.4%	26.9%
		Median	3.8%	27.7%	18.2%	27.8%	23.6%
	Level 4	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Soil	Number	35	13	12	21	34
		Minimum	0.3%	10.6%	11.2%	3.7%	0.4%
		Maximum	117.0%	42.0%	70.2%	69.0%	72.0%
		Mean	18.2%	29.7%	38.9%	35.8%	25.6%
		Median	8.4%	31.5%	41.1%	31.6%	22.7%

Table E-1. Evaluation of Accuracy - Relative Percent Differences versus Reference Laboratory Data Calculated for the Oxford ED2000 (Continued)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
			Ref Lab	ERA Spike							
Sediment	Level 1	Number	4	4	17	3	20	7	3	15	3
		Minimum	130.9%	1.7%	12.3%	7.5%	0.1%	3.4%	181.3%	2.4%	16.1%
		Maximum	150.9%	29.5%	168.7%	19.7%	77.6%	23.1%	190.1%	72.2%	87.7%
		Mean	136.4%	9.2%	61.1%	12.7%	28.9%	13.9%	185.0%	25.1%	44.2%
		Median	131.9%	2.9%	42.3%	10.8%	24.8%	15.3%	183.5%	16.1%	28.8%
	Level 2	Number	3	3	4	4	3	4	18	4	4
		Minimum	104.1%	0.1%	2.1%	0.1%	4.6%	9.8%	70.5%	4.1%	1.7%
		Maximum	157.1%	0.4%	34.7%	5.1%	8.3%	13.6%	150.8%	8.6%	101.6%
		Mean	136.3%	0.3%	15.2%	1.9%	6.4%	11.8%	93.9%	6.1%	36.9%
		Median	147.6%	0.4%	12.0%	1.1%	6.2%	11.9%	90.3%	5.9%	22.1%
	Level 3	Number	3	3	2	3	3	10	4	3	3
		Minimum	92.7%	4.3%	17.9%	4.6%	4.7%	0.6%	67.2%	2.5%	0.9%
		Maximum	143.5%	74.8%	21.9%	23.8%	9.5%	19.0%	103.6%	15.8%	23.5%
		Mean	111.6%	28.1%	19.9%	14.0%	6.5%	7.9%	84.7%	7.0%	15.8%
		Median	98.7%	5.2%	19.9%	13.5%	5.4%	7.3%	84.1%	2.7%	23.1%
	Level 4	Number	--	--	--	--	--	--	6	--	--
		Minimum	--	--	--	--	--	--	29.8%	--	--
		Maximum	--	--	--	--	--	--	83.8%	--	--
		Mean	--	--	--	--	--	--	59.2%	--	--
		Median	--	--	--	--	--	--	65.9%	--	--
	All Sediment	Number	11	11	23	10	26	21	31	22	10
		Minimum	92.7%	0.1%	2.1%	0.1%	0.1%	0.6%	29.8%	2.4%	0.9%
		Maximum	157.1%	74.8%	168.7%	23.8%	77.6%	23.1%	190.1%	72.2%	101.6%
		Mean	127.8%	11.1%	49.5%	8.7%	23.7%	10.7%	94.8%	19.2%	32.8%
		Median	131.5%	2.2%	37.3%	6.3%	17.9%	10.3%	88.7%	11.5%	23.3%

**Table E-1. Evaluation of Accuracy - Relative Percent Differences versus Reference Laboratory Data Calculated for the Oxford ED2000
(Continued)**

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Sediment	Level 1	Number	17	5	5	6	18
		Minimum	0.3%	8.5%	3.2%	5.5%	10.9%
		Maximum	94.3%	44.1%	19.3%	51.4%	50.3%
		Mean	24.7%	29.4%	11.6%	22.9%	25.4%
		Median	19.7%	30.7%	11.7%	19.6%	22.2%
	Level 2	Number	6	4	4	8	5
		Minimum	2.3%	21.0%	5.3%	7.1%	3.2%
		Maximum	19.5%	41.1%	30.7%	51.7%	25.3%
		Mean	7.2%	29.2%	17.2%	29.3%	11.8%
		Median	4.8%	27.3%	16.4%	32.9%	8.8%
	Level 3	Number	4	3	3	3	4
		Minimum	2.0%	17.5%	12.8%	15.7%	1.0%
		Maximum	13.5%	37.3%	63.6%	28.6%	16.5%
		Mean	5.8%	28.7%	37.0%	20.8%	8.3%
		Median	3.9%	31.4%	34.5%	18.0%	7.8%
	Level 4	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Sediment	Number	27	12	12	17	27
		Minimum	0.3%	8.5%	3.2%	5.5%	1.0%
		Maximum	94.3%	44.1%	63.6%	51.7%	50.3%
		Mean	18.0%	29.2%	19.8%	25.6%	20.4%
		Median	8.6%	31.0%	13.5%	27.2%	17.4%

**Table E-1. Evaluation of Accuracy - Relative Percent Differences versus Reference Laboratory Data Calculated for the Oxford ED2000
(Continued)**

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
			Ref Lab	ERA Spike							
All Samples	ED2000	Number	29	16	45	26	60	46	69	54	26
		Minimum	0.6%	0.1%	2.1%	0.1%	0.1%	0.2%	5.4%	1.2%	0.0%
		Maximum	161.6%	74.8%	182.8%	61.3%	114.0%	74.4%	190.1%	122.6%	101.6%
		Mean	111.9%	13.6%	64.6%	22.9%	31.4%	16.4%	85.7%	19.6%	28.4%
		Median	117.7%	3.9%	49.7%	18.3%	24.8%	13.5%	78.1%	14.3%	23.3%
All Samples	All Developers	Number	206	110	320	209	338	363	558	392	192
		Minimum	0.1%	0.1%	0.2%	0.1%	0.1%	0.2%	0.0%	0.1%	0.0%
		Maximum	181.5%	162.0%	182.8%	168.1%	151.7%	111.1%	190.1%	135.2%	158.1%
		Mean	80.6%	62.7%	36.6%	29.6%	30.8%	24.6%	35.4%	30.9%	62.5%
		Median	84.3%	70.6%	26.2%	16.7%	26.0%	16.2%	26.0%	21.5%	58.6%

**Table E-1. Evaluation of Accuracy - Relative Percent Differences versus Reference Laboratory Data Calculated for the Oxford ED2000
(Continued)**

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
All Samples	ED2000	Number	62	25	24	38	61
		Minimum	0.3%	8.5%	3.2%	3.7%	0.4%
		Maximum	117.0%	44.1%	70.2%	69.0%	72.0%
		Mean	18.1%	29.4%	29.3%	31.2%	23.3%
		Median	8.6%	31.4%	23.0%	30.1%	19.3%
All Samples	All Developers	Number	403	195	177	218	471
		Minimum	0.3%	0.0%	0.0%	0.1%	0.0%
		Maximum	146.5%	127.1%	129.7%	129.5%	138.0%
		Mean	31.0%	32.0%	36.0%	42.2%	26.3%
		Median	25.4%	16.7%	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 Reference laboratory (Shealy Environmental Services, Inc.).

RPD Relative percent difference.

XRF X-ray fluorescence.

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for the Oxford ED2000

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
Soil	Low	Number	9	15	7	28	16	5	16	7
		Minimum	1.2%	1.8%	2.1%	2.5%	1.3%	0.4%	2.6%	6.4%
		Maximum	31.1%	19.2%	92.8%	64.1%	22.8%	1.2%	34.6%	22.6%
		Mean	8.2%	7.4%	24.5%	10.9%	8.3%	0.7%	14.7%	14.0%
		Median	2.2%	4.7%	5.4%	5.7%	5.3%	0.7%	13.5%	14.8%
	Medium	Number	5	4	7	4	7	13	4	7
		Minimum	2.5%	0.3%	1.5%	2.9%	0.8%	0.8%	2.4%	1.9%
		Maximum	3.1%	2.5%	3.6%	28.6%	4.4%	15.8%	2.9%	7.0%
		Mean	2.8%	1.6%	2.3%	10.6%	1.9%	2.5%	2.6%	4.0%
		Median	2.8%	1.7%	2.3%	5.4%	1.4%	1.4%	2.5%	4.0%
	High	Number	4	4	2	2	2	13	8	2
		Minimum	0.8%	0.2%	1.0%	0.8%	1.9%	0.9%	0.3%	2.1%
		Maximum	5.5%	1.0%	2.8%	1.9%	2.7%	48.5%	7.3%	5.1%
		Mean	2.5%	0.7%	1.9%	1.3%	2.3%	9.6%	2.3%	3.6%
		Median	1.8%	0.9%	1.9%	1.3%	2.3%	3.1%	1.7%	3.6%
	Very High	Number	--	--	--	--	--	7	5	--
		Minimum	--	--	--	--	--	1.2%	1.4%	--
		Maximum	--	--	--	--	--	17.4%	48.2%	--
		Mean	--	--	--	--	--	4.0%	23.5%	--
		Median	--	--	--	--	--	1.8%	23.2%	--
	All Soil	Number	18	23	16	34	25	38	33	16
		Minimum	0.8%	0.2%	1.0%	0.8%	0.8%	0.4%	0.3%	1.9%
		Maximum	31.1%	19.2%	92.8%	64.1%	22.8%	48.5%	48.2%	22.6%
		Mean	5.4%	5.2%	12.0%	10.3%	6.0%	5.0%	11.6%	8.3%
		Median	2.6%	3.1%	2.9%	5.6%	4.2%	1.6%	8.4%	6.0%

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for the Oxford ED2000 (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Low	Number	24	4	3	13	20
		Minimum	1.9%	1.4%	2.3%	4.5%	1.7%
		Maximum	53.9%	12.6%	4.0%	23.1%	28.9%
		Mean	8.1%	5.5%	3.4%	15.0%	7.8%
		Median	5.6%	4.0%	3.8%	17.2%	6.5%
	Medium	Number	5	5	3	4	6
		Minimum	0.8%	1.2%	0.6%	2.9%	1.6%
		Maximum	5.8%	4.0%	4.8%	13.3%	3.4%
		Mean	3.3%	2.8%	2.6%	5.9%	2.1%
		Median	3.7%	3.2%	2.4%	3.7%	1.7%
	High	Number	6	4	6	4	8
		Minimum	1.1%	0.8%	1.8%	4.1%	0.5%
		Maximum	15.8%	12.7%	9.4%	9.4%	2.2%
		Mean	4.3%	4.4%	3.5%	6.0%	1.2%
		Median	1.7%	2.0%	2.3%	5.2%	1.0%
	Very High	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Soil	Number	35	13	12	21	34
		Minimum	0.8%	0.8%	0.6%	2.9%	0.5%
		Maximum	53.9%	12.7%	9.4%	23.1%	28.9%
		Mean	6.8%	4.1%	3.2%	11.5%	5.3%
		Median	4.7%	2.7%	2.5%	10.9%	3.7%

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for the Oxford ED2000 (Continued)

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
Sediment	Low	Number	4	17	3	21	8	3	16	3
		Minimum	2.5%	1.1%	1.3%	0.6%	1.6%	0.1%	2.9%	8.0%
		Maximum	41.0%	69.9%	4.3%	47.0%	21.3%	0.5%	37.3%	44.5%
		Mean	13.3%	10.7%	2.8%	12.4%	7.1%	0.3%	11.8%	23.0%
		Median	4.9%	5.5%	2.6%	9.2%	4.8%	0.3%	8.4%	16.4%
	Medium	Number	4	4	4	3	4	19	4	4
		Minimum	1.3%	0.7%	1.0%	2.3%	1.0%	0.3%	0.2%	1.1%
		Maximum	2.8%	4.2%	26.0%	4.7%	3.9%	101.8%	2.5%	3.9%
		Mean	1.8%	1.9%	7.9%	3.6%	2.1%	11.2%	1.6%	2.6%
		Median	1.6%	1.3%	2.2%	3.8%	1.7%	1.5%	1.9%	2.6%
	High	Number	3	2	3	3	10	4	3	3
		Minimum	2.4%	4.4%	2.4%	2.2%	0.3%	0.5%	0.3%	1.4%
		Maximum	97.7%	14.5%	41.1%	4.6%	3.2%	3.6%	2.1%	3.0%
		Mean	34.6%	9.4%	15.5%	3.0%	1.4%	1.9%	0.9%	2.0%
		Median	3.6%	9.4%	3.1%	2.4%	1.4%	1.9%	0.4%	1.6%
	Very High	Number	--	--	--	--	--	6	--	--
		Minimum	--	--	--	--	--	0.4%	--	--
		Maximum	--	--	--	--	--	45.5%	--	--
		Mean	--	--	--	--	--	9.3%	--	--
		Median	--	--	--	--	--	2.1%	--	--
	All Sediment	Number	11	23	10	27	22	32	23	10
		Minimum	1.3%	0.7%	1.0%	0.6%	0.3%	0.1%	0.2%	1.1%
		Maximum	97.7%	69.9%	41.1%	47.0%	21.3%	101.8%	37.3%	44.5%
		Mean	14.9%	9.1%	8.6%	10.4%	3.6%	8.6%	8.6%	8.5%
		Median	2.8%	5.0%	2.6%	7.3%	2.0%	1.5%	4.9%	2.9%

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for the Oxford ED2000 (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Sediment	Low	Number	18	5	5	6	19
		Minimum	2.1%	2.8%	2.7%	11.5%	3.4%
		Maximum	12.4%	14.2%	23.8%	26.9%	13.4%
		Mean	5.5%	6.5%	8.0%	18.8%	9.3%
		Median	4.6%	5.1%	4.2%	18.3%	10.3%
	Medium	Number	6	4	4	8	5
		Minimum	2.2%	1.2%	0.9%	3.1%	1.2%
		Maximum	4.9%	3.6%	3.1%	40.9%	1.8%
		Mean	3.2%	2.4%	1.9%	19.2%	1.5%
		Median	3.0%	2.4%	1.7%	17.5%	1.7%
	High	Number	4	3	3	3	4
		Minimum	2.0%	1.7%	2.7%	6.2%	1.3%
		Maximum	3.7%	2.1%	41.4%	7.1%	2.4%
		Mean	2.8%	1.9%	15.7%	6.8%	1.7%
		Median	2.8%	1.8%	2.9%	7.1%	1.6%
	Very High	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Sediment	Number	28	12	12	17	28
		Minimum	2.0%	1.2%	0.9%	3.1%	1.2%
		Maximum	12.4%	14.2%	41.4%	40.9%	13.4%
		Mean	4.6%	4.0%	7.9%	16.9%	6.8%
		Median	4.1%	3.2%	3.0%	16.5%	6.1%

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for the Oxford ED2000 (Continued)

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
All Samples	ED2000	Number	29	46	26	61	47	70	56	26
		Minimum	0.8%	0.2%	1.0%	0.6%	0.3%	0.1%	0.2%	1.1%
		Maximum	97.7%	69.9%	92.8%	64.1%	22.8%	101.8%	48.2%	44.5%
		Mean	9.0%	7.1%	10.7%	10.4%	4.9%	6.7%	10.4%	8.4%
		Median	2.7%	4.0%	2.7%	5.6%	2.6%	1.5%	6.3%	4.7%
All Samples	All Developers	Number	206	320	209	338	363	558	392	192
		Minimum	0.5%	0.2%	0.4%	0.6%	0.1%	0.1%	0.2%	1.0%
		Maximum	97.7%	71.7%	92.8%	116.3%	58.3%	101.8%	115.6%	137.1%
		Mean	8.9%	11.2%	8.2%	15.9%	7.5%	5.2%	9.3%	14.3%
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for Oxford ED2000 (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
All Samples	ED2000	Number	63	25	24	38	62
		Minimum	0.8%	0.8%	0.6%	2.9%	0.5%
		Maximum	53.9%	14.2%	41.4%	40.9%	28.9%
		Mean	5.8%	4.1%	5.6%	13.9%	6.0%
		Median	4.3%	2.8%	2.8%	12.5%	4.7%
All Samples	All Developers	Number	403	195	177	218	471
		Minimum	0.3%	0.1%	0.6%	0.4%	0.1%
		Maximum	164.2%	98.8%	125.3%	86.1%	192.9%
		Mean	10.8%	7.2%	10.3%	12.5%	8.0%
		Median	7.0%	4.5%	5.2%	8.5%	5.3%

Notes:

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

Table E-3. Evaluation of Precision - Relative Standard Deviations Calculated for the Reference Laboratory

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

**Table E-3. Evaluation of Precision Relative Standard Deviations Calculated for the Reference Laboratory
(Continued)**

Matrix	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
All Soil	Number	35	13	13	21	35
	Minimum	0.0%	0.0%	2.3%	0.0%	1.0%
	Maximum	44.9%	22.7%	37.1%	18.1%	46.5%
	Mean	11.4%	8.9%	12.4%	8.4%	10.4%
	Median	10.0%	7.1%	7.5%	6.6%	9.1%
All Sediment	Number	27	12	10	17	27
	Minimum	0.6%	1.3%	1.0%	2.2%	1.4%
	Maximum	35.8%	37.3%	21.3%	21.9%	35.8%
	Mean	9.4%	10.0%	9.4%	8.4%	8.9%
	Median	7.3%	7.6%	6.6%	8.1%	6.9%
All Samples	Number	62	25	23	38	62
	Minimum	0.0%	0.0%	1.0%	0.0%	1.0%
	Maximum	44.9%	37.3%	37.1%	21.9%	46.5%
	Mean	10.6%	9.4%	11.1%	8.4%	9.8%
	Median	8.2%	7.4%	7.1%	7.2%	7.4%

Table E-4. Evaluation of the Effects of Inteferent Metals on RPDs (Accuracy) of Other Target Elements ¹

Parameter	Statistic	Lead Effects on Arsenic			Copper Effects on Nickel			Nickel Effects on Copper		
		<5	5 - 10	>10	<5	5 - 10	>10	<5	5 - 10	>10
Interferent/Metal Ratio		<5	5 - 10	>10	<5	5 - 10	>10	<5	5 - 10	>10
Number of Samples		29	6	9	43	5	14	37	1	8
RPD of Target Metal	Minimum	-112.2%	-125.1%	-168.7%	-94.3%	-17.0%	-117.0%	-74.4%	-16.1%	-43.9%
	Maximum	23.4%	-55.2%	-103.0%	20.6%	7.2%	3.3%	19.0%	-16.1%	13.6%
	Mean	-33.4%	-78.4%	-137.0%	-8.6%	-5.5%	-36.4%	-14.9%	-16.1%	-11.1%
	Median	-34.7%	-74.8%	-138.3%	-3.4%	-8.5%	-23.6%	-11.3%	-16.1%	-10.6%
RPD of Target Metal (Absolute Value)	Minimum	2.1%	55.2%	103.0%	0.3%	0.3%	3.3%	0.2%	16.1%	3.4%
	Maximum	112.2%	125.1%	168.7%	94.3%	17.0%	117.0%	74.4%	16.1%	43.9%
	Mean	35.1%	78.4%	137.0%	13.2%	8.3%	36.9%	16.4%	16.1%	16.2%
	Median	34.7%	74.8%	138.3%	7.2%	8.5%	23.6%	13.5%	16.1%	12.6%
Interferent Concentration Range	Minimum	26	640	1119	51	953	1006	71	378	1240
	Maximum	783	29881	12680	1259	1980	4365	852	378	3207
	Mean	188	9004	3570	253	1349	2624	206	378	2285
	Median	76	5087	2239	141	1160	2409	156	378	2284
Target Metal Concentration Range	Minimum	20	204	161	56	108	71	51	92	79
	Maximum	3229	3814	6307	3207	258	443	4365	92	143
	Mean	354	1481	1077	595	175	189	1341	92	114
	Median	148	1057	373	191	156	149	1006	92	121

Table E-4. Evaluation of the Effects of Inteferent Metals on RPDs (Accuracy) of Other Target Elements ¹ (Continued)

Parameter	Statistic	Zinc Effects on Copper			Copper Effects on Zinc		
		<5	5 - 10	>10	<5	5 - 10	>10
Interferent/Metal Ratio		<5	5 - 10	>10	<5	5 - 10	>10
Number of Samples		34	1	11	48	3	10
RPD of Target Metal	Minimum	-74.4%	-11.3%	-37.5%	-72.0%	-34.9%	-33.8%
	Maximum	19.0%	-11.3%	13.6%	4.2%	-10.9%	3.6%
	Mean	-13.7%	-11.3%	-13.2%	-24.3%	-22.3%	-17.0%
	Median	-10.7%	-11.3%	-15.9%	-21.0%	-20.9%	-17.3%
RPD of Target Metal (Absolute Value)	Minimum	0.2%	11.3%	1.0%	0.4%	10.9%	3.6%
	Maximum	74.4%	11.3%	37.5%	72.0%	34.9%	33.8%
	Mean	16.7%	11.3%	15.8%	24.5%	22.3%	17.8%
	Median	12.4%	11.3%	15.9%	21.0%	20.9%	17.3%
Interferent Concentration Range	Minimum	54	889	899	51	1027	1696
	Maximum	11812	889	9830	3072	1646	4365
	Mean	1416	889	3807	562	1311	2814
	Median	207	889	3940	179	1259	2329
Target Metal Concentration Range	Minimum	51	179	79	54	140	75
	Maximum	4365	179	299	11812	232	277
	Mean	1438	179	141	1932	173	168
	Median	1093	179	135	619	146	177

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-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements

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	--	--	--	--	1	1
			Minimum	--	--	--	--	-182.8%	182.8%
			Maximum	--	--	--	--	-182.8%	182.8%
			Mean	--	--	--	--	-182.8%	182.8%
			Median	--	--	--	--	-182.8%	182.8%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	1	1	7	7
			Minimum	-133.9%	71.7%	42.1%	42.1%	-157.2%	13.1%
			Maximum	-71.7%	133.9%	42.1%	42.1%	-13.1%	157.2%
			Mean	-111.7%	111.7%	42.1%	42.1%	-70.0%	70.0%
			Median	-120.7%	120.7%	42.1%	42.1%	-74.5%	74.5%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	2	2	1	1
			Minimum	-103.8%	73.7%	20.3%	20.3%	-49.7%	49.7%
			Maximum	-73.7%	103.8%	26.0%	26.0%	-49.7%	49.7%
			Mean	-88.7%	88.7%	23.2%	23.2%	-49.7%	49.7%
			Median	-88.7%	88.7%	23.2%	23.2%	-49.7%	49.7%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	2	2	--	--	--	--
			Minimum	-16.7%	0.6%	--	--	--	--
			Maximum	-0.6%	16.7%	--	--	--	--
			Mean	-8.6%	8.6%	--	--	--	--
			Median	-8.6%	8.6%	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	4	4	11	11
			Minimum	-131.5%	96.0%	-0.1%	0.1%	-168.7%	2.1%
			Maximum	-96.0%	131.5%	4.3%	4.3%	23.4%	168.7%
			Mean	-110.8%	110.8%	1.7%	1.8%	-48.0%	52.7%
			Median	-107.7%	107.7%	1.3%	1.3%	-38.7%	38.7%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (Continued)

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
Sediment	RF	Silty fine sand (tailings)	Number	5	5	5	5	12	12
			Minimum	-143.5%	92.7%	-74.8%	0.4%	-138.3%	17.9%
			Maximum	-92.7%	143.5%	5.2%	74.8%	-17.9%	138.3%
			Mean	-120.7%	120.7%	-20.6%	22.7%	-43.9%	43.9%
			Median	-130.9%	130.9%	-3.6%	5.2%	-37.0%	37.0%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	7	7	1	1	5	5
			Minimum	-161.6%	104.7%	7.1%	7.1%	-112.2%	16.9%
			Maximum	-104.7%	161.6%	7.1%	7.1%	-16.9%	112.2%
			Mean	-122.5%	122.5%	7.1%	7.1%	-47.3%	47.3%
			Median	-115.5%	115.5%	7.1%	7.1%	-32.7%	32.7%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	3	3	3	2	2
			Minimum	-157.1%	147.6%	-1.7%	0.1%	-132.5%	113.8%
			Maximum	-147.6%	157.1%	0.1%	1.7%	-113.8%	132.5%
			Mean	-151.9%	151.9%	-0.7%	0.7%	-123.2%	123.2%
			Median	-150.9%	150.9%	-0.4%	0.4%	-123.2%	123.2%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	2	2	--	--	6	6
			Minimum	-123.7%	121.2%	--	--	-142.7%	36.6%
			Maximum	-121.2%	123.7%	--	--	-36.6%	142.7%
			Mean	-122.4%	122.4%	--	--	-98.9%	98.9%
			Median	-122.4%	122.4%	--	--	-114.0%	114.0%
All	All		Number	29	29	16	16	45	45
			Minimum	-161.6%	0.6%	-74.8%	0.1%	-182.8%	2.1%
			Maximum	-0.6%	161.6%	42.1%	74.8%	23.4%	182.8%
			Mean	-111.9%	111.9%	-0.2%	13.6%	-63.4%	64.6%
			Median	-117.7%	117.7%	0.3%	3.9%	-49.7%	49.7%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (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
Soil	AS	Fine to medium sand (steel processing)	Number	3	3	2	2	3	3
			Minimum	16.7%	16.7%	50.2%	50.2%	-16.2%	1.0%
			Maximum	48.7%	48.7%	91.5%	91.5%	17.1%	17.1%
			Mean	35.0%	35.0%	70.8%	70.8%	0.6%	11.4%
			Median	39.5%	39.5%	70.8%	70.8%	1.0%	16.2%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	5	5	7	7	6	6
			Minimum	21.3%	21.3%	8.3%	8.3%	-29.6%	10.3%
			Maximum	54.2%	54.2%	61.2%	61.2%	-10.3%	29.6%
			Mean	38.5%	38.5%	29.7%	29.7%	-21.6%	21.6%
			Median	35.2%	35.2%	22.9%	22.9%	-24.5%	24.5%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	2	2	3	3
			Minimum	16.8%	16.8%	13.6%	13.6%	-36.6%	0.2%
			Maximum	44.2%	44.2%	114.0%	114.0%	0.2%	36.6%
			Mean	30.5%	30.5%	63.8%	63.8%	-19.4%	19.6%
			Median	30.5%	30.5%	63.8%	63.8%	-21.9%	21.9%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	4	4	2	2
			Minimum	--	--	-77.6%	40.6%	-32.2%	11.0%
			Maximum	--	--	-40.6%	77.6%	-11.0%	32.2%
			Mean	--	--	-61.8%	61.8%	-21.6%	21.6%
			Median	--	--	-64.4%	64.4%	-21.6%	21.6%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	5	5	11	11	4	4
			Minimum	-7.5%	1.4%	-66.5%	4.6%	-43.9%	7.1%
			Maximum	13.5%	13.5%	9.5%	66.5%	7.1%	43.9%
			Mean	2.1%	6.8%	-24.0%	25.7%	-13.7%	17.3%
			Median	1.4%	7.4%	-14.8%	14.8%	-9.1%	9.1%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (Continued)

Matrix	Site	Matrix Description	Statistic	Iron		Lead		Mercury	
				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	13	13	13	13	5	5
			Minimum	-150.8%	67.2%	-72.2%	2.4%	-24.0%	16.1%
			Maximum	-67.2%	150.8%	8.6%	72.2%	28.8%	28.8%
			Mean	-88.7%	88.7%	-19.1%	22.3%	-11.0%	22.5%
			Median	-86.1%	86.1%	-9.9%	9.9%	-20.1%	23.5%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	12	12	7	7	11	11
			Minimum	-138.0%	51.4%	-30.8%	1.2%	-41.9%	4.6%
			Maximum	-51.4%	138.0%	39.5%	39.5%	70.7%	70.7%
			Mean	-68.7%	68.7%	-0.1%	15.4%	16.7%	35.3%
			Median	-60.8%	60.8%	-2.9%	10.4%	10.5%	28.9%
Sediment	TL	Silt and clay (slag-enriched)	Number	7	7	4	4	3	3
			Minimum	-107.3%	40.8%	-22.3%	2.5%	23.1%	23.1%
			Maximum	-40.8%	107.3%	4.1%	22.3%	101.6%	101.6%
			Mean	-86.8%	86.8%	-7.8%	9.8%	70.8%	70.8%
			Median	-98.4%	98.4%	-6.5%	7.3%	87.7%	87.7%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	7	7	6	6	--	--
			Minimum	-90.0%	54.9%	-20.0%	4.3%	--	--
			Maximum	-54.9%	90.0%	45.7%	45.7%	--	--
			Mean	-74.7%	74.7%	7.3%	18.2%	--	--
			Median	-78.1%	78.1%	3.4%	15.4%	--	--
	All		Number	70	70	55	55	26	26
			Minimum	-190.1%	5.4%	-72.2%	1.2%	-41.9%	0.0%
			Maximum	42.1%	190.1%	122.6%	122.6%	101.6%	101.6%
			Mean	-84.3%	85.6%	-2.7%	20.2%	12.4%	28.4%
			Median	-78.4%	78.4%	-2.5%	14.5%	0.9%	23.3%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (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
Soil	AS	Fine to medium sand (steel processing)	Number	3	3	1	1	1	1
			Minimum	-16.7%	4.6%	32.3%	32.3%	17.3%	17.3%
			Maximum	-4.6%	16.7%	32.3%	32.3%	17.3%	17.3%
			Mean	-8.8%	8.8%	32.3%	32.3%	17.3%	17.3%
			Median	-5.2%	5.2%	32.3%	32.3%	17.3%	17.3%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	6	6	4	4	4	4
			Minimum	-21.0%	1.2%	26.5%	26.5%	-11.2%	11.2%
			Maximum	-1.2%	21.0%	32.1%	32.1%	59.7%	59.7%
			Mean	-11.8%	11.8%	30.1%	30.1%	32.7%	38.3%
			Median	-12.3%	12.3%	30.9%	30.9%	41.1%	41.1%
Soil	CN	Sandy loam (burn pit residue)	Number	3	3	2	2	2	2
			Minimum	-37.8%	6.1%	16.7%	16.7%	28.9%	28.9%
			Maximum	10.5%	37.8%	42.0%	42.0%	43.4%	43.4%
			Mean	-7.0%	18.1%	29.4%	29.4%	36.2%	36.2%
			Median	6.1%	10.5%	29.4%	29.4%	36.2%	36.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	3	3	--	--	--	--
			Minimum	-17.6%	1.5%	--	--	--	--
			Maximum	1.5%	17.6%	--	--	--	--
			Mean	-6.8%	7.8%	--	--	--	--
			Median	-4.4%	4.4%	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	11	11	5	5	4	4
			Minimum	-94.3%	2.0%	10.6%	10.6%	-34.5%	5.3%
			Maximum	20.6%	94.3%	44.1%	44.1%	19.1%	34.5%
			Mean	-22.7%	27.4%	28.4%	28.4%	-0.2%	17.1%
			Median	-12.4%	19.5%	28.2%	28.2%	7.4%	14.2%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (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	13	13	5	5	5	5
			Minimum	-13.5%	0.3%	17.5%	17.5%	-63.6%	3.2%
			Maximum	11.1%	13.5%	33.2%	33.2%	11.7%	63.6%
			Mean	1.1%	6.4%	29.1%	29.1%	-11.1%	19.5%
			Median	2.3%	5.4%	31.4%	31.4%	3.2%	11.7%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	11	11	3	3	1	1
			Minimum	-28.3%	0.3%	33.2%	33.2%	-65.4%	65.4%
			Maximum	8.4%	28.3%	37.8%	37.8%	-65.4%	65.4%
			Mean	-3.4%	6.9%	35.6%	35.6%	-65.4%	65.4%
			Median	0.3%	4.0%	35.7%	35.7%	-65.4%	65.4%
Sediment	TL	Silt and clay (slag-enriched)	Number	6	6	4	4	4	4
			Minimum	-34.5%	8.6%	8.5%	8.5%	14.2%	14.2%
			Maximum	-8.6%	34.5%	41.1%	41.1%	30.7%	30.7%
			Mean	-25.5%	25.5%	25.3%	25.3%	22.7%	22.7%
			Median	-28.5%	28.5%	25.8%	25.8%	23.0%	23.0%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	7	7	1	1	3	3
			Minimum	-117.0%	3.4%	28.9%	28.9%	14.2%	14.2%
			Maximum	7.2%	117.0%	28.9%	28.9%	70.2%	70.2%
			Mean	-47.5%	49.5%	28.9%	28.9%	46.4%	46.4%
			Median	-19.1%	19.1%	28.9%	28.9%	54.6%	54.6%
	All		Number	63	63	25	25	24	24
			Minimum	-117.0%	0.3%	8.5%	8.5%	-65.4%	3.2%
			Maximum	20.6%	117.0%	44.1%	44.1%	70.2%	70.2%
			Mean	-14.2%	18.0%	29.4%	29.4%	13.7%	29.3%
			Median	-5.4%	8.6%	31.4%	31.4%	15.8%	23.0%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (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	1	1	3	3
			Minimum	27.2%	27.2%	-33.9%	3.6%
			Maximum	27.2%	27.2%	3.6%	33.9%
			Mean	27.2%	27.2%	-14.3%	16.6%
			Median	27.2%	27.2%	-12.4%	12.4%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	7	7
			Minimum	-31.6%	20.4%	-48.7%	14.3%
			Maximum	24.8%	31.6%	-14.3%	48.7%
			Mean	-4.1%	26.7%	-24.9%	24.9%
			Median	-4.9%	27.5%	-23.2%	23.2%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	3	3
			Minimum	3.7%	3.7%	-47.3%	13.3%
			Maximum	3.7%	3.7%	-13.3%	47.3%
			Mean	3.7%	3.7%	-27.7%	27.7%
			Median	3.7%	3.7%	-22.6%	22.6%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	2	2
			Minimum	--	--	-34.9%	19.2%
			Maximum	--	--	-19.2%	34.9%
			Mean	--	--	-27.1%	27.1%
			Median	--	--	-27.1%	27.1%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	9	9	10	10
			Minimum	-51.4%	8.3%	-69.6%	11.4%
			Maximum	-8.3%	51.4%	-11.4%	69.6%
			Mean	-29.7%	29.7%	-35.5%	35.5%
			Median	-30.0%	30.0%	-34.6%	34.6%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (Continued)

Matrix	Site	Matrix Description	Statistic	Vanadium		Zinc	
				Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val
Sediment	RF	Silty fine sand (tailings)	Number	3	3	13	13
			Minimum	-40.7%	18.0%	-25.3%	1.0%
			Maximum	-18.0%	40.7%	4.2%	25.3%
			Mean	-31.4%	31.4%	-12.3%	13.1%
			Median	-35.7%	35.7%	-13.8%	13.8%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	10	10	11	11
			Minimum	-69.0%	11.8%	-72.0%	6.7%
			Maximum	-11.8%	69.0%	-6.7%	72.0%
			Mean	-44.6%	44.6%	-23.2%	23.2%
			Median	-51.6%	51.6%	-16.9%	16.9%
Sediment	TL	Silt and clay (slag-enriched)	Number	7	7	7	7
			Minimum	-7.1%	5.5%	-33.8%	10.9%
			Maximum	51.7%	51.7%	-10.9%	33.8%
			Mean	12.9%	18.4%	-19.6%	19.6%
			Median	12.0%	12.0%	-17.4%	17.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	3	6	6
			Minimum	-41.4%	34.0%	-70.5%	0.4%
			Maximum	-34.0%	41.4%	-0.4%	70.5%
			Mean	-37.2%	37.2%	-26.9%	26.9%
			Median	-36.4%	36.4%	-20.9%	20.9%
	All		Number	38	38	62	62
			Minimum	-69.0%	3.7%	-72.0%	0.4%
			Maximum	51.7%	69.0%	4.2%	72.0%
			Mean	-21.5%	31.2%	-22.9%	23.2%
			Median	-29.3%	30.1%	-19.3%	19.3%

Table E-5. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements (Continued)

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 (raw value).
RPD ABS Val	Relative Percent Difference (absolute value).