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Innovative Technology Verification Report

XRF Technologies for Measuring Trace Elements in Soil and Sediment

Xcalibur ElvaX XRF Analyzer

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Xcalibur ElvaX XRF Analyzer

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Office of Research and Development
U.S. Environmental Protection Agency

Notice

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.
Director
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Abstract

The Elvatech, Ltd. ElvaX (ElvaX) x-ray fluorescence (XRF) analyzer distributed in the United States by Xcalibur XRF Services (Xcalibur), 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 ElvaX 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 ElvaX 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 ElvaX is a portable bench-top energy-dispersive XRF analyzer. The ElvaX is capable of detecting elements from sodium through plutonium and can be applied in the jewelry, metallurgy, customs, forensics, medical diagnostics, food testing, and environmental testing markets. The ElvaX can be used for qualitative or quantative analysis of metal alloys, liquid food, and biological samples. The ElvaX can analyze liquids and powders as well as samples deposited on surfaces or filters.

The ElvaX analyzer system includes two primary components: an XRF spectrometer and a personal computer. The XRF spectrometer contains a 5-watt x-ray tube excitation source with tungsten, titanium, or rhodium as the anode target material and with an adjustable 4- to 50-kilovolt power supply. The detector is a Peltier-cooled, solid-state silicon-PiN diode with 180-electron volt resolution. The XRF spectrometer may be set up in the field but must be in a stable environment. No portable battery systems are currently available for the ElvaX spectrometer.

A personal computer (laptop) with Microsoft Windows Millennium Edition® software is used to operate the XRF spectrometer and specifically to select x-ray tube parameters, store data, and provide radiation safety. The laptop is also used to display the x-ray spectrum and to process the data. Some examples of data processing steps included automatic peak search, overlapped peak deconvolution, background removal, automatic element identification, and background subtraction.

This report describes the results of the evaluation of the ElvaX 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 ElvaX 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 Elvatech Ltd. ElvaX XRF analyzer (distributed by Xcalibur) 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, 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

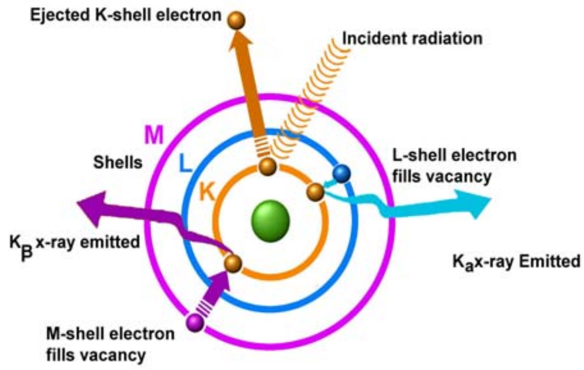

Figure 1-1. The XRF process.

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.

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

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

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

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

1.5 Properties of the Target Elements

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

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

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

1.5.1 Antimony

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

1.5.2 Arsenic

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

1.5.3 Cadmium

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

1.5.4 Chromium

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

1.5.5 Copper

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

1.5.6 Iron

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

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

1.5.7 Lead

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

1.5.8 Mercury

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

1.5.9 Nickel

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

1.5.10 Selenium

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

1.5.11 Silver

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

1.5.12 Vanadium

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

1.5.13 Zinc

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

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Chapter 2
Field Sample Collection Locations

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

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

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

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

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

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

2.1 Alton Steel Mill Site

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

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

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

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

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

Notes (in order of appearance in table):

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

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

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

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

2.2 Burlington Northern-ASARCO Smelter Site

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

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

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

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

2.3 Kennedy Athletic, Recreational and Social Park Site

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

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

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

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

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

2.4 Leviathan Mine Site

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

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

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

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

2.5 Navy Surface Warfare Center, Crane Division Site

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

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

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

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

2.6 Ramsay Flats-Silver Bow Creek Site

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

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

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

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

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

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

2.7 Sulphur Bank Mercury Mine Site

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

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

and gravel with trace silt. The matrix of the background soil sample was brown sandy loam.

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

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

2.8 Torch Lake Superfund Site

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

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

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

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

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

2.9 Wickes Smelter Site

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

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

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

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

Chapter 3
Field Demonstration

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

3.1 Bulk Sample Processing

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

3.1.1 Bulk Sample Collection and Shipping

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

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

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

3.1.2 Bulk Sample Preparation and Homogenization

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

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

After each bulk sample had been sieved and crushed, the sample was mixed and homogenized using a Model T 50A Turbula shaker-mixer. This shaker was capable of handling up to 50 gallons (190 liters) of sample material; thus, this shaker could handle the complete volume of each bulk sample. Bulk samples of smaller volume were mixed and homogenized using a Model T 10B Turbula shaker-mixer that was capable of handling up to 10 gallons (38 liters).

Aliquots from each homogenized bulk sample were then sampled and analyzed in triplicate for the 13 target elements using ICP-AES and CVAA. If the relative percent difference between the highest and lowest result exceeded 10 percent for any element, the entire batch was returned to the shaker-mixer for additional homogenization. The entire processing scheme for the bulk samples is shown in Figure 3-1.

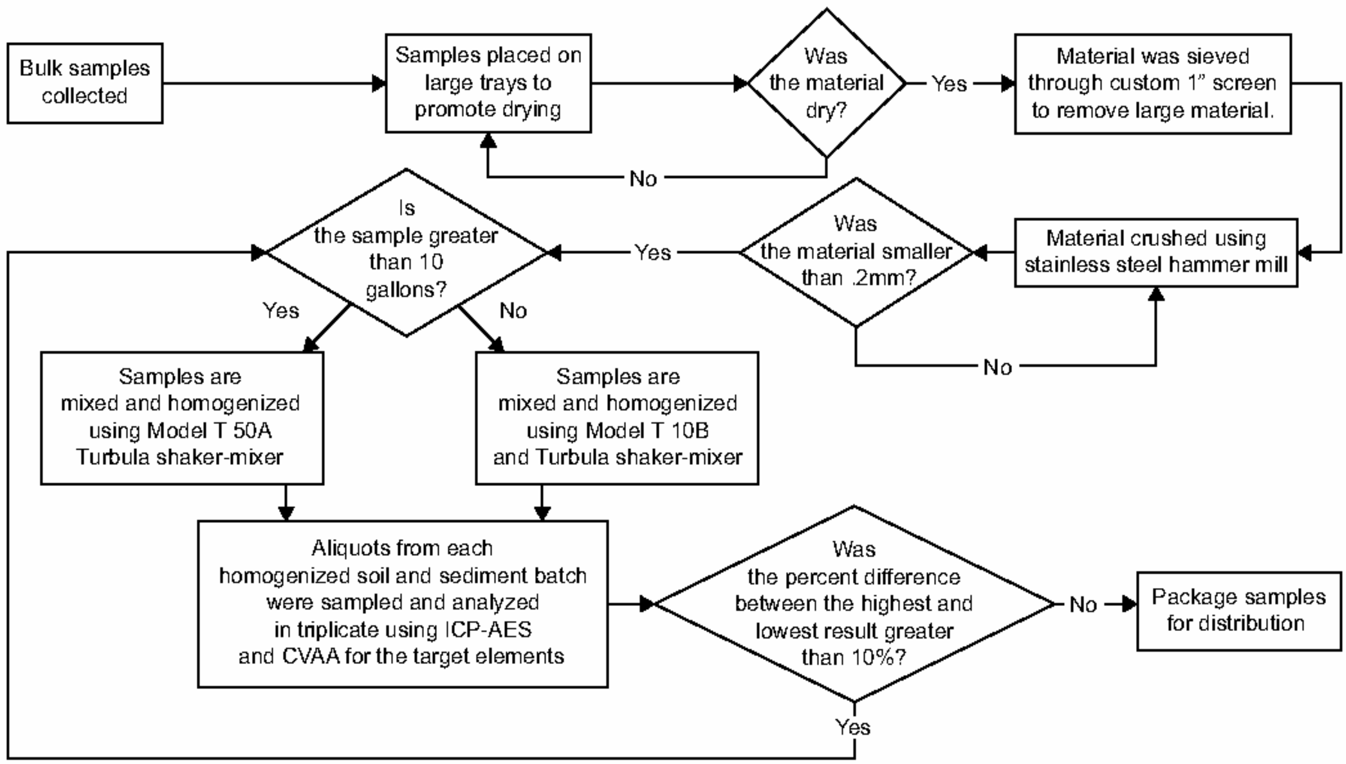

Figure 3-1. Bulk sample processing diagram.

3.2 Demonstration Samples

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

3.2.1 Environmental Samples

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

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

3.2.2 Spiked Samples

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

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

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

3.2.3 Demonstration Sample Set

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

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

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

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

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

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

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

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

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

3.3 Demonstration Site and Logistics

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

3.3.1 Demonstration Site Selection

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

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

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

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

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

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

3.3.2 Demonstration Site Logistics

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

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

Figure 3-2. KARS Park recreation building.

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

Figure 3-4. Visitors day presentation.

3.3.3 EPA Demonstration Team and Developer Field Team Responsibilities

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

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

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

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

3.3.4 Sample Management during the Field Demonstration

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

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

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

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

Figure 3-5. Sample storage room.

3.3.5 Data Management

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

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

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

Chapter 4
Evaluation Design

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

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

4.1 Evaluation Objectives

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

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

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

4.2 Experimental Design

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

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

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

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

4.2.1 Primary Objective 1 — Method Detection Limits

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

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

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:

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

where

RSD = Relative standard deviation

SD = Standard deviation

$\overline{C}$ = Mean concentration.

The standard deviation was calculated using the equation:

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

where

SD = Standard deviation

n = Number of replicate samples

C_k = Concentration of sample K

$\overline{C}$ = Mean concentration.

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

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

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

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

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

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

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

4.2.4 Primary Objective 4 — Impact of Chemical and Spectral Interferences

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

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

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

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

4.2.5 Primary Objective 5 — Effects of Soil Characteristics

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

4.2.6 Primary Objective 6 — Sample Throughput

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

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

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

4.2.7 Primary Objective 7 — Technology Costs

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

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

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

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

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

4.2.8 Secondary Objective 1 — Training Requirements

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

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

4.2.9 Secondary Objective 2 — Health and Safety

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

4.2.10 Secondary Objective 3 — Portability

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

4.2.11 Secondary Objective 4 — Durability

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

4.2.12 Secondary Objective 5 — Availability

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

4.3 Deviations from the Demonstration Plan

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

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

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

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

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

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

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

Chapter 5
Reference Laboratory

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

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

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

5.1 Selection of Reference Methods

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

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

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

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

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

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

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

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

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

5.2 Selection of Reference Laboratory

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

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

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

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

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

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

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

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

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

5.3 QA/QC Results for Reference Laboratory

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

5.3.1 Reference Laboratory Data Validation

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

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

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

5.3.2 Reference Laboratory Technical Systems Audit

The TSA of the Shealy laboratory was conducted by the project chemist on October 19, 2004, as part of the selection process for the reference laboratory. The audit included the review of element analysis practices (including sample preparation) for 12 elements by EPA Methods 3050B and 6010B and for total mercury by EPA Method 7471A. All decision-making personnel for Shealy were present during the TSA, including the laboratory director, QA officer, director of inorganics analysis, and the inorganics laboratory supervisor.

Project-specific requirements were reviewed with the Shealy project team as were all the QA criteria and reporting requirements in the demonstration plan. It was specifically noted that the demonstration samples would be dried, ground, and sieved before they were submitted to the laboratory, and that the samples would be received with no preservation required (specifically, no chemical preservation and no ice). The results of the performance audit were also reviewed.

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

5.3.3 Other Reference Laboratory Data Evaluations

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

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

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:
[1]Values shown in bold fall outside the 75 to 125 percent acceptance criterion for percent recovery.
ERA = Environmental Resource Associates, Inc.
NC = Not calculated.
%R = Percent recovery.
Source of certified values: Environmental Resource Associates, Inc.
Sb Antimony
As Arsenic
Cd Cadmium
Cr Chromium
Cu Copper
Fe Iron
Pb Lead
Hg Mercury
Ni Nickel
Se Selenium
Ag Silver
V Vanadium
Zn Zinc

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

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

Notes:
[1]Values shown in bold fall outside precision criterion of less than or equal to 25 %RSD.
%RSD = Percent relative standard deviation.
Based on the three to seven replicate samples included in Blends 1 through 70.

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

Chapter 6
Technology Description

The ElvaX XRF analyzer is manufactured by Elvatech, Ltd. in Kiev, Ukraine and distributed in the United States by Xcalibur XRF Services, Inc. This chapter provides a technical description of the ElvaX based on information obtained from Xcalibur and observation of this analyzer during the field demonstration. This chapter also provides Xcalibur contact information, where additional technical information may be obtained.

6.1 General Description

The ElvaX is a portable energy-dispersive XRF analyzer. The ElvaX is capable of detecting elements from sodium (atomic number 11) through plutonium (atomic number 94) and can be applied in the jewelry, metallurgy, customs, forensics, medical diagnostics, food testing, and environmental testing markets. The ElvaX can be used for qualitative or quantitative analysis of metal alloys, liquid food, and biological samples. The ElvaX can analyze liquids and powders as well as samples deposited on surfaces or filters.

The ElvaX analyzer system includes two primary components: an XRF spectrometer, and a personal computer. The XRF spectrometer contains a 5-watt x-ray tube excitation source with tungsten, titanium, or rhodium as the anode target material and with an adjustable 4- to 50–kilovolt (kV) power supply. The detector is a Peltier-cooled, solid-state silicon-PiN diode with 180-electron volt (eV) resolution. The XRF spectrometer may be set up in the field but must be in a stable environment. No portable battery systems are currently available for the ElvaX spectrometer.

A personal computer (laptop) with Microsoft Windows Millennium Edition (ME) software is used to operate the XRF spectrometer and specifically to select x-ray tube parameters, store data, and provide radiation safety. The laptop is also used to display the x-ray spectrum and to process the data. Some examples of data processing steps included automatic peak search, overlapped peak deconvolution, background removal, automatic element identification, and background subtraction.

The ElvaX analyzer can be calibrated using standardless fundamental parameters (FP), site-specific samples, or known standards. An experienced operator can set up the instrument and peripherals and initialize the computer software in 1 to 2 hours, while an inexperienced technician may require 2 to 3 hours. The technical specifications for the ElvaX XRF analyzer are presented in Table 6-1. The ElvaX analyzer is shown in a bench-top configuration in Figure 6-1.

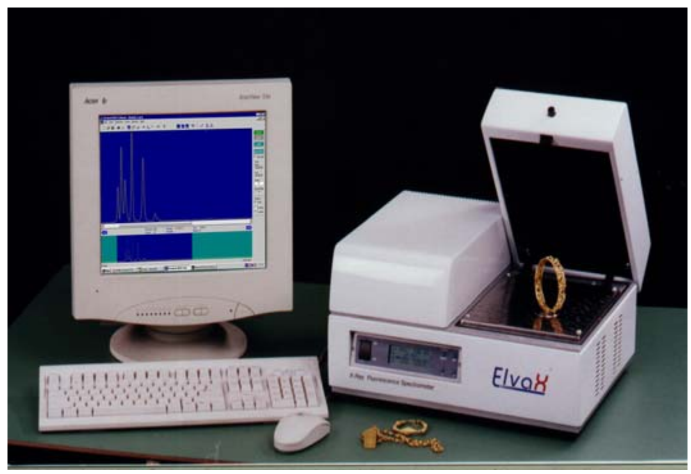

Figure 6-1. ElvaX XRF analyzer set up for bench-top analysis.

6.2 Instrument Operations during the Demonstration

The ElvaX analyzer and accessories were shipped to the demonstration site in three boxes. The instrument was contained in an overpacked cardboard box with styrofoam padding. The instrument was housed in an aluminum case inside the box for further protection. The peripherals were shipped in two additional cardboard boxes and included some soil standards, the laptop computer, and disposable laboratory supplies.

6.2.1 Set up and Calibration

The ElvaX was calibrated using data from pre-demonstration soil samples of known element concentration along with NIST standards. The calibration information was designed to account for differences in sample matrix and to cover the concentration range of elements found in pre-demonstration samples. The ElvaX analyzer was set on a bench and plugged into a 110-volt (V) electrical outlet. After the instrument was connected to an accompanying laptop computer, the ElvaX software was initialized and the pre-demonstration calibration information loaded. The ElvaX detector was allowed to warm up for about 10 minutes before analysis began. The ElvaX software is self-explanatory for instrument start-up; each menu guides the user through the process of turning on the x-ray tube and initializing the spectrometer optics and detector. The elements to be evaluated and their characteristic energy wavelengths and units of measure were selected through the computer software. The calibration of the XRF analyzer was verified by using the calibration reference materials provided with the NIST standards.

6.2.2 Demonstration Sample Processing

A two-person field team was provided during the field demonstration to analyze samples using the ElvaX. One field team member was a Ph.D. chemist supplied by the instrument manufacturer (ElvaTech), who operated the instrument. The second field team member was a sales engineer from Xcalibur, who served as the sample processing and data technician.

The Xcalibur representative noted that the instrument could be operated by a single trained operator but that a second person was provided for sample preparation so that all samples could be analyzed within the designated week of the field demonstration.

Table 6.1. Xcalibur ElvaX XRF Analyzer Technical Specifications

Weight:	18 kilograms (kg)
Size:	43 x 34 x 21 centimeters
Excitation source:	5-watt x-ray tube; 4 to 50 kV (1 to 100 microamps [μA]) adjustable power supply; tungsten anode (titanium and rhodium also available); air cooled; 0.14 millimeter (mm) Beryllium window; stability 0.1% over 8 hours.
Detector:	PF-550 from Moxtek, Inc., 7 mm^2 Si-PiN, 8 mm beryllium window, Peltier cooled, 180-eV resolution (full width of peak at half maximum height [FWHM]) at 5.9 keV.
Signal Processing:	Multi-channel analyzer; fast-shaping amplifier; pile-up rejection; automatic adaptation to count rate; ADC resolution 4,096 channels, 10^{32} counts/channel (with successive approximation and "sliding scaling"); real and "live" time.
Software:	ElvaX menu-driven software (Windows 9x/2000/NT/XP) with USB support for: • Instrument control – tube parameters, spectrometric processor, detector temperature, radiation safety, data acquisition, and sample and filter selection. • Display – spectra, marker scaling, peak attributes, analysis parameters. • Data processing – calibrations, automatic peak search and identification, deconvolution of overlapped peaks, background subtraction, and analytical intensities. • Quantitative analysis – standardless FP, FP regression with post processing, full-square regression with standards.
Element Range:	From sodium (atomic number 11) through plutonium (atomic number 94).
Power:	110-220V, 50 hertz (Hz), 50 W.

Steps in sample preparation included:

- Labeling each cup to identify the sample.
- Filling the sample cup with the homogenized soil or sediment sample (see Figure 6-2).
- Placing Mylar film on the sample cup with a snap ring to hold the film in place.
- Gently tapping the inverted sample cup against a hard surface to ensure good contact with the Mylar film and a uniform surface for analysis.

Figure 6-2. Xcalibur technician preparing samples for analysis.

After these sample preparation steps, the sample cups were passed to the instrument operator and then manually placed in the spectrometer chamber for analysis. One sample was analyzed at a time and required 6 to 8 minutes of instrument run time. A minute or two of additional instrument operator time was needed between each sample analysis to record the data to the personal computer and to reduce the data (Figure 6-3). Once the analysis of a sample batch was completed, the used sample cups were emptied, cleaned, and reused. (This step was needed only because of a shortage of sample cups in the supplies package.)

The observer noted that a standard operations manual should be developed that discusses applicable sample media, sample preparation, calibration, and quality control checks for environmental samples. Elvatech and Xcalibur have worked with users of the ElvaX to develop sample preparation and analysis techniques for environmental samples, but these procedures are not well documented.

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Figure 6-3. Instrument setup during the field demonstration.

6.3 General Demonstration Results

The two-person field team analyzed all 326 soil and sediment samples using the ElvaX analyzer in 4.5 days (excluding equipment unpacking, setup, and re-packing), thus averaging 72 samples per day. Routine analysis for this demonstration involved analyzing about six samples per hour (10 minutes per sample), with spectrometer run time varying between 6 and 8 minutes. Analytical results were recorded using the ElvaX computer software developed specifically for the ElvaX analyzer. The data were reduced by the instrument operator using the same software and then transferred to the data technician on a CD for final formatting into an Excel spreadsheet on a second laptop computer. The observer noted that the data processing observed during this demonstration were labor intensive but could be improved, if a tight time deadline were not in place, by using a single operator for all tasks.

6.4 Contact Information

Additional information on the ElvaX XRF analyzer is available from the following source:

Mr. Ron Williams
Xcalibur XRF Services, Inc.
1340 Lincoln Avenue, Unit #6
Holbrook, NY 11749
Telephone: (631) 435-9749
Cellular: (516) 885-7398
Email: ronupa@aol.com

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Chapter 7
Performance Evaluation

As discussed in Chapter 6, Xcalibur analyzed all 326 demonstration samples of soil and sediment at the field demonstration site between January 25 and 29, 2005. (Arrival of Xcalibur staff was delayed by weather, precluding the analysis of any demonstration samples on Monday, January 24, 2005. For this reason, the Xcalibur team remained at the demonstration site until Saturday, January 29, 2005, to complete the sample analyses.) A complete set of electronic data for the ElvaX in Microsoft Excel® spreadsheet format was delivered to Tetra Tech when Xcalibur departed from the demonstration site on January 29, 2005. All the data provided by Xcalibur are tabulated and compared with the reference laboratory data and the ERA-certified spike concentrations in Appendix D.

The ElvaX 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. Xcalibur reported non-detect values as “0”, or else reported no result (Appendix D). In selecting samples from among the 12 blends for the calculation of MDLs, blends where one or more of the seven replicates was reported as non-detect were generally not used. In essence, this meant that all seven replicates had to have detected concentrations, as reported by Xcalibur, to calculate an MDL for a blend. Because no blends met this requirement for selenium, one blend was used for the MDL calculation where only six of the seven replicates incorporated detections for selenium. Iron was not included in the MDL evaluation, as was discussed in Section 4.2.1.

The MDLs calculated for the ElvaX are presented in Table 7-1. In addition to selenium, only a few MDLs (between one and three) could be calculated for vanadium, mercury, and cadmium. Five or more MDLs could be calculated for the remaining target elements. Also shown in Table 7-1 are the mean MDLs calculated for each target element, which are classified as follows:

- Very low (1 to 20 ppm): antimony and mercury.
- Low (20 to 50 ppm): arsenic, copper, nickel, silver, vanadium, and zinc.
- Medium (50 to 100 ppm): cadmium, chromium, and lead.
- High (greater than 100 ppm): selenium.

The highest mean MDL for a target element was 156 ppm for selenium; no other mean MDLs were above 81 ppm. As noted above, this mean MDL was based on only a single MDL blend and; therefore, is somewhat uncertain. Blend 8 from the Wickes Smelter site produced anomalously high soil MDLs for antimony, cadmium, nickel, and silver. Blend 8 was a roaster slag matrix that contained high concentrations of other potentially interfering elements, such as arsenic, copper, lead, and zinc. Generalized biases in the MDL blend concentrations were also apparent for some elements. For example, the ElvaX reported detections in multiple MDL blends for antimony that were reported as nondetect by the reference laboratory (which used a more sensitive method), indicating the possibility of a generalized high bias at low concentrations for this element. Conversely, the ElvaX reported nondetections for chromium and vanadium in multiple blends where the reference laboratory reported concentrations that should have been detectable by the ElvaX (that is, concentrations near or above the final mean MDL calculated for these elements), indicating a potential low bias.

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Xcalibur ElvaX[1]

Matrix	Blend No.	Antimony ElvaX MDL[2]	Antimony ElvaX Conc.[3]	Antimony Ref. Lab Conc[4]	Arsenic ElvaX MDL[2]	Arsenic ElvaX Conc.[3]	Arsenic Ref. Lab Conc.[4]	Cadmium ElvaX MDL[2]	Cadmium ElvaX Conc.[3]	Cadmium Ref. Lab Conc.[4]	Chromium ElvaX MDL[2]	Chromium ElvaX Conc.[3]	Chromium Ref. Lab Conc.[4]
Soil	2	**12**	18	17	NC	ND	2	NC	ND	ND	**44**	204	167
Soil	5	**3**	1	ND	**58**	95	47	NC	ND	2	NC	ND	121
Soil	6	**23**	33	8	NC	382	477	NC	ND	12	NC	ND	133
Soil	8	**51**	191	118	NC	4289	3,943	**86**	106	91	NC	ND	55
Soil	10	**11**	6	ND	**47**	75	39	NC	ND	1	**36**	84	116
Soil	12	**10**	64	62	NC	698	559	**46**	310	263	**63**	73	101
Soil	18	**15**	6	ND	**34**	67	9	NC	ND	ND	**88**	137	150
Sediment	29	**16**	7	ND	NC	ND	10	NC	ND	ND	NC	ND	63
Sediment	31	**13**	6	ND	**70**	52	11	NC	ND	ND	**76**	114	133
Sediment	32	**13**	4	ND	**16**	80	31	NC	ND	ND	NC	ND	75
Sediment	39	**11**	2	ND	**42**	60	14	NC	ND	ND	**74**	75	102
Sediment	65	**15**	6	11	**49**	424	250	**25**	61	44	NC	215	303
Mean ElvaX MDL		**16**			**45**			**52**			**64**		

Matrix	Blend No.	Copper ElvaX MDL[2]	Copper ElvaX Conc.[3]	Copper Ref. Lab Conc[4]	Lead ElvaX MDL[2]	Lead ElvaX Conc.[3]	Lead Ref. Lab Conc.[4]	Mercury ElvaX MDL[2]	Mercury ElvaX Conc.[3]	Mercury Ref. Lab Conc.[4]	Nickel ElvaX MDL[2]	Nickel ElvaX Conc.[3]	Nickel Ref. Lab Conc.[4]
Soil	2	**11**	38	47	NC	1206	1200	NC	ND	ND	**25**	92	83
Soil	5	**20**	65	49	**57**	171	78	NC	ND	ND	**41**	48	60
Soil	6	**60**	177	160	NC	8321	3986	NC	ND	1	**37**	59	70
Soil	8	NC	1134	1,243	NC	54373	33,429	NC	ND	15	**80**	68	57
Soil	10	**24**	36	31	**91**	115	72	NC	ND	0	**31**	67	60
Soil	12	NC	852	747	NC	6562	4,214	NC	ND	2	**19**	78	91
Soil	18	**16**	29	50	NC	ND	17	**22**	17	56	**41**	179	213
Sediment	29	NC	2648	1986	**96**	135	33	NC	ND	0	**36**	110	72
Sediment	31	NC	1818	1514	**87**	160	51	NC	ND	ND	**75**	203	196
Sediment	32	**30**	19	36	NC	ND	26	NC	ND	ND	**36**	117	174
Sediment	39	**28**	62	94	NC	ND	27	NC	ND	ND	**45**	142	202
Sediment	65	**16**	213	69	**76**	51	25	**17**	12	32	**23**	183	214
Mean ElvaX MDL		**25**			**81**			**19**			**41**		

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Xcalibur ElvaX[1] (Continued)

Matrix	Blend No.	Selenium			Silver			Vanadium			Zinc		
		ElvaX MDL[2]	ElvaX Conc.[3]	Ref. Lab Conc[4]	ElvaX MDL[2]	ElvaX Conc.[3]	Ref. Lab Conc.[4]	ElvaX MDL[2]	ElvaX Conc.[3]	Ref. Lab Conc.[4]	ElvaX MDL[2]	ElvaX Conc.[3]	Ref. Lab Conc.[4]
Soil	2	NC	ND	ND	NC	ND	ND	NC	ND	1	**28**	65	24
Soil	5	NC	ND	ND	NC	ND	1	**45**	33	55	**65**	368	229
Soil	6	NC	ND	ND	**24**	26	14	NC	ND	56	NC	1085	886
Soil	8	NC	ND	ND	**120**	160	144	NC	ND	34	NC	6364	5,657
Soil	10	NC	ND	ND	NC	ND	ND	NC	ND	51	**61**	173	92
Soil	12	**156** [5]	71 [5]	15	**40**	44	38	NC	ND	45	NC	3361	2,114
Soil	18	NC	ND	ND	NC	ND	ND	NC	ND	67	**21**	106	90
Sediment	29	NC	ND	ND	NC	ND	ND	NC	ND	96	**29**	177	160
Sediment	31	NC	ND	ND	NC	ND	6	NC	ND	76	**19**	103	137
Sediment	32	NC	ND	5	**18**	9	ND	NC	ND	57	**43**	110	69
Sediment	39	NC	ND	ND	NC	ND	ND	NC	ND	38	**85**	208	137
Sediment	65	NC	ND	22	**34**	46	41	NC	ND	31	NC	3425	1,843
Mean ElvaX MDL		**156**			**47**			**45**			**44**		

Notes:

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

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

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

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

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

Conc. Concentration.

MDL Method detection limit.

NC The MDL was not calculated because reference laboratory concentrations exceeded five times the expected MDL range (approximately 50 ppm, depending on the element) or an insufficient number of detected concentrations were reported.

ND One or more results for this blend were reported as "Not Detected." Excepted as noted, blends with one or more ND result as reported by the XRF analyzer were not used for calculating the MDL for this element.

Ref. Lab. Reference laboratory.

The mean MDLs calculated for the ElvaX are compared in Table 7-2 with the mean MDLs for all XRF instruments 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 ElvaX were lower than the available mean MDLs calculated from EPA Method 6200 data for all elements except lead. When compared with the overall average results for all eight XRF instruments that participated in the demonstration, the ElvaX exhibited high relative mean MDLs for arsenic, lead, selenium, silver, and vanadium. Mean MDLs for the ElvaX were somewhat lower than the all-instrument means for antimony, cadmium, chromium, and nickel. The ElvaX and all-instrument means were essentially equivalent for copper, mercury, and zinc.

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, ranged from 16 blends for vanadium to 70 blends for iron. RPDs between the mean concentrations obtained from the ElvaX and the reference laboratory were calculated for each blend that met the criteria. Table 7-3 presents the median RPDs for each target element, along with the number of RPD results used to calculate the median. These statistics are provided for all blends 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).

Table 7-2. Comparison of Mean ElvaX MDLs to All-Instrument Mean MDLs and EPA Method 6200 Data[1]

Element	ElvaX Mean MDLs[2]	All XRF Instrument Mean MDLs[3]	EPA Method 6200 Mean Detection Limits[4]
Antimony	16	61	55 [5]
Arsenic	45	26	92
Cadmium	52	70	NR
Chromium	64	83	376
Copper	25	23	171
Lead	81	40	78
Mercury	19	23	NR
Nickel	41	50	100 [5]
Selenium	156 [5]	8	NR
Silver	47	42	NR
Vanadium	45	28	NR
Zinc	44	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 overall average of the mean MDLs calculated for all eight XRF instruments that participated in this EPA technology demonstration.

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

5 Only one value reported.

EPA U.S. Environmental Protection Agency.

MDL Method detection limit.

NR Not reported; no MDLs reported for this element.

Accuracy was classified as follows for the target elements based on the overall median RPDs:

- Very good (median RPD less than 10 percent): None.
- Good (median RPD between 10 and 25 percent): cadmium, iron, nickel, and silver.
- Fair (median RPD between 25 percent and 50 percent): antimony, arsenic, chromium, copper, and zinc.
- Poor (median RPD greater than 50 percent): lead, mercury, selenium, and vanadium.

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 for the demonstration showed that they were generally right-skewed or lognormal.) However, the classification of the elements based on accuracy stayed the same for all target elements except one (silver) when the mean rather than the median RPD was used for the evaluation (Table E-1). Review of the median RPDs revealed few trends with respect to media type (soil versus sediment) or concentration level. The most notable trends are summarized below:

- Higher overall median RPDs were observed in sediment than in soil for silver, vanadium, and zinc. RPDs were generally high for these metals in sediment blends associated with the Leviathan Mine, Torch Lake, and Ramsey Flats sampling sites.

- High median RPDs in the soil matrices were observed in the Level 1 samples for arsenic (with concentrations between 50 and 500 ppm). The median RPDs of 58.3 percent at this concentration level (classified in the "poor" range) was much higher than those for higher concentration levels in soil, where the median RPDs were in the "good" range. The high RPDs in the Level 1 soil samples for arsenic appeared to be generalized and not traceable to specific blends or sampling sites.

- For many other target elements, however, accuracy appeared to decrease with increasing concentration. RPDs for chromium, selenium, and vanadium increased as concentrations increased from the Level 1 to the Level 3 ranges for both soil and sediment. For seven other target elements, RPDs decreased in Level 2 soil and sediment relative to the Level 1 samples, indicating improved accuracy, but then increased again in Level 3 samples. These observations imply that the ElvaX provides the best overall accuracy over a fairly narrow range of moderate element concentrations. Accuracy appears to decline in more complex soil and sediment matrixes with high element concentrations.

As an additional basis for 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 ElvaX were equivalent to or below the all-instrument medians for five of the 13 target elements. The median RPDs for the ElvaX were somewhat higher than the all-instrument medians for arsenic, chromium, copper, and zinc, and were significantly higher for lead, mercury, selenium, and vanadium.

Section 5.3.3 discussed how the reference laboratory data for antimony were consistently biased low when compared with the ERA-certified spike concentrations. This effect may be caused by volatilization of the antimony compounds used for spiking, resulting in loss of antimony during the sample digestion process at the reference laboratory. Therefore, Table 7-3 includes a second evaluation of accuracy for antimony, comparing the results from the ElvaX with the ERA-certified values. Unlike most of the other XRF instruments that participated in the demonstration, however, use of these values did not improve the RPDs for antimony. The mean RPD for the antimony data set actually increased significantly from 45.5 percent ("fair") to 137.4 percent ("poor") when the ERA-certified values were used. The ElvaX data displayed a consistent low bias for antimony when compared with the ERA-certified spike concentrations.

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

Table 7-3. Evaluation of Accuracy — Relative Percent Differences versus Reference Laboratory Data for the Xcalibur ElvaX

Matrix	Sample Group	Statistic	Antimony Ref Lab	Antimony ERA Spike	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	9	1	15	7	23	16	5	7	5	24	2	3	3	20
		Median	47.0%	154.0%	58.3%	16.4%	21.2%	62.7%	25.5%	50.5%	107.9%	16.7%	2.3%	11.6%	35.4%	21.6%
	Level 2	Number	5	1	4	7	4	8	13	4	7	5	5	3	3	6
		Median	31.7%	141.6%	15.9%	12.4%	39.9%	5.7%	10.3%	36.2%	90.4%	30.9%	72.7%	7.6%	70.6%	35.2%
	Level 3	Number	3	2	4	2	2	2	13	8	2	6	4	7	4	9
		Median	71.9%	147.7%	20.1%	13.2%	47.9%	62.0%	29.1%	55.5%	115.7%	13.6%	88.3%	16.5%	78.6%	22.2%
	Level 4	Number	--	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	--	30.9%	22.3%	--	--	--	--	--	--
	All Soil	Number	17	4	23	16	29	26	38	24	14	35	11	13	10	35
		Median	43.9%	147.7%	45.0%	13.9%	29.4%	26.6%	24.1%	47.0%	96.2%	17.0%	75.7%	12.4%	67.4%	22.3%
Sediment	Level 1	Number	3	3	17	3	6	8	3	12	3	18	3	4	0	17
		Median	43.8%	114.1%	42.5%	14.0%	20.6%	33.0%	19.2%	68.1%	89.8%	22.1%	20.0%	48.6%	NC	33.9%
	Level 2	Number	4	4	4	4	3	4	19	3	4	6	4	4	3	5
		Median	38.4%	135.6%	29.9%	29.6%	34.6%	4.8%	14.0%	59.9%	79.2%	17.4%	67.7%	30.4%	74.2%	50.8%
	Level 3	Number	3	3	2	3	3	10	4	3	3	4	3	3	3	4
		Median	62.3%	138.7%	49.2%	17.7%	37.8%	27.2%	23.0%	81.2%	88.5%	18.5%	97.4%	68.3%	83.3%	30.6%
	Level 4	Number	--	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	--	30.6%	--	--	--	--	--	--	--
	All Sediment	Number	10	10	23	10	12	22	32	18	10	28	10	11	6	26
		Median	45.6%	135.6%	42.5%	21.7%	32.4%	25.1%	17.6%	66.9%	84.5%	20.0%	69.1%	50.3%	79.5%	37.2%
All Samples	Xcalibur	Number	27	14	46	26	41	48	70	42	24	63	21	24	16	61
	ElvaX	Median	45.5%	137.4%	44.2%	16.3%	30.7%	25.1%	19.5%	54.6%	90.7%	17.5%	75.7%	18.3%	74.0%	32.5%
All Samples	All XRF	Number	206	110	320	209	338	363	558	392	192	403	195	177	218	471
	Instruments	Median	84.3%	70.6%	26.2%	16.7%	26.0%	16.2%	26.0%	21.5%	58.6%	25.4%	16.7%	28.7%	38.3%	19.4%

Notes:

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

--	No samples reported by the reference laboratory in this concentration range.
ERA	Environmental Resource Associates, Inc.
Number	Number of samples appropriate for accuracy evaluation.
Ref Lab	Reference laboratory (Shealy Environmental Services, Inc.)
RPD	Relative percent difference.

ElvaX 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 arsenic, cadmium, 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.

Figure 7-1. Linear correlation plot for ElvaX showing high correlation for nickel.

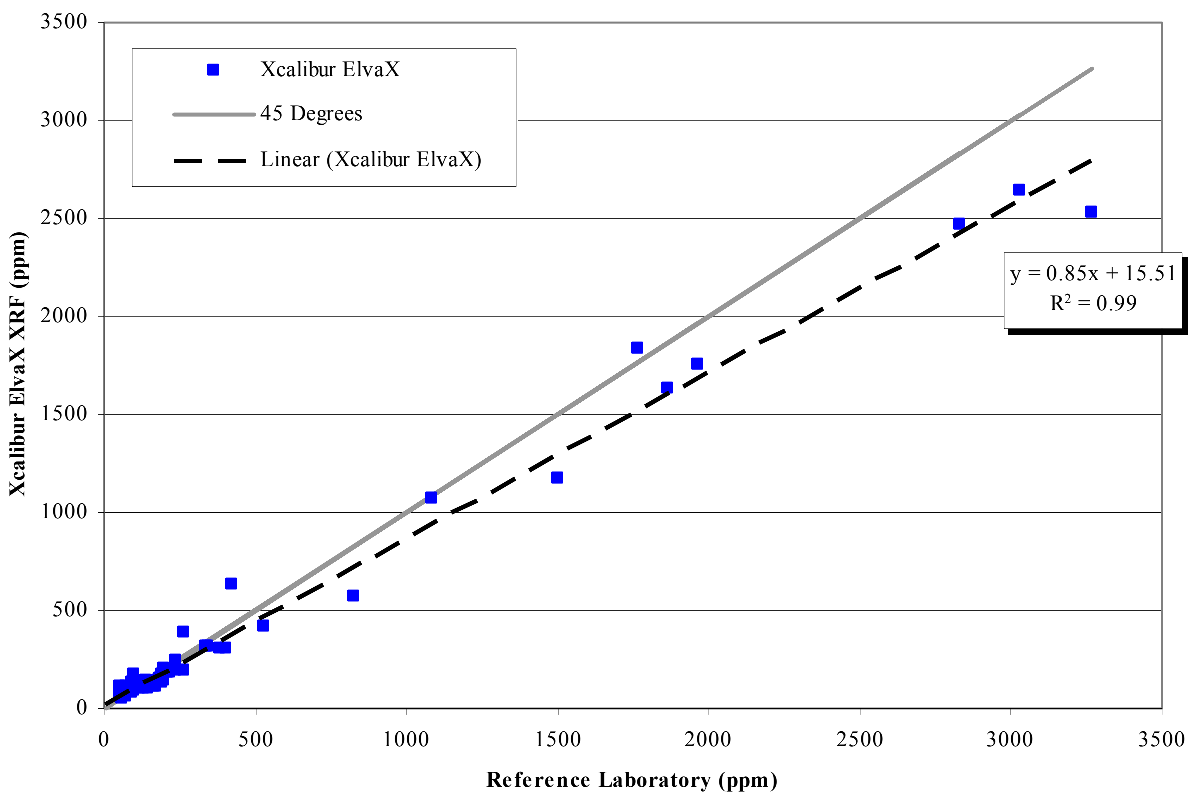


Table 7-4. Summary of Correlation Evaluation for the ElvaX

Target Element	m	b	r^2	Correlation	Bias
Antimony (vs. Reference Lab)	0.44	50.8	0.83	Moderate	Low [1]
Antimony (vs. ERA Certified Value)	0.15	19.4	0.98	High	Low
Arsenic	**1.23**	**35.1**	**0.97**	**High**	**--**
Cadmium	**1.24**	**-24.9**	**0.98**	**High**	**--**
Chromium	0.62	34.9	0.98	High	Low
Copper	1.59	-212.6	0.93	High	High
Iron	0.67	5071.9 [2]	0.93	High	Low
Lead	1.40	-324.1	0.94	High	High
Mercury	0.19	113.6	0.82	Moderate	Low
Nickel	**0.85**	**15.5**	**0.99**	**High**	**--**
Selenium	2.99	-97.0	0.96	High	High
Silver	1.28	30.0	0.63	Moderate	High
Vanadium	0.33	25.8	0.80	Moderate	Low
Zinc	1.03	223.1	0.85	Moderate	--

Notes:

[1] Although the overall bias for antimony was low, a high bias was indicated at low concentrations by the high relative y-intercept. This high bias was noted as part of the MDL evaluation in Section 7.1.

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

-- No bias observed.

b Y-intercept of correlation line.

m Slope of correlation line.

r^2 Correlation coefficient of correlation line.

General observations from the correlation plots are as follows:

- A moderate degree of correlation and a somewhat low overall bias were observed between the data for the ElvaX and the reference laboratory for antimony. Table 7-4 and Figure E-1 show a second correlation analysis for antimony, comparing the mean ElvaX concentrations for spiked blends with the ERA-certified values. Although a slightly better correlation was observed relative to the ERA-certified values (r^2 improved from 0.83 to 0.98), a much lower slope of 0.15 indicates a significant low bias in the ElvaX data when compared with these values. This observation was consistent with the RPD evaluation and showed that, unlike many other XRF instruments in the demonstration, comparisons to ERA-certified concentrations did not improve the apparent accuracy of the ElvaX data for antimony.

- Large y-intercepts were calculated for lead and iron. Examination of the plots for these elements (Figures E-6 and E-7) reveals that these y-intercepts are small relative to the extreme range of concentrations in the demonstration samples. Smaller but significant y-intercepts were also observed for copper and zinc. However, these intercepts were again small enough relative to the concentration range of the demonstration samples to have a minor effect on the overall correlation and bias observed for these metals.

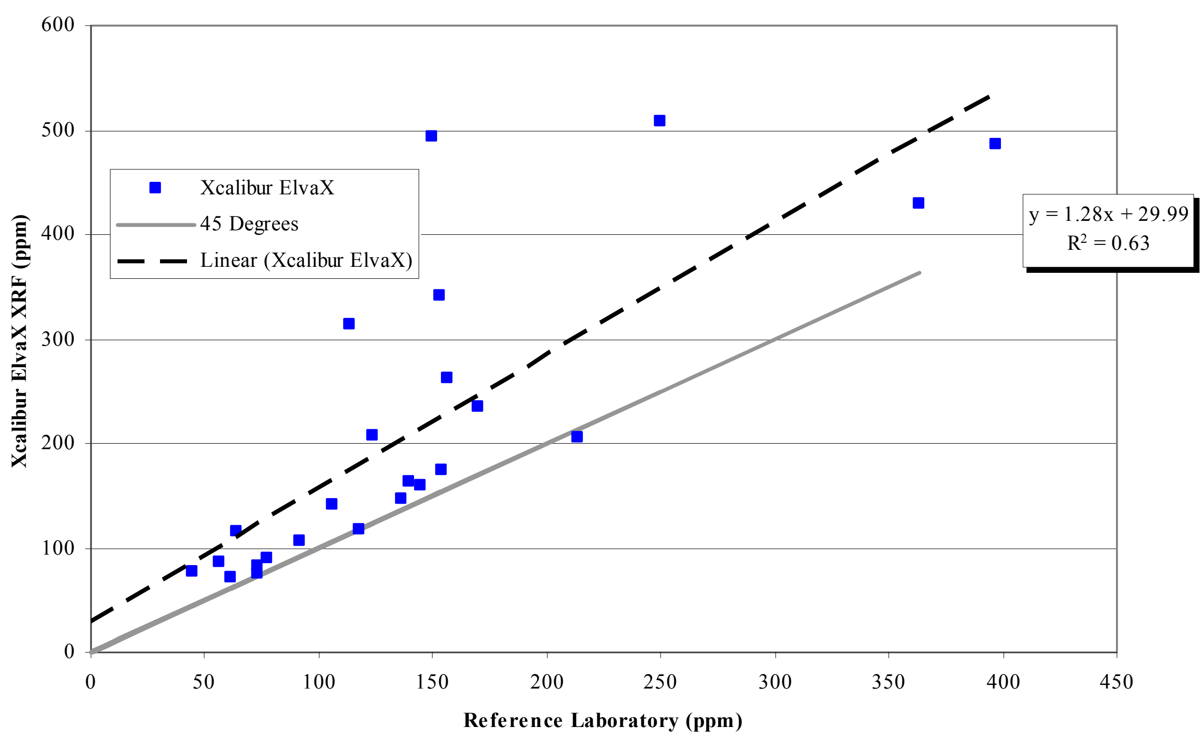
- Mercury exhibited a moderate r^2 value (0.82) and a low bias (m = 0.19). Removing two extreme Level 4 concentrations (Blends 21 and 22) from the plots produced a poorer correlation coefficient (in the range of 0.64) without improving the low bias. Similar instrument performance (moderate correlation with a significant low bias) was also observed for vanadium. According to the developer, interference from titanium (which was not characterized in the demonstration and therefore could not be adjusted for) affected instrument accuracy for vanadium (Appendix B).

- Significant high biases were observed for selenium, lead, and copper. Whereas the bias appeared to be generalized for selenium (Figure E-10), the bias observed for lead and copper appeared to be influenced by the extremely high concentrations of a few sample blends. These blends were generally associated with the Wickes Smelter site (Blends 7 through 9 and 51), along with one blend from the Alton Steel site (Blend 53). Although no bias was observed for zinc, removal of another high-concentration outlier from the Alton Steel site (Blend 17) improved the correlation coefficient for zinc from 0.85 to 0.97.

- The lowest degree of correlation ($r^2 = 0.63$) was observed for silver, and appeared to be caused by broad variability in the data rather than a few outliers. Figure 7-2 shows the correlation plot for silver.

In conclusion, the evaluations of accuracy showed an acceptable overall level of performance by the ElvaX for the target elements. Correlations with the reference laboratory were generally high and, for most target elements, the median RPDs for the ElvaX were equivalent or better than the overall median of all eight XRF instruments that participated in the demonstration. However, the ElvaX showed significant biases for some elements, and accuracy tended to decrease in high concentration samples and complex matrixes. In addition, ElvaX results for antimony did not agree with the certified spike concentrations in the spiked sample blends, showing a low bias. Although some pre-demonstration samples were used in the initial calibration process on the first day of the demonstration, it is possible that the ElvaX

Figure 7-2. Linear correlation plot for ElvaX showing high data variability for silver.



analysis procedures and quantitation algorithms (which were developed for broad-based and not specifically for environmental applications) may have limited instrument accuracy. Development of a detailed standard operating procedure (SOP) and an instrument set-up that are more targeted to environmental soil and sediment samples might improve the comparability of the ElvaX data with that of the reference laboratory.

7.3 Primary Objective 3 — Precision

As described in Section 4.2.3, the precision of the ElvaX was evaluated by calculating RSDs for the replicate measurements from each sample blend. Median RSDs for the various concentration levels and media (soil and sediment), as well as for the demonstration sample set as a whole, are presented in Table 7-5. An expanded set of summary statistics for the RSDs (including minimum, maximum, and mean) is provided in Appendix E (Table E-2).

The median RSDs calculated for the target elements ranged as high as 24.5 percent (vanadium). The ranges of median RSDs are further summarized below:

- Very low (median RSD between 0 and 5 percent): copper and iron.
- Low (median RSD between 5 and 10 percent): antimony, cadmium, lead, nickel, selenium, silver, and zinc.
- Moderate (median RSD between 10 and 20 percent): arsenic, chromium, and mercury.
- High (median RSD greater than 20 percent): vanadium.

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

Further review of the median RSDs in Table 7-5 based on concentration range reveals higher RSDs (in other words, lower precision) for the target elements in Level 1 samples when compared with the rest of the data set. This effect was observed for most of the target elements in both soil and sediment, with large relative effects for cadmium, chromium, mercury, selenium, and vanadium. This observation indicates that analytical precision for the ElvaX may depend on concentration.

As an additional comparison, Table 7-5 presents the overall average of the median RSDs for all eight XRF instruments that participated in the demonstration. Complete summary statistics for the RSDs across all eight XRF instruments are included in Table E-2. Table 7-5 indicates that the median RSDs for the ElvaX were equivalent to or above the all-instrument medians, indicating equivalent or lower precision, for 12 of the 13 target elements. Slightly lower median RSDs for the ElvaX than the all-instrument medians were observed for antimony.

Table 7-6 presents median RSD statistics for the reference laboratory and compares these with the summary data for the ElvaX. (Complete summary statistics are provided in Table E-3 of Appendix E.) For seven of the 13 target elements, Table 7-6 indicates that the median RSDs for the ElvaX were equivalent to or lower than the RSDs for the reference laboratory. The ElvaX exhibited higher RSDs than the reference laboratory for arsenic, chromium, mercury, selenium, silver, and vanadium. Thus, the ElvaX exhibited slightly better precision overall than the reference laboratory. In comparison to the ElvaX, the median RSDs for all XRF instruments were equivalent to or lower than for the reference laboratory for 11 of 13 target elements.

Table 7-5. Evaluation of Precision — Relative Standard Deviations for the Xcalibur ElvaX

Matrix	Sample Group	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	9	15	7	23	16	5	7	5	24	2	3	3	20
		Median	6.7%	16.1%	20.5%	20.9%	6.2%	4.4%	7.0%	37.7%	10.3%	27.0%	11.5%	49.4%	8.1%
	Level 2	Number	5	4	7	4	8	13	4	7	5	5	3	3	6
		Median	3.0%	7.7%	2.2%	5.5%	3.8%	2.7%	4.2%	12.6%	6.3%	5.2%	7.3%	23.3%	2.3%
	Level 3	Number	3	4	2	2	2	13	8	2	6	4	7	4	9
		Median	3.6%	16.0%	4.5%	2.4%	12.8%	3.8%	2.6%	6.3%	2.6%	8.3%	19.2%	28.9%	1.8%
	Level 4	Number	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	4.5%	10.4%	--	--	--	--	--	--
	All Soil	Number	17	23	16	29	26	38	24	14	35	11	13	10	35
		Median	6.5%	14.8%	6.3%	17.7%	6.0%	4.1%	4.6%	15.7%	7.9%	8.5%	11.5%	39.9%	6.4%
Sediment	Level 1	Number	3	17	3	6	8	3	12	3	18	3	4	0	17
		Median	5.1%	18.5%	8.1%	30.0%	6.1%	4.9%	12.5%	42.8%	9.9%	17.3%	9.4%	NC	8.3%
	Level 2	Number	4	4	4	3	4	19	3	4	6	4	4	3	5
		Median	6.3%	4.6%	5.5%	5.4%	2.3%	2.2%	3.2%	5.9%	5.9%	9.6%	5.6%	25.7%	3.3%
	Level 3	Number	3	2	3	3	10	4	3	3	4	3	3	3	4
		Median	3.4%	2.2%	3.2%	5.0%	4.3%	2.4%	2.7%	5.2%	1.8%	3.6%	3.2%	5.6%	1.8%
	Level 4	Number	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	3.2%	--	--	--	--	--	--	--
	All Sediment	Number	10	23	10	12	22	32	18	10	28	10	11	6	26
		Median	5.2%	14.1%	6.5%	12.5%	4.3%	2.5%	6.4%	6.5%	8.2%	9.6%	7.0%	12.1%	5.8%
All Samples	Xcalibur ElvaX	Number	27	46	26	41	48	70	42	24	63	21	24	16	61
		Median	5.4%	14.1%	6.5%	17.2%	4.9%	3.2%	6.0%	12.5%	8.0%	8.9%	9.4%	24.5%	6.1%
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 ElvaX and All Demonstration Instruments

Matrix	Sample Group	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Ref. Lab	Number	17	23	15	34	26	38	33	16	35	13	13	21	35
		Median	9.8%	12.4%	9.0%	10.6%	9.1%	8.7%	13.2%	6.6%	10.0%	7.1%	7.5%	6.6%	9.1%
Sediment	Ref. Lab	Number	7	24	10	26	21	31	22	10	27	12	10	17	27
		Median	9.1%	9.2%	8.2%	7.5%	8.9%	8.1%	7.4%	6.9%	7.3%	7.6%	6.6%	8.1%	6.9%
All Samples	Ref. Lab	Number	24	47	25	60	47	69	55	26	62	25	23	38	62
		Median	9.5%	9.5%	9.0%	8.4%	8.9%	8.5%	8.6%	6.6%	8.2%	7.4%	7.1%	7.2%	7.4%
All Samples	Xcalibur ElvaX	Number	27	46	26	41	48	70	42	24	63	21	24	16	61
		Median	5.4%	14.1%	6.5%	17.2%	4.9%	3.2%	6.0%	12.5%	8.0%	8.9%	9.4%	24.5%	6.1%
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%

7.4 Primary Objective 4 — Impact of Chemical and Spectral Interferences

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

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

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

Interferent-to-element ratios were calculated using the mean concentrations the reference laboratory reported for each blend, classified as low (less than 5X), moderate (5 to 10X), or high (greater than 10X). Table 7-7 presents median RPD data for arsenic, nickel, copper, and zinc that are grouped based on this classification scheme. Complete summary statistics are presented in Appendix E (Table E-4). The tables indicate significant interference effects of zinc on copper. Specifically, as zinc concentrations increased to greater than 10 times the copper concentration, the median RPD for copper increased from 21 percent (within the "good" range defined in Section 7.2) to 99 percent (in the "poor" range). Slight effects were also observed for copper as an interferent for nickel; as copper concentrations increased to greater than 10 times the nickel concentration, the median RPD for nickel increased from 17 percent ("good") to 28 percent ("fair"). Evaluation of the effects of lead on arsenic, nickel on copper, and copper on zinc did not appear to show any consistent trends.

In presenting statistics for the raw RPDs as well as the absolute values of the RPDs, Table E-4 further shows that the interferences from zinc appeared to produce an increasingly high bias in the copper data (as indicated by more negative raw RPDs). A similar trend was observed in the effect of copper on nickel.

7.5 Primary Objective 5 — Effects of Soil Characteristics

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

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.

Review of Table 7-8 indicates that the median RPDs were highly variable and that trends or differences between sample sites were difficult to discern. Evaluations relative to sampling site were further complicated by the low numbers of samples for many target elements. (Table 7-8 indicates that only one to three samples were available from many sampling sites for evaluation of specific target elements.) Extreme RPDs were observed in Alton Steel blends for copper and zinc, the Leviathan Mine blends for lead, Torch Lake blends for nickel, and Sulphur Bank blends for copper and silver. Other individual extreme values noted previously for copper and lead were associated with the Wickes Smelter site (Section 7.2).

Table 7-7. Effects of Interferent Elements on the RPDs (Accuracy) for Other Target Elements for the Xcalibur ElvaX[1]

Parameter	Lead Effects on Arsenic			Copper Effects on Nickel			Nickel Effects on Copper			Zinc Effects on Copper			Copper Effects on Zinc		
Interferent/ Element Ratio	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10	<5	5 – 10	>10
Number of Samples	29	7	10	44	5	14	39	1	8	35	2	11	49	3	9
Median RPD of Target Element [2]	45.1%	21.5%	50.6%	17.2%	15.7%	27.6%	21.0%	70.7%	49.4%	21.0%	8.0%	99.1%	32.5%	47.1%	31.3%
Median Interferent Concentration	152	8802	4699	73	1143	2298	128	307	1793	177	5169	3528	177	1085	3670
Median Target Element Concentration	178	1129	70	146	131	122	852	37	122	909	775	332	1085	99	103

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, Xcalibur ElvaX

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	3
			Median	--	82.6%	13.1%	8.8%	83.7%	19.7%	48.1%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	7	5	5	6	7	6
			Median	12.6%	33.7%	10.1%	43.2%	12.1%	10.3%	43.8%
Soil	CN	Sandy loam (burn pit residue)	Number	2	1	2	1	3	3	2
			Median	68.9%	66.2%	11.7%	21.2%	21.0%	2.8%	51.7%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	1	--	--	4	2	6	6
			Median	48.2%	--	--	7.5%	19.2%	15.1%	5.3%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	4	11	5	4	4	12	4
			Median	45.5%	53.8%	24.9%	41.6%	28.9%	28.4%	84.2%
Sediment	RF	Silty fine sand (tailings)	Number	4	12	5	8	13	13	10
			Median	59.0%	41.8%	21.0%	32.4%	11.6%	16.2%	59.0%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	6	5	1	10	4	12	--
			Median	32.9%	48.6%	16.3%	22.9%	76.1%	33.7%	--
Sediment	TL	Silt and clay (slag-enriched)	Number	3	2	2	1	7	7	4
			Median	45.5%	36.2%	24.1%	15.6%	28.6%	15.7%	96.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	7	3	6	6	7	7
			Median	47.0%	22.2%	14.7%	38.7%	9.5%	23.8%	70.5%
	All		Number	27	46	26	41	48	70	42
			Median	45.5%	44.2%	16.3%	30.7%	25.1%	19.5%	54.6%

Table 7-8. Effect of Soil Type on RPDs (Accuracy) of Target Elements, Xcalibur ElvaX (Continued)

Matrix	Site	Matrix Description	Statistic	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	AS	Fine to medium sand (steel processing)	Number	--	3	1	1	--	3
			Median	--	25.8%	68.5%	16.5%	--	65.0%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	1	6	2	4	2	7
			Median	26.6%	20.1%	63.4%	5.1%	103.4%	33.4%
Soil	CN	Sandy loam (burn pit residue)	Number	2	3	2	2	1	3
			Median	99.2%	27.6%	36.8%	13.5%	70.6%	52.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	3	--	--	--	2
			Median	--	9.8%	--	--	--	52.2%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	3	11	5	4	5	8
			Median	88.5%	14.3%	84.1%	22.4%	64.2%	34.2%
Sediment	RF	Silty fine sand (tailings)	Number	5	13	3	4	3	13
			Median	89.8%	19.9%	91.7%	52.6%	87.0%	38.4%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	10	11	3	1	3	11
			Median	96.2%	17.5%	86.3%	75.8%	35.4%	7.8%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	6	4	4	--	7
			Median	79.1%	36.4%	51.1%	46.8%	--	31.3%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	7	1	4	2	7
			Median	--	16.3%	106.0%	14.3%	66.9%	24.9%
	All		Number	24	63	21	24	16	61
			Median	90.7%	17.5%	75.7%	18.3%	74.0%	32.5%

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 (absolute value).

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 distributed among six of the nine sampling sites. This evaluation verified the slight prevalence of outliers in the Torch Lake and Sulphur Bank Mine blends. 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 precluded the identification of high statistical outliers or extreme values for antimony, arsenic, cadmium, copper, mercury, selenium, and silver.

7.6 Primary Objective 6 — Sample Throughput

The Elvatech/Xcalibur two-person field team was able to analyze all 326 demonstration samples in 5 days at the demonstration site. Once the ElvaX instrument had been set up and operations had been streamlined, the Elvatech/Xcalibur field team was able to analyze a maximum of 83 samples during an extended work day. This sample throughput was achieved by using the different members of the field team to separately perform the sample preparation and instrumental analysis activities. Without an extended work day, it was estimated that the Elvatech/Xcalibur field team could have only processed 49 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 long run time in the XRF spectrometer (8 minutes per sample initially). The instrument run time was shortened during the field demonstration to allow sample processing to be completed during the designated week and based on the belief that a reduction in run time could still provide data of sufficient accuracy and precision. If this shorter instrument run time had been implemented at the start of the field demonstration, then the estimated sample throughput for a normal 8-hour work day would have been similar to the average of the other XRF instruments.

A detailed discussion of the time required to complete the various steps of sample analysis using the ElvaX is included as part of the labor cost analysis in Section 8.3.

7.7 Primary Objective 7 — Technology Costs

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

7.8 Secondary Objective 1 — Training Requirements

Technology users must be suitably trained to set up and operate the instrument to obtain the level of data quality required for specific projects. The amount of training required depends on the configuration and complexity of the instrument, along with the associated software. Xcalibur offers on-site training and telephone support to instrument users on an informal, as needed basis. During the demonstration, it was apparent to the observer that the instrument can be operated by a single trained operator. A degreed chemist is advisable but not required to operate the instrument. A second staff member can be added to provide support with sample preparation activities. This second staff member does not require any technical training or expertise.

The instrument software includes on-screen prompts to assist the user with instrument setup, operation, and shut-down. The operation manual provided for the demonstration was abbreviated and general, including only a brief presentation of the procedures for instrument set-up, operation, and shut-down. The manual did not include any discussion of sample preparation techniques, quality control requirements, or specific procedures for the analysis of environmental samples.

7.9 Secondary Objective 2 — Health and Safety

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

The ElvaX appears to be a safe instrument. It uses an x-ray tube that includes two levels of lead shielding between the tube and the operator. In certain modes, compressed helium gas may be used. Proper procedures for the safe handling and use of compressed gas cylinders include the use of hand-trucks for transport, belts for securing the cylinder to a wall or large object for stability, and the use of an appropriate gas regulator. Risks from exposure to radiation, electricity, or reagents are minimal when the manufacturer's recommended operational guidelines are followed.

7.10 Secondary Objective 3 — Portability

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

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

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

Based on its dimensions and power requirements, the ElvaX is defined as transportable. It is a bench-top unit that can be set on a table or bench in an office or mobile laboratory, or on the back of a truck bed, for field analysis. It is not capable of providing in situ analysis of soil. The instrument is fairly light and compact for a bench-top unit, weighing about 18 kilograms with dimensions of less than 1.5 feet on a side. The only major component other than the XRF instrument itself is the personal computer that houses the operating and data processing systems. Each component requires 110 volts of electricity for operation; no portable battery systems are available at this time.

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 was also evaluated. Weather resistance was evaluated by examining the instrument for exposed electrical connections and openings that may allow water to penetrate (for portable instruments only).

The ElvaX system is constructed from impact-resistant coated metal and molded plastic. However, the instrument is not weather resistant. It must operate in a stable environment; both physical (like a bench-top or firm table) and environmental (free from excessive temperature or moisture extremes).

Purchased models include a 1-year parts and labor warranty. The x-ray tube has a lifespan of about 2 to 5 years of normal usage. Upgrades in software are available and are provided at no extra to instrument owners.

7.12 Secondary Objective 5 — Availability

The ElvaX is manufactured by Elvatech, Ltd., in Kiev, Ukraine. Xcalibur XRF Services, Inc., distributes and services the ElvaX from locations on the East and West Coasts of the U.S. and in the Great Lakes region. Xcalibur has a staff of five U.S.-based service technicians and delivers instruments internationally. The company also has a free service line available to access qualified technicians 365 days per year with or without a service contract. The developer indicates that the call-back time on the free service line is less than 20 minutes.

Chapter 8
Economic Analysis

This chapter provides cost information for the ElvaX 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 ElvaX.

8.1 Equipment Costs

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

The base price of the ElvaX instrument, as used at the demonstration, is $35,000. A video camera and helium purge unit can be added for $10,000. An autosampler can also be added to the system for $20,000. Observations during the field demonstration indicated that inclusion of the autosampler accessory could have reduced labor requirements and allowed all sample processing tasks to be performed by one person instead of a two-person team.

Purchased models include a 1-year warranty. The lifespan of the x-ray tube is about 2 to 5 years for normal usage. Xcalibur indicated that the ElvaX is not available for rental; however, leasing programs are available. For evaluation purposes later in this chapter an estimated rental cost was derived based on similar XRF technologies where both purchase and rental prices were available.

The purchase price, rental cost, and shipping cost for the ElvaX compare favorably 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	ElvaX	XRF Demonstration Average [1]
Shipping	$400	$410
Capital Cost (Purchase) [2]	$35,000	$54,300
Weekly Rental	$1,600	$2,813
Autosampler (for Overnight Analysis)	Not Included	N/A

Notes:
[1] Average for all eight instruments in the demonstration
[2] Does not include the ElvaX autosampler accessory (recommended).
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 could include helium gas for the helium purge unit.

The ElvaX was operated for 5 days to complete the analysis of all 326 samples during the field demonstration. The supplies required to process samples were similar for all XRF instruments that participated in the demonstration and were estimated to cost about $245 for 326 samples or $0.75 per sample.

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 required to complete each of these activities using the Elva-X 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 each activity using the ElvaX is compared with the average of all XRF instruments in Table 8-2 and with the range of all XRF instruments in Figure 8-1. Specifically, the ElvaX exhibited lower-than-average times for sample preparation and end of project packing, and exhibited higher-than-average times for initial setup and calibration, sample analysis, daily shutdown and startup and total time per sample.

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

Activity	ElvaX	Average [2]
Initial Setup and Calibration	90	54
Sample Preparation	2.2	3.1
Sample Analysis/Data Reduction	9.5	6.7
Daily Shutdown/Startup	30	10
End of Project Packing	30	43
Total Processing Time per Sample	12.2	10.0

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

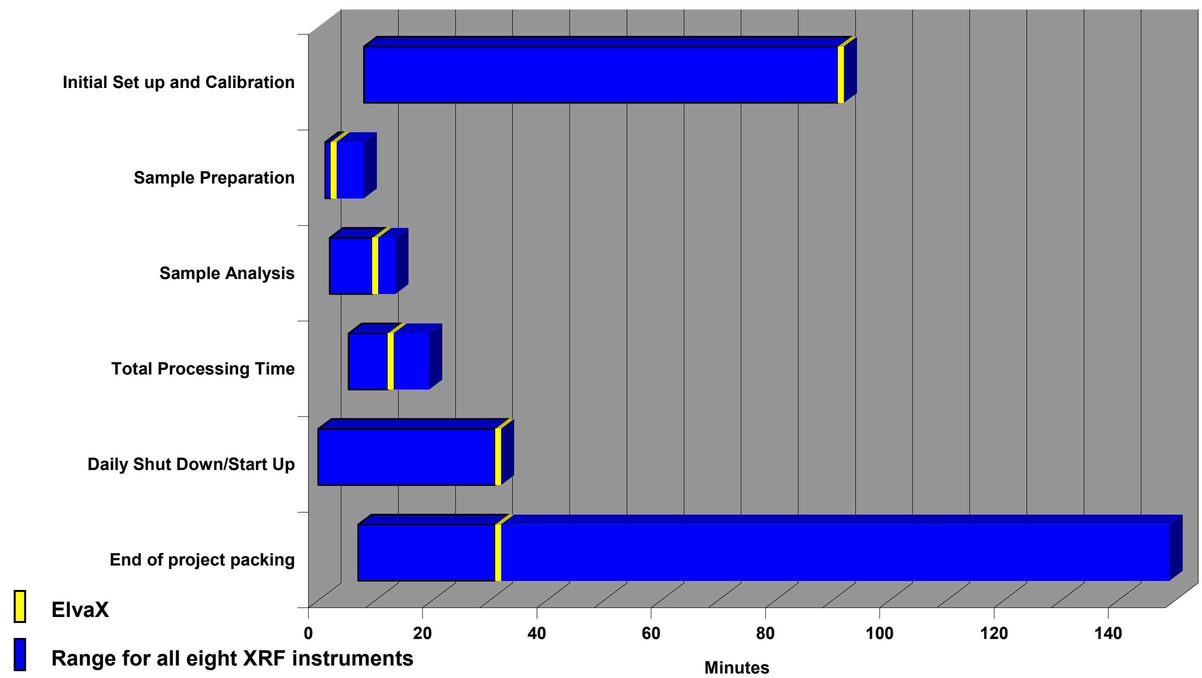


Figure 8.1. Comparison of activity times for the ElvaX versus other XRF instruments.

The Elvatech/Xcalibur field team expended about 81 labor hours to complete all sample processing activities during the field demonstration using the ElvaX. This was slightly higher than the overall average of 69 hours for all instruments that participated in the demonstration. The primary reasons that labor hours were slightly higher for the ElvaX include:

- The instrument run time was initially set at about 8 minutes, which was substantially longer than most other instruments. The Elvatech/Xcalibur field team reduced the instrument run time later during the demonstration based on the belief that the longer run time was not needed to obtain good precision and accuracy.

- The autosampler accessory was not included with the instrument for the demonstration and would have reduced the time spent processing samples through the instrument.

8.4 Comparison of XRF Analysis and Reference Laboratory Costs

Two scenarios were evaluated to compare the cost for XRF analysis using the ElvaX 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 ElvaX was based on equipment rental for 1 week, along with labor and supplies estimates established during the field demonstration. As noted previously, the estimate used a hypothetical rental rate for the ElvaX based on a survey of rental and purchase costs of similar instruments. This hypothetical rate was used because Xcalibur does not offer a rental program. 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 ElvaX analysis cost was not adjusted for one element versus 13 elements.

Table 8-3 summarizes the costs for the ElvaX versus the cost for analysis in a fixed laboratory. This comparison shows that the ElvaX compares favorably to a fixed laboratory in terms of overall cost when a large number of elements are to be determined. The ElvaX compares unfavorably to a fixed laboratory when one element is to be determined. Use of the ElvaX 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 ElvaX in the example scenario (326 samples) was estimated at $8,436 whether one or a number of elements was analyzed. This estimate compares favorably with the average of $8,932 for all XRF instruments that participated in the demonstration. The ElvaX cost for the example scenario compares very favorably with other bench-top XRF instruments.

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

Analytical Approach	Quantity	Item	Unit Rate	Total
ElvaX (1 to 13 elements)				
Shipping	1	Roundtrip	$400	$400
Weekly Rental (estimated)[1]	1	Week	$1,800	$1,800
Supplies	326	Sample	$0.75	$245
Labor	135	Hours	$43.75	$5,901
IDW	N/A	N/A	N/A	$90
Total ElvaX Analysis Cost (1 to 13 elements)				**$8,436**
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] Extimated values as Xcalibur currently does not have a rental rate for the ElvaX.

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 ElvaX XRF analyzer. The evaluation design incorporated 13 target elements, 70 distinct sample blends, and a total of 326 samples. The blends included both soil and sediment samples from nine sampling locations. A rigorous program of sample preparation and characterization, reference laboratory analysis, QA/QC oversight, and data reduction supported the evaluation of XRF instrument performance.

One important aspect of the demonstration was the sample blending and processing procedures (including drying, sieving, grinding, and homogenization) that significantly reduced uncertainties associated with the demonstration sample set. These procedures minimized the impacts of heterogeneity on method precision and on the comparability between XRF data and reference laboratory data. In like manner, project teams are encouraged to assess the effects of sampling uncertainty on data quality and to adopt appropriate sample preparation protocols before XRF is used for large-scale data collection, particularly if the project will involve comparisons to other methods (such as off-site laboratories). An initial pilot-scale method evaluation, carried out in cooperation with an instrument vendor, can yield site-specific SOPs 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 ElvaX for each primary and secondary objective of the technology demonstration are summarized in Tables 9-1 and 9-2. The ElvaX and the average performance of all eight instruments that participated in the XRF technology demonstration are compared in Figure 9-1. The comparison in Figure 9-1 indicates that, when compared with the mean performance of all eight XRF instruments, the ElvaX showed:

- Better MDLs for four elements, including antimony, cadmium, chromium, and nickel (iron was not included in the MDL evaluation).
- Better accuracy (lower RPDs) for four target elements, including antimony, iron, nickel, and silver. However, when RPDs for antimony are calculated versus sample spike levels rather than reference laboratory data (which may be biased low), accuracy for antimony is lower than for the program as whole.
- Better precision (lower RSDs) for one target element (antimony).

As a transportable bench-top instrument that requires AC power, the ElvaX must be operated in a mobile laboratory or other stable environment, and cannot be used for in situ soil analysis. Although good overall performance was observed for this instrument, the developer may want to consider whether instrument accuracy and sensitivity could be further improved for environmental applications through refined calibration protocols, quantitation algorithms, or other method modifications that could be documented in a written procedure for soil and sediment analysis.

Table 9-1. Summary of Xcalibur ElvaX Performance – Primary Objectives

Objective	Performance Summary

P1: Method Detection Limits	• Low numbers of detections in the MDL blends produced limited data and therefore, uncertainty in the MDL calculations for cadmium, mercury, selenium, and vanadium.
	• Mean MDLs for the target elements ranged as follows:
	  o MDLs of 1 to 20 ppm: antimony and mercury.
	  o MDLs of 20 to 50 ppm: arsenic, copper, nickel, silver, vanadium, and zinc.
	  o MDLs of 50 to 100 ppm: cadmium, chromium, and lead.
	  o MDLs of greater than 100 ppm: selenium. (This MDL was 156 ppm, and was based on only a single sample blend.)
	• The MDLs calculated for the ElvaX were generally lower than reference MDL data from EPA Method 6200 (higher MDLs were observed only for lead).

P2: Accuracy and Comparability	• Median RPDs relative to reference laboratory data revealed the following, with lower RPDs indicating greater accuracy:
	  o RPDs less than 10 percent: none.
	  o RPDs of 10 to 25 percent: cadmium, iron, nickel, and silver.
	  o RPDs of 25 to 50 percent: antimony, arsenic, chromium, copper, and zinc.
	  o RPDs of greater than 50 percent: lead, mercury, selenium, and vanadium.
	• 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 moderate when the ElvaX data were compared with the reference laboratory data (with a median RPD of 45.5 percent) but increased considerably when compared with certified spike values (where the median RPD was 137.4 percent). Thus, comparison to the spike concentrations did not improve the apparent accuracy of the ElvaX for antimony.
	• Correlation plots relative to reference laboratory data indicated:
	  o High correlation coefficients (greater than 0.9) for eight of the 13 target elements.
	  o Moderate correlation coefficients (between 0.5 and 0.9) for the remaining five elements (antimony, mercury, selenium, vanadium, and zinc).
	  o High biases in the XRF data versus the reference laboratory data for copper, lead, selenium, and silver. Low biases were observed for antimony, chromium, iron, mercury, and vanadium.

P3: Precision	• Median RSDs for sample replicates were as follows, with lower RSDs indicating greater precision:
	  o RSDs below 5 percent: copper and iron.
	  o RSDs between 5 and 10 percent: antimony, cadmium, lead, nickel, selenium, silver, and zinc.
	  o RSDs between 10 and 20 percent: arsenic, chromium, and mercury.
	  o RSDs greater than 20 percent: vanadium.
	• RSDs were slightly higher (that is, precision was lower) in the lowest concentration sample blends for many of the target elements, indicating a slight concentration dependence for precision.
	• For seven of the 13 target elements, median RSDs for the ElvaX were equivalent to or lower than the RSDs calculated for the reference laboratory data, indicating similar to slightly better precision for the ElvaX.

Table 9-1. Summary of Xcalibur ElvaX Performance – Primary Objectives (continued)

Objective	Performance Summary
P4: Effects of Sample Interferences	• High relative concentrations (greater than 10X) of zinc as an interfering element reduced accuracy for copper from "good" (median RPDs less than 25 percent) to "poor" (median RPDs greater 50 percent). Further, the high concentrations of zinc produced an increasingly high bias in copper results. • A slight interference effect (decreasing accuracy from good to fair, and an increasing negative bias) were observed for nickel in samples containing high concentrations of copper as an interferent. • Evaluation of high concentrations of lead on arsenic, nickel on copper, and copper on zinc did not appear to show significant interference trends.
P5: Effects of Soil Type	• A slight prevalence of outliers was noted for some of the target elements in the Leviathan Mine, Torch Lake, Sulphur Bank Mine, Alton Steel, and Wickes Smelter sample blends. However, sample matrix had a minor effect on the overall accuracy of the ElvaX data given that the ranges of RPDs observed for the target elements were very broad.
P6: Sample Throughput	• With an average sample preparation time of 2.2 minutes and an instrument analysis time of 9.5 minutes per sample, the total sample processing time was 12.2 minutes per sample. • A maximum sample throughput of 83 samples per day was achieved during the demonstration during an extended work day. A more typical sample throughput was estimated to be 49 samples per day for an 8-hour work day. • Use of the optional autosampler may increase sample throughput. However, throughput would have decreased significantly without a second staff member to perform sample preparation and final data processing, which was labor-intensive.
P7: Costs	• Base purchase cost is about $35,000 for the instrument as equipped in the demonstration. Accessories not used in the demonstration include a video camera and helium purge unit ($10,000), as well as an autosampler ($20,000). The base purchase cost includes training and technical support. • The Elvatech/Xcalibur field team expended approximately 81 labor hours to complete the processing of the demonstration sample set (326 samples). In comparison, the average for all participating XRF instruments was 69 man-hours. • By approximating a 1-week rental cost (based on similar bench-top instruments) and adding labor and miscellaneous costs for shipping and supplies, a total project cost of $8,436 was estimated for a project the size of the demonstration. In comparison, the project cost averaged $8,932 for all participating XRF instruments and the cost for fixed-laboratory analysis of all samples for 13 elements was $63,896.

Table 9-2. Summary of Xcalibur ElvaX Performance – Secondary Objectives

Objective	Performance Summary
S1: Training Requirements	• A degreed chemist is recommended but not required to operate the ElvaX. A second technician without specific training is also recommended to assist with sample preparation and data processing activities to improve sample throughput. • Xcalibur offers unlimited product support throughout the lifetime of the instrument, including telephone support and training as needed. • The instrument software includes on-screen prompts to assist the user with instrument setup, operation, and shut-down. However, the operation manual provided for the demonstration was abbreviated and general, and no specific procedures for the preparation or analysis of environmental samples was provided.
S2: Health and Safety	• The ElvaX's x-ray tube is totally encased in two layers of lead shielding, and emits no detectable radiation outside of the instrument cabinet. • Purging of the sample chamber with helium requires the safe management of pressurized gas cylinders.
S3: Portability	• Based on dimensions and weight, the ElvaX is a transportable instrument, designed to be used on a table top or possibly a truck bed. • The instrument and its laptop computer operating system require 110 volt AC power.
S4: Durability	• The ElvaX's x-ray tube has a lifespan of about 2 to 5 years of normal usage. • The instrument is fully warranted for 1 year, and software is upgradeable at no cost. • The instrument must be operated in a stable environment, free from excessive temperature and moisture extremes.
S5: Availability	• The ElvaX is manufactured by Elvatech, Ltd., in Kiev, Ukraine. Xcalibur XRF Services, Inc., distributes and services the ElvaX from locations on the east and west coasts of the U.S. and in the Great Lakes region. • The ElvaX is available for purchase and long-term leasing; no short-term rental options are currently available.

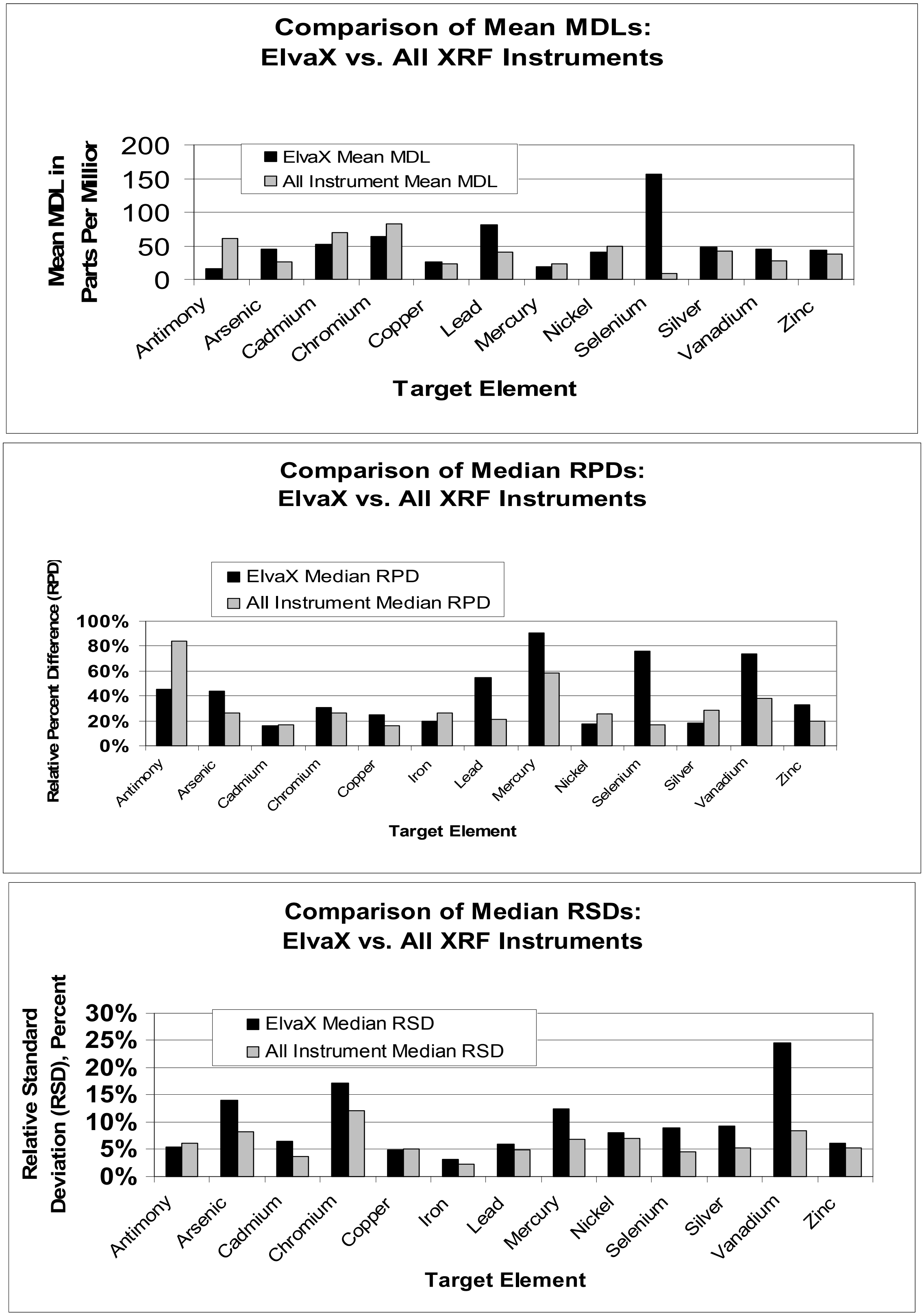


Figure 9-1. Method detection limits (sensitivity), accuracy, and precision of the ElvaX in comparison to the average of all eight XRF instruments.

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Chapter 10
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APPENDIX A

VERIFICATION STATEMENT

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, DC 20460

SITE Monitoring and Measurement Technology Program Verification Statement

TECHNOLOGY TYPE:	X-ray Fluorescence (XRF) Analyzer
APPLICATION:	MEASUREMENT OF TRACE ELEMENTS IN SOIL AND SEDIMENT
TECHNOLOGY NAME:	ElvaX XRF Analyzer
COMPANY:	Xcalibur XRF Services
ADDRESS:	1340 Lincoln Avenue, Unit #6
	Holbrook, NY 11749
Telephone:	(631) 435-9749
Fax:	(516) 885-7398
Email:	ronupa@aol.com
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VERIFICATION PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Superfund Innovative Technology Evaluation (SITE) Monitoring and Measurement Technology (MMT) Program to facilitate deployment of innovative technologies through performance verification and information dissemination. The goal of this program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The program assists and informs those involved in designing, distributing, permitting, and purchasing environmental technologies. This document summarizes the results of a demonstration of the Xcalibur ElvaX 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 instruments to measure trace elements in soil and sediment was conducted from January 24 through 28, 2005, at the Kennedy Athletic, Recreational and Social (KARS) Park, which is part of the Kennedy Space Center on Merritt Island, Florida. A total of 326 samples were analyzed by each XRF instrument, including the ElvaX, 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 Xcalibur ElvaX XRF analyzer. More detailed discussion can be found in the *Innovative Technology Verification Report – XRF Technologies for Measuring Trace Elements in Soil and Sediment: Xcalibur ElvaX XRF Analyzer* (EPA/540/R-06/006).

TECHNOLOGY DESCRIPTION

XRF spectroscopy is an analytical technique that exposes a solid sample. 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 ElvaX is a transportable energy dispersive XRF analyzer capable of detecting elements from sodium (atomic number [Z] = 11) through plutonium (Z = 94) that has applications in the jewelry, metallurgy, customs, forensics, medical diagnostics, food testing, and environmental testing markets. The ElvaX can be used for qualitative or quantitative analysis of environmental samples, metal alloys, liquid food, biological samples, and powder assays, as well as samples deposited on surfaces or filters.

The ElvaX contains a 5 watt x-ray tube with an adjustable power supply (5 to 40 kV) and a choice of three different anode materials (tungsten, titanium, or rhodium). The detector is a Peltier-cooled, solid-state silicon-PiN diode with 180 eV resolution. The instrument operating system runs on a laptop computer using Windows®-based software that has a broad range of data analysis capabilities. The ElvaX analyzer can be calibrated using a standardless fundamental parameters calibration or by using an empirical calibration with known standards or site samples. For this demonstration, the ElvaX was calibrated using pre-demonstration samples from the demonstration sampling sites, along with NIST standards.

VERIFICATION OF PERFORMANCE

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

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

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

Accuracy: Accuracy was evaluated based on the agreement of the ElvaX 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 ElvaX 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%	None.
Moderate	10% - 25%	Cadmium, Iron, Nickel, and Silver.
Low	25% - 50%	Antimony, Arsenic, Chromium, Copper, and Zinc.
Very Low	> 50%	Lead, Mercury, Selenium, and Vanadium.

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

	Antimony*	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Correlation	0.83	0.97	0.98	0.98	0.93	0.93	0.94	0.82	0.99	0.96	0.63	0.80	0.85
Bias	Low	--	--	Low	High	Low	High	Low	--	High	High	Low	--

Notes: -- = No significant bias
* Correlation is 0.98 with a lower 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 ElvaX was classified from high to very low for each target element, as indicated in the table below, based on the overall median RSDs. These results indicated an equivalent or higher level of precision in the ElvaX data than in the reference laboratory data for seven of the 13 target elements.

Relative Precision	Median RSD	Target Elements
High	0% - 5%	Copper and Iron.
Moderate	5% - 10%	Antimony*, Cadmium, Lead, Nickel, Selenium, Silver, and Zinc.
Low	10% - 20%	Arsenic, Chromium, and Mercury.
Very Low	> 20%	Vanadium.

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

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. Accuracy for copper was reduced from "moderate" (median RPDs less than 25 percent) to "very low" (median RPDs greater than 50 percent) by high relative concentrations of zinc (greater than 10X the arsenic concentration). Similarly, accuracy for nickel was reduced from "moderate" to "low" by high relative concentrations of copper. Low biases were produced in both the copper and nickel results by these interferences.

Effects of Soil Characteristics: The RPDs from the evaluation of accuracy were also further evaluated in terms of sampling site and soil type. A slight prevalence of outliers was noted for some of the target elements in complex mining wastes from the Leviathan Mine, Torch Lake, Sulphur Bank Mine, Alton Steel, Wickes Smelter, and Ramsey Flats sample blends. However, the ranges of RPDs observed for the target elements were very broad and sample matrix appeared to have only a minor effect on these RPDs.

Sample Throughput: The total processing time per sample was estimated at 12.2 minutes, which included 2.2 minutes of sample preparation and 9.5 minutes of instrument analysis time. On this basis, a sample throughput of

49 samples per 8-hour work day was estimated. It is possible that sample throughput could be increased by using the autosampler accessory available from the developer. As noted above, however, the sample blends had undergone rigorous pre-processing before the demonstration. Sample throughput would have decreased if these processing steps (grinding, drying, sieving) had been performed during the demonstration; these steps can add from 10 minutes to 2 hours to the sample processing time.

Costs: A cost assessment for the ElvaX identified a purchase cost of $35,000 as equipped for the demonstration. An autosampler ($20,000) and a helium purge unit ($10,000) are available as accessories. Using a hypothetical rental cost approximated from similar types of instruments, a total cost of $8,436 (with a labor cost of $5,901 at $43.75/hr) associated with sample preparation and analysis was estimated for a project similar to the demonstration (326 samples of soil and sediment). In comparison, the project cost averaged $8,932 for all eight XRF instruments participating in the demonstration, and $63,896 for fixed-laboratory analysis of all samples for the 13 target elements.

Skills and Training Required: A degreed chemist is recommended, but not required, to operate the ElvaX. A second technician without specific training is also recommended to assist with sample preparation and data processing if the autosampler is not used. Xcalibur offers technical support and training through a telephone helpline and on an as needed basis. The operating software features helpful on-screen prompts for instrument operation. However, the abbreviated operating instructions available during the demonstration included no specific procedures to assist with the preparation and analysis of environmental samples.

Health and Safety Aspects: The ElvaX's x-ray tube emits no detectable radiation thanks to two layers of lead shielding. The option of purging the sample chamber with helium requires the safe management of pressurized gas cylinders.

Portability: Based on its dimensions (48 X 38 X 20 centimeters) and weight (18 kilograms), the ElvaX is a compact transportable instrument designed to be used on a table top or possibly a truck bed. The instrument and its laptop computer require 110 volt AC power.

Durability: The ElvaX's x-ray tube has an expected lifespan of about 2 to 5 years of normal usage. The instrument is fully warranted for 1 year, and software is upgradeable for no cost. The instrument must be operated in a stable environment, free from excessive temperature and moisture extremes.

Availability: The ElvaX is manufactured by Elvatech, Ltd., in Kiev, Ukraine. Xcalibur XRF Services, Inc., distributes and services the ElvaX from locations on the east and west coasts of the U.S., and in the Great Lakes region. The ElvaX is available for purchase and long-term leasing; no short-term rental options are currently available.

RELATIVE PERFORMANCE

The overall performance of the ElvaX 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	●	O	●	●	Same	NC	O	Same	●	O	O	O	Same
Accuracy	●	O	Same	O	O	●	O	O	●	O	●	O	O
Precision	●	O	O	O	Same	O	O	O	O	O	O	O	O

Key: ● Better O 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 – Xcalibur ElvaX XRF Analyzer

1. Portability

The ElvaX offers compact size, low power consumption and can be connected to a DC to AC converter, making it useful as a portable EDXRF system. The ElvaX can be connected to a laptop computer via USB port and can be used in a truck bed or van for field measurement when heightened sensitivity or a longer measurement time is required. Although not available for this demonstration, the system can be equipped with an auto-sampler (carousel/changer) to allow greater measurement versatility. The optional auto-sampler does not add to the overall system size and only marginally adds to the weight.

2. Measurement Performance

The ElvaX is capable of measurement without standards (Fundamental Parameters method); however, the results will be normalized to 100%. This means that the measurement results will be less accurate when less detectable elements are present. Using the dual filter and detection methods increases the detection of lower levels of elements in the lower end of the spectrum. The best results are obtained using a full range of standards. Our standards were limited to those soil samples used in the first stages of this study and the limited NIST standards available.

Although Tetra Tech did an excellent job of providing prepared soil samples for this evaluation, optimum performance could have been achieved by further preparing the samples in pressed pellets. This would have added considerable time to sample preparation in exchange for better overall performance. The sample preparation we used was in keeping with the understood study concept of “field portability”.

The ElvaX has demonstrated excellent performance in measuring antimony, iron, nickel and chromium. Better results on other elements depend on the number of elements being measured simultaneously. Overlapping spectral peaks may pose a concern in certain situations where multiple elements are present (as in the case of arsenic and lead, where the lead La peak and arsenic Ka peaks overlap). This can be overcome by using alternate peaks if present (e.g., arsenic Kb at 11.7 kev and lead Lb at 12.6 kev); however, the detection limits may be higher.

3. Individual Element Comments

Arsenic & Lead: For the calibration and detection, we used Ka for arsenic and La for lead. But with standards, we got very good de-convolution.

Vanadium: The main problem in measuring vanadium was the overlapping Ti peaks which were not certified in the standards. As such it was not possible to calibrate the system for this element.

Selenium: A few more standards programmed into the Product File (calibration) would be required for better performance results on this element.

Overall performance can be enhanced with the addition of more standards for antimony, cadmium, and mercury.

4. Conclusion

This was the first inclusion in the EPA SITE program for Xcalibur XRF Services and ElvaTech. Additional preparation and standards would dramatically increase the accuracy of measurements in this test. The ElvaX system will continue to evolve as a reliable, efficient, cost-effective tool for elemental analysis.

APPENDIX C

DATA VALIDATION SUMMARY REPORT

Contents

ABBREVIATIONS AND ACRONYMS

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

1.0 INTRODUCTION

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

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

2.0 VALIDATION METHODOLOGY

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

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

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

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

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

- Holding times

- Initial and continuing calibrations
- Laboratory blank results
- Laboratory control sample (LCS) and laboratory control sample duplicates (LCSD) results
- Matrix spike (MS) and matrix spike duplicate (MSD) results
- Serial dilutions results

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

- ICP interference check samples (ICS)
- Target analyte identification and quantitation
- Quantitation limit verification

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

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

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

- Reviewing precision and accuracy of laboratory QC data
- Reviewing the overall analytical process, including holding time, calibration, analytical or matrix performance, and analyte identification and quantitation
- Assigning qualifiers to affected data when QA/QC criteria were not achieved
- Reviewing and summarizing implications of the frequency and severity of qualifiers in the validated data

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

- Total metal for 22 analytes by inductively coupled plasma atomic emission spectroscopy (ICP-AES) according to EPA Methods 3050B/6010B (EPA 1996)

- Total mercury by cold vapor atomic absorption spectroscopy (CVAA) according to EPA Method 7471A (EPA 1996)

3.0 DATA VALIDATION RESULTS

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

- Acceptable – All criteria were met and no data were qualified on that basis

- Acceptable with qualification – Most criteria were met, but at least one data point was qualified as estimated because of issues related to the review component

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

3.1 Holding Time

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

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

3.2 Calibration

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

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

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

3.3 Laboratory Blanks

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

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

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

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

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

3.4 Laboratory Control Samples

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

3.5 Matrix Spike Samples

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

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

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

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

3.6 Serial Dilution Results

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

3.7 ICP Interference Check Samples

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

3.8 Target Analyte Identification and Quantitation

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

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

3.9 Quantitation Limit Verification

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

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

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

4.1 Precision

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

4.2 Accuracy

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

4.3 Representativeness

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

4.4 Completeness

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

4.5 Comparability

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

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

5.0 CONCLUSIONS FOR DATA QUALITY AND DATA USABILITY

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

6.0 REFERENCES

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

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

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

TABLES

TABLE 1: DATA VALIDATION QUALIFIERS AND COMMENT CODES

Qualifier	Definition
No Qualifier	Indicates that the data are acceptable both qualitatively and quantitatively.
U	Indicates compound was analyzed for but not detected above the concentration listed. The value listed is the sample quantitation limit.
J	Indicates an estimated concentration value. The result is considered qualitatively acceptable, but quantitatively unreliable.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	Indicates an estimated quantitation limit. The compound was analyzed for, but was considered non-detected.
R	The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
Comment Code	**Definition**
a	Surrogate recovery exceeded (not applicable to this data set)
b	Laboratory method blank and common blank contamination
c	Calibration criteria exceeded
d	Duplicate precision criteria exceeded
e	Matrix spike or laboratory control sample recovery exceeded
f	Field blank contamination (not applicable to this data set)
g	Quantification below reporting limit
h	Holding time exceeded
i	Internal standard criteria exceeded (not applicable to this data set)
j	Other qualification (will be specified in report)

TABLE 2: QC CRITERIA

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

TABLE 3: DATA QUALIFICATION: LABORATORY METHOD BLANK CONTAMINATION

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

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

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

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

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

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

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

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

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Notes:

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

TABLE 5: DATA QUALIFICATION: SERIAL DILUTION EXCEEDANCES

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

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

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

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, Xcalibur ElvaX and Reference Laboratory

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
1	KP-SO-06-XX	Reference Laboratory	8.1 J+	1 J-	0.1 U	290	26	1,400	620	0.059 U
1	KP-SO-10-XX	Reference Laboratory	6.1 J+	1 J-	0.1 U	300	26	1,600	560	0.028 U
1	KP-SO-15-XX	Reference Laboratory	6.3 J+	1 J-	0.1 U	340	26	1,600	510	0.029 U
1	KP-SO-18-XX	Reference Laboratory	6.7 J+	1 J-	0.1 U	250	24	1,200	500	0.016 U
1	KP-SO-22-XX	Reference Laboratory	8.3 J+	1 J-	0.1 U	260	29	1,300	650	0.027 U
1	KP-SO-06-XC	Xcalibur XRF Services	8	68	25	306	17	1,292	478	
1	KP-SO-10-XC	Xcalibur XRF Services	12	111		323	14	1,287	527	2
1	KP-SO-15-XC	Xcalibur XRF Services	6	84		298	16	1,407	541	
1	KP-SO-18-XC	Xcalibur XRF Services	8	76		277	22	1,206	518	
1	KP-SO-22-XC	Xcalibur XRF Services	6	57	23	326	16	1,389	551	
2	KP-SO-07-XX	Reference Laboratory	17 J+	2 J-	0.1 U	170	48	990	1,200	0.027 U
2	KP-SO-13-XX	Reference Laboratory	16 J+	1 J-	0.045 U	180	52	980	1,200	0.037 U
2	KP-SO-20-XX	Reference Laboratory	19 J+	2 J-	0.1 U	160	46	910	1,300	0.03 U
2	KP-SO-24-XX	Reference Laboratory	17 J+	1 J-	0.1 U	160	49	900	1,100	0.017 U
2	KP-SO-27-XX	Reference Laboratory	15 J+	1 J-	0.05 U	170	45	970	1,200	0.021 U
2	KP-SO-29-XX	Reference Laboratory	18 J+	2 J-	0.1 U	150	42	870	1,200	0.013 U
2	KP-SO-32-XX	Reference Laboratory	16 J+	2 J-	0.045 U	180	50	970	1,200	0.014 U
2	KP-SO-07-XC	Xcalibur XRF Services	16	83	26	198	39	906	1,192	
2	KP-SO-13-XC	Xcalibur XRF Services	14	98		219	39	1,001	1,248	3
2	KP-SO-20-XC	Xcalibur XRF Services	20	73		193	35	933	1,156	
2	KP-SO-24-XC	Xcalibur XRF Services	21	85		205	33	930	1,150	
2	KP-SO-27-XC	Xcalibur XRF Services	23	43	26	182	43	922	1,252	
2	KP-SO-29-XC	Xcalibur XRF Services	14	44	30	217	37	964	1,279	
2	KP-SO-32-XC	Xcalibur XRF Services	21	0		216	40	914	1,168	
3	KP-SO-04-XX	Reference Laboratory	94 J+	3	0.046 U	180	200	1,300	5,800	0.018 U
3	KP-SO-16-XX	Reference Laboratory	93 J+	3	0.063 U	200	230	1,400	6,100	0.016 U
3	KP-SO-23-XX	Reference Laboratory	86 J+	3	0.048 U	180	190	1,300	5,300	0.017 U
3	KP-SO-26-XX	Reference Laboratory	90 J+	4	0.061 U	210	230	1,500	6,500	0.013 U
3	KP-SO-31-XX	Reference Laboratory	88	28	0.1 U	140	200	1,100	5,700	0.017 U
3	KP-SO-04-XC	Xcalibur XRF Services	71	176	36	163	172	1,057	5,603	5
3	KP-SO-16-XC	Xcalibur XRF Services	80	70	62	168	149	981	5,618	3
3	KP-SO-23-XC	Xcalibur XRF Services		77	82	185	183	1,079	5,944	3
3	KP-SO-26-XC	Xcalibur XRF Services	82	63		171	176	1,088	6,142	5
3	KP-SO-31-XC	Xcalibur XRF Services	74	0	24	146	156	903	5,458	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
1	KP-SO-06-XX	Reference Laboratory	140	0.25 U	0.25 U	2 J	11
1	KP-SO-10-XX	Reference Laboratory	150	0.22 U	0.25 U	2 J	12
1	KP-SO-15-XX	Reference Laboratory	170	0.25 U	0.25 U	2 J	15
1	KP-SO-18-XX	Reference Laboratory	120	0.25 U	0.25 U	2 J	11
1	KP-SO-22-XX	Reference Laboratory	130	0.25 U	0.25 U	2 J	11
1	KP-SO-06-XC	Xcalibur XRF Services	138	38		0	41
1	KP-SO-10-XC	Xcalibur XRF Services	144		13	0	0
1	KP-SO-15-XC	Xcalibur XRF Services	160			0	33
1	KP-SO-18-XC	Xcalibur XRF Services	138			0	21
1	KP-SO-22-XC	Xcalibur XRF Services	141	14	14	0	37
2	KP-SO-07-XX	Reference Laboratory	87	0.21 U	0.25 U	1 J	26
2	KP-SO-13-XX	Reference Laboratory	90	0.25 U	0.25 U	1 J	24
2	KP-SO-20-XX	Reference Laboratory	79	0.25 U	0.25 U	1 J	25
2	KP-SO-24-XX	Reference Laboratory	78	0.25 U	0.25 U	1 J	22
2	KP-SO-27-XX	Reference Laboratory	87	0.25 U	0.25 U	1 J	24
2	KP-SO-29-XX	Reference Laboratory	73	0.25 U	0.25 U	1 J	22
2	KP-SO-32-XX	Reference Laboratory	88	0.51	0.25 U	1 J	24
2	KP-SO-07-XC	Xcalibur XRF Services	93		13	0	67
2	KP-SO-13-XC	Xcalibur XRF Services	92		4	0	75
2	KP-SO-20-XC	Xcalibur XRF Services	86	11	5	0	56
2	KP-SO-24-XC	Xcalibur XRF Services	87			0	60
2	KP-SO-27-XC	Xcalibur XRF Services	107	35	3	0	78
2	KP-SO-29-XC	Xcalibur XRF Services	95	23	12	0	55
2	KP-SO-32-XC	Xcalibur XRF Services	83	50	6	0	60
3	KP-SO-04-XX	Reference Laboratory	93	0.28 U	0.16 J	1 J	45
3	KP-SO-16-XX	Reference Laboratory	100	0.25 U	0.16 J	1 J	47
3	KP-SO-23-XX	Reference Laboratory	91	0.25 U	0.13 J	1 J	41
3	KP-SO-26-XX	Reference Laboratory	110	0.22 U	0.17 J	1 J	52
3	KP-SO-31-XX	Reference Laboratory	68	0.25 U	0.4	2 J	38
3	KP-SO-04-XC	Xcalibur XRF Services	77	25		34	82
3	KP-SO-16-XC	Xcalibur XRF Services	70	6	11	0	80
3	KP-SO-23-XC	Xcalibur XRF Services	101	54	20	0	79
3	KP-SO-26-XC	Xcalibur XRF Services	87	59	4	0	94
3	KP-SO-31-XC	Xcalibur XRF Services	74	51	12	0	68

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
4	KP-SO-02-XX	Reference Laboratory	410	10	0.1	6	780	1,700	18,000	0.043 U
4	KP-SO-03-XX	Reference Laboratory	360	9	0.074 U	5	670	1,600	19,000	0.044 U
4	KP-SO-05-XX	Reference Laboratory	410	12	0.13 U	6	780	2,000	24,000	0.044 U
4	KP-SO-09-XX	Reference Laboratory	420	11	0.094 U	5	780	1,800	22,000	0.046 U
4	KP-SO-21-XX	Reference Laboratory	370	10	0.098 U	5	700	1,700	19,000	0.042 U
4	KP-SO-02-XC	Xcalibur XRF Services	233	0	32		614	1,047	18,603	15
4	KP-SO-03-XC	Xcalibur XRF Services	242	94			641	1,084	19,642	12
4	KP-SO-05-XC	Xcalibur XRF Services	237	0			609	1,130	19,166	12
4	KP-SO-09-XC	Xcalibur XRF Services	240	49			641	1,070	19,206	11
4	KP-SO-21-XC	Xcalibur XRF Services	252	86			666	1,167	20,448	13
5	WS-SO-06-XX	Reference Laboratory	1.3 U	48	1.9	120	50	28,000	110	0.07 U
5	WS-SO-08-XX	Reference Laboratory	1.3	45	2	120	47	26,000	71	0.063 U
5	WS-SO-12-XX	Reference Laboratory	1.3 UJ	43	1.8	110	45	25,000	65	0.068 UJ
5	WS-SO-17-XX	Reference Laboratory	1.3 UJ	47	1.9	120	49	28,000	70	0.069 UJ
5	WS-SO-27-XX	Reference Laboratory	1.3 UJ	49	2	120	51	28,000	72	0.11 J-
5	WS-SO-30-XX	Reference Laboratory	1.2 J-	51	2	130	53	29,000	81	0.069 UJ
5	WS-SO-35-XX	Reference Laboratory	1.3 UJ	49	2	130	51	28,000	74	0.071 UJ
5	WS-SO-06-XC	Xcalibur XRF Services	1	105	31	61	68	21,856	202	
5	WS-SO-08-XC	Xcalibur XRF Services	1	108		79	73	21,786	180	
5	WS-SO-12-XC	Xcalibur XRF Services	1	113		59	69	21,623	164	3
5	WS-SO-17-XC	Xcalibur XRF Services	1	102	25	49	70	21,219	181	5
5	WS-SO-27-XC	Xcalibur XRF Services	3	81		64	61	18,393	172	
5	WS-SO-30-XC	Xcalibur XRF Services	1	60	41	39	56	19,246	149	
5	WS-SO-35-XC	Xcalibur XRF Services	1	94	30		61	19,086	152	
6	WS-SO-03-XX	Reference Laboratory	8.9 J-	500	12	140	170	32,000	4,300	0.86 J-
6	WS-SO-05-XX	Reference Laboratory	8.6 J-	440	12	140	160	31,000	4,000	0.76 J-
6	WS-SO-09-XX	Reference Laboratory	7.1 J-	480	12	130	160	30,000	4,000	0.89 J-
6	WS-SO-14-XX	Reference Laboratory	8.4 J-	430	11	120	150	28,000	3,700	0.74 J-
6	WS-SO-26-XX	Reference Laboratory	7.6 J-	520	12	140	160	30,000	4,000	0.83 J-
6	WS-SO-31-XX	Reference Laboratory	7.2 J-	520	12	140	170	32,000	4,200	0.85 J-
6	WS-SO-33-XX	Reference Laboratory	6.9 J-	450 J-	11 J-	120 J-	150 J-	28,000 J-	3,700 J-	0.87 J-
6	WS-SO-03-XC	Xcalibur XRF Services	36	473	34	52	199	23,201	9,334	
6	WS-SO-05-XC	Xcalibur XRF Services	41	537	10	56	187	23,725	9,860	
6	WS-SO-09-XC	Xcalibur XRF Services	38	490	12	72	195	23,021	9,696	
6	WS-SO-14-XC	Xcalibur XRF Services	39	427		64	182	22,896	9,433	7
6	WS-SO-26-XC	Xcalibur XRF Services	23	297	36	51	150	19,241	6,110	27
6	WS-SO-31-XC	Xcalibur XRF Services	26	252			158	19,974	7,009	
6	WS-SO-33-XC	Xcalibur XRF Services	25	195	28	45	165	20,042	6,809	9

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
4	KP-SO-02-XX	Reference Laboratory	4	0.42 U	0.82	0 J	100
4	KP-SO-03-XX	Reference Laboratory	3	0.25 U	0.73	0 J	92
4	KP-SO-05-XX	Reference Laboratory	4	0.24 U	0.82	0 J	110
4	KP-SO-09-XX	Reference Laboratory	3	0.25 U	0.84	0 J	110
4	KP-SO-21-XX	Reference Laboratory	4	0.25 U	0.76	0 J	100
4	KP-SO-02-XC	Xcalibur XRF Services	2	275	42	0	176
4	KP-SO-03-XC	Xcalibur XRF Services	2	230	31	0	123
4	KP-SO-05-XC	Xcalibur XRF Services	3	231	13	0	154
4	KP-SO-09-XC	Xcalibur XRF Services	2	240	25	0	178
4	KP-SO-21-XC	Xcalibur XRF Services	3	211	27	0	196
5	WS-SO-06-XX	Reference Laboratory	61	1.3 U	0.93 J	56	230
5	WS-SO-08-XX	Reference Laboratory	58	1.3 U	0.86 J	52	220
5	WS-SO-12-XX	Reference Laboratory	55	1.3 U	0.94 J	49	210
5	WS-SO-17-XX	Reference Laboratory	59	1.3 U	0.89 J	56	230
5	WS-SO-27-XX	Reference Laboratory	61	1.3 U	0.9 J	57	230
5	WS-SO-30-XX	Reference Laboratory	65	1.3 U	1 J	58	240
5	WS-SO-35-XX	Reference Laboratory	62	1.3 U	1 J	57	240
5	WS-SO-06-XC	Xcalibur XRF Services	39	34	10	23	380
5	WS-SO-08-XC	Xcalibur XRF Services	57	15	11	24	387
5	WS-SO-12-XC	Xcalibur XRF Services	39	5	10	48	385
5	WS-SO-17-XC	Xcalibur XRF Services	38		10	15	384
5	WS-SO-27-XC	Xcalibur XRF Services	55		6	36	345
5	WS-SO-30-XC	Xcalibur XRF Services	35			28	345
5	WS-SO-35-XC	Xcalibur XRF Services	70		6	55	348
6	WS-SO-03-XX	Reference Laboratory	75	1.6	15	58	930
6	WS-SO-05-XX	Reference Laboratory	71	1.3 U	15	57	900
6	WS-SO-09-XX	Reference Laboratory	70	1.3 U	14	56	870
6	WS-SO-14-XX	Reference Laboratory	64	1.3 U	13	50	820
6	WS-SO-26-XX	Reference Laboratory	70	1.3 U	14	56	900
6	WS-SO-31-XX	Reference Laboratory	72	1.2 U	15	60	950
6	WS-SO-33-XX	Reference Laboratory	65 J-	1.3 U	13 J-	53 J-	830 J-
6	WS-SO-03-XC	Xcalibur XRF Services	71		27	23	1,191
6	WS-SO-05-XC	Xcalibur XRF Services	58	43	38	36	1,132
6	WS-SO-09-XC	Xcalibur XRF Services	55	19	30	13	1,190
6	WS-SO-14-XC	Xcalibur XRF Services	67		24	46	1,134
6	WS-SO-26-XC	Xcalibur XRF Services	60	16	18	31	934
6	WS-SO-31-XC	Xcalibur XRF Services	35		29	64	996
6	WS-SO-33-XC	Xcalibur XRF Services	67		15	0	1,020

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
7	WS-SO-01-XX	Reference Laboratory	41 J-	1900	47	100	590	32,000	18,000	5.8 J
7	WS-SO-04-XX	Reference Laboratory	45 J-	2000	50	94	640	34,000	20,000	6.5
7	WS-SO-15-XX	Reference Laboratory	48 J-	2300	56	82	720	37,000	24,000	5.8
7	WS-SO-22-XX	Reference Laboratory	41 J-	1900	47	84	620	33,000	17,000	4.8
7	WS-SO-34-XX	Reference Laboratory	45 J-	2000	50	91	660	36,000	22,000	5.4
7	WS-SO-01-XC	Xcalibur XRF Services	144	2,917	67	85	725	29,297	42,266	
7	WS-SO-04-XC	Xcalibur XRF Services	152	2,719	102	81	658	25,925	39,796	
7	WS-SO-15-XC	Xcalibur XRF Services	140	2,780	44	64	734	29,457	42,430	
7	WS-SO-22-XC	Xcalibur XRF Services	137	1,689	76	63	695	27,447	37,445	90
7	WS-SO-34-XC	Xcalibur XRF Services	136	2,066	47	60	597	23,244	30,445	65
8	WS-SO-02-XX	Reference Laboratory	130 J-	4200	98	49	1,300	44,000	35,000	17
8	WS-SO-16-XX	Reference Laboratory	110 J-	3900	91	59	1,300	42,000	24,000	15
8	WS-SO-18-XX	Reference Laboratory	130 J-	4100	95	63	1,300	44,000	37,000	17
8	WS-SO-21-XX	Reference Laboratory	120 J-	3900	90	43	1,200	40,000	43,000	14
8	WS-SO-24-XX	Reference Laboratory	97 J-	3600	81	54	1,100	38,000	27,000	16
8	WS-SO-29-XX	Reference Laboratory	120 J-	3800	90	51	1,200	40,000	42,000	15
8	WS-SO-37-XX	Reference Laboratory	120 J-	4100	95	63	1,300	42,000	26,000	14
8	WS-SO-02-XC	Xcalibur XRF Services	178	5,187	145	101	1,266	33,774	64,837	
8	WS-SO-16-XC	Xcalibur XRF Services	170	4,682	122	98	1,259	34,523	61,339	30
8	WS-SO-18-XC	Xcalibur XRF Services	194	3,655	120		1,211	31,756	59,680	46
8	WS-SO-21-XC	Xcalibur XRF Services	177	4,194	114		1,144	32,103	56,773	43
8	WS-SO-24-XC	Xcalibur XRF Services	202	4,123	82		932	24,425	40,706	116
8	WS-SO-29-XC	Xcalibur XRF Services	213	3,683	98	51	1,052	27,220	48,582	100
8	WS-SO-37-XC	Xcalibur XRF Services	202	4,497	62		1,071	28,622	48,690	28
9	WS-SO-13-XX	Reference Laboratory	200 J-	5800	150	53	1800	47,000	45,000	11
9	WS-SO-19-XX	Reference Laboratory	150 J-	5000	130	66	1500	39,000	24,000	12
9	WS-SO-28-XX	Reference Laboratory	120 J-	4200	100	54	1200	33,000	30,000	11
9	WS-SO-32-XX	Reference Laboratory	190 J-	5500	140	54	1700	44,000	30,000	11
9	WS-SO-36-XX	Reference Laboratory	120 J-	3800	92	51	1100	30,000	45,000	13
9	WS-SO-13-XC	Xcalibur XRF Services	208	7,772	139	67	1,511	35,738	47,796	
9	WS-SO-19-XC	Xcalibur XRF Services	178	4,948	184		1,559	39,189	48,951	84
9	WS-SO-28-XC	Xcalibur XRF Services	258	5,176	130		1,207	27,159	39,360	90
9	WS-SO-32-XC	Xcalibur XRF Services	237	5,696	131		1,331	29,668	41,616	52
9	WS-SO-36-XC	Xcalibur XRF Services	260	6,570	107		1,260	29,661	39,863	48

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se		Ag		V	Zn
7	WS-SO-01-XX	Reference Laboratory	66	1.3	U	69	J-	42	3,000
7	WS-SO-04-XX	Reference Laboratory	62	1.3	U	76	J-	44	3,100
7	WS-SO-15-XX	Reference Laboratory	58	1.3	U	90	J-	52	3,400
7	WS-SO-22-XX	Reference Laboratory	57	1.3	U	72	J-	44	3,000
7	WS-SO-34-XX	Reference Laboratory	60	1.3	U	78	J-	47	3,200
7	WS-SO-01-XC	Xcalibur XRF Services	42			105		0	4,227
7	WS-SO-04-XC	Xcalibur XRF Services	38			99		0	4,061
7	WS-SO-15-XC	Xcalibur XRF Services	79			104		0	4,265
7	WS-SO-22-XC	Xcalibur XRF Services	67			86		0	4,070
7	WS-SO-34-XC	Xcalibur XRF Services	65			59		0	3,534
8	WS-SO-02-XX	Reference Laboratory	57	1.3	U	150	J-	36	6,000
8	WS-SO-16-XX	Reference Laboratory	60	1.1	J	150	J-	35	5,700
8	WS-SO-18-XX	Reference Laboratory	62	1.9		140	J-	36	5,900
8	WS-SO-21-XX	Reference Laboratory	51	1.6		150	J-	33	5,500
8	WS-SO-24-XX	Reference Laboratory	54	2.1		140	J-	30	5,200
8	WS-SO-29-XX	Reference Laboratory	55	1.7		140	J-	33	5,500
8	WS-SO-37-XX	Reference Laboratory	63	3		140	J-	34	5,800
8	WS-SO-02-XC	Xcalibur XRF Services	95			195		0	7,030
8	WS-SO-16-XC	Xcalibur XRF Services	85			182		17	6,880
8	WS-SO-18-XC	Xcalibur XRF Services	42			199		41	6,777
8	WS-SO-21-XC	Xcalibur XRF Services	42			179		0	6,552
8	WS-SO-24-XC	Xcalibur XRF Services	90	126		105		12	5,451
8	WS-SO-29-XC	Xcalibur XRF Services	80			144		0	5,972
8	WS-SO-37-XC	Xcalibur XRF Services	39	44		117		0	5,885
9	WS-SO-13-XX	Reference Laboratory	75	3.7		170	J-	24	9,000
9	WS-SO-19-XX	Reference Laboratory	74	3.7		160	J-	20	7,700
9	WS-SO-28-XX	Reference Laboratory	59	2.3		130	J-	16	6,100
9	WS-SO-32-XX	Reference Laboratory	73	3.7		190	J-	23	8,500
9	WS-SO-36-XX	Reference Laboratory	55	1.7		120	J-	15	5,700
9	WS-SO-13-XC	Xcalibur XRF Services	112			206		0	9,997
9	WS-SO-19-XC	Xcalibur XRF Services	43			223		0	10,289
9	WS-SO-28-XC	Xcalibur XRF Services	90			137		0	8,400
9	WS-SO-32-XC	Xcalibur XRF Services	97			163		73	8,948
9	WS-SO-36-XC	Xcalibur XRF Services	100	14		144		77	8,632

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
10	BN-SO-01-XX	Reference Laboratory	1.3 UJ	38	0.94	120	32	24,000	63	0.13
10	BN-SO-10-XX	Reference Laboratory	1.3 UJ	50	1.2	110	35	24,000	140	0.14
10	BN-SO-15-XX	Reference Laboratory	1.3 UJ	34	0.82	110	29	22,000	56	0.15
10	BN-SO-18-XX	Reference Laboratory	1.3 U	37	0.89	110	29	22,000	59	0.13
10	BN-SO-28-XX	Reference Laboratory	1.5	35	0.87	100	28	22,000	58	0.16
10	BN-SO-31-XX	Reference Laboratory	1.3	41	1	140	33	26,000	65	0.14
10	BN-SO-35-XX	Reference Laboratory	1.4	37	0.98	120	30	23,000	60	0.15
10	BN-SO-01-XC	Xcalibur XRF Services	10	47	11	86	26	17,877	115	
10	BN-SO-10-XC	Xcalibur XRF Services	4	64		64	37	18,087	126	
10	BN-SO-15-XC	Xcalibur XRF Services	8	79	25	82	31	18,208	93	
10	BN-SO-18-XC	Xcalibur XRF Services	8	74	33	101	30	18,517	84	
10	BN-SO-28-XC	Xcalibur XRF Services	0	89		92	39	18,150	88	2
10	BN-SO-31-XC	Xcalibur XRF Services	7	84	25	85	46	18,130	163	
10	BN-SO-35-XC	Xcalibur XRF Services	9	89		78	45	18,569	134	5
11	BN-SO-02-XX	Reference Laboratory	11	140	50	90	170	28,000	840	0.37
11	BN-SO-04-XX	Reference Laboratory	9.1	120	42	79	140	24,000	700	0.36
11	BN-SO-17-XX	Reference Laboratory	9.3	110	39	79	140	23,000	680	0.39
11	BN-SO-22-XX	Reference Laboratory	7.3	98	34	65	110	20,000	590	0.37
11	BN-SO-27-XX	Reference Laboratory	9.6	110	39	78	130	24,000	660	0.38
11	BN-SO-02-XC	Xcalibur XRF Services	12	164	26	43	149	17,560	1,155	
11	BN-SO-04-XC	Xcalibur XRF Services	13	160	39	56	146	18,022	1,154	
11	BN-SO-17-XC	Xcalibur XRF Services	13	130	46	62	162	18,512	1,176	
11	BN-SO-22-XC	Xcalibur XRF Services	4	203	73	50	158	18,691	1,137	
11	BN-SO-27-XC	Xcalibur XRF Services	23	155	64	36	156	18,679	1,192	
12	BN-SO-03-XX	Reference Laboratory	65	620	290	120	840	25,000	4,700	1.6
12	BN-SO-06-XX	Reference Laboratory	60	600	280	94	810	24,000	4,500	2
12	BN-SO-08-XX	Reference Laboratory	57	570	270	100	750	22,000	4,300	2
12	BN-SO-13-XX	Reference Laboratory	65	320	150	98	410	17,000	2,400	1.6
12	BN-SO-20-XX	Reference Laboratory	57	540	260	88	730	22,000	4,100	1.6
12	BN-SO-30-XX	Reference Laboratory	64	630	300	100	860	26,000	4,800	1.6
12	BN-SO-34-XX	Reference Laboratory	68	630	290	110	830	25,000	4,700	2
12	BN-SO-03-XC	Xcalibur XRF Services	61	684	298	75	850	21,467	6,485	
12	BN-SO-06-XC	Xcalibur XRF Services	66	751	284	104	869	21,431	6,565	
12	BN-SO-08-XC	Xcalibur XRF Services	63	731	319	91	851	21,601	6,593	11
12	BN-SO-13-XC	Xcalibur XRF Services	70	715	326	60	848	21,842	6,781	9
12	BN-SO-20-XC	Xcalibur XRF Services	62	752	321	76	837	21,577	6,406	
12	BN-SO-30-XC	Xcalibur XRF Services	63	597	307	61	837	21,784	6,448	
12	BN-SO-34-XC	Xcalibur XRF Services	62	652	312	44	873	22,434	6,660	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
10	BN-SO-01-XX	Reference Laboratory	63	1.3 U	1.3 UJ	55	92
10	BN-SO-10-XX	Reference Laboratory	54	1.2 J	1.3 UJ	55	110
10	BN-SO-15-XX	Reference Laboratory	58	1.3 U	1.3 UJ	49	89
10	BN-SO-18-XX	Reference Laboratory	59	1.3	0.94 U	46	88
10	BN-SO-28-XX	Reference Laboratory	54	1.3 U	0.77 U	48	81
10	BN-SO-31-XX	Reference Laboratory	71	1.3 U	0.97 U	54	94
10	BN-SO-35-XX	Reference Laboratory	63	1.2 J	0.85 U	50	87
10	BN-SO-01-XC	Xcalibur XRF Services	54		11	0	155
10	BN-SO-10-XC	Xcalibur XRF Services	69		11	15	172
10	BN-SO-15-XC	Xcalibur XRF Services	60			34	157
10	BN-SO-18-XC	Xcalibur XRF Services	85	2	18	0	169
10	BN-SO-28-XC	Xcalibur XRF Services	66		8	0	159
10	BN-SO-31-XC	Xcalibur XRF Services	71			46	206
10	BN-SO-35-XC	Xcalibur XRF Services	61		6	44	193
11	BN-SO-02-XX	Reference Laboratory	54	4.3	7.6	60	470
11	BN-SO-04-XX	Reference Laboratory	48	2.9	6.5	50	400
11	BN-SO-17-XX	Reference Laboratory	47	2.7	6.3	49	390
11	BN-SO-22-XX	Reference Laboratory	40	2.8	5.4	43	330
11	BN-SO-27-XX	Reference Laboratory	46	3.7	6.1	52	380
11	BN-SO-02-XC	Xcalibur XRF Services	32	6		29	665
11	BN-SO-04-XC	Xcalibur XRF Services	33			0	643
11	BN-SO-17-XC	Xcalibur XRF Services	57	72	14	42	646
11	BN-SO-22-XC	Xcalibur XRF Services	59			14	669
11	BN-SO-27-XC	Xcalibur XRF Services	34		14	24	661
12	BN-SO-03-XX	Reference Laboratory	100	17	42	48	2,300
12	BN-SO-06-XX	Reference Laboratory	92	15	41	48	2,300
12	BN-SO-08-XX	Reference Laboratory	94	14	38	39	2,200
12	BN-SO-13-XX	Reference Laboratory	71	9.2	21	37	1,200
12	BN-SO-20-XX	Reference Laboratory	84	14	37	44	2,100
12	BN-SO-30-XX	Reference Laboratory	99	17	44	50	2,400
12	BN-SO-34-XX	Reference Laboratory	100	17	42	49	2,300
12	BN-SO-03-XC	Xcalibur XRF Services	83	67	33	61	3,299
12	BN-SO-06-XC	Xcalibur XRF Services	72		44	44	3,454
12	BN-SO-08-XC	Xcalibur XRF Services	79	6	41	0	3,331
12	BN-SO-13-XC	Xcalibur XRF Services	69	114	65	0	3,358
12	BN-SO-20-XC	Xcalibur XRF Services	86	65	31	0	3,356
12	BN-SO-30-XC	Xcalibur XRF Services	76	101	37	0	3,325
12	BN-SO-34-XC	Xcalibur XRF Services	82		58	0	3,406

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
13	BN-SO-07-XX	Reference Laboratory	110 J-	990 J+	520	82	1,400	23,000	6,900	3.4
13	BN-SO-16-XX	Reference Laboratory	120 J-	1,100 J+	570	86	1,500	25,000	8,100	3.4
13	BN-SO-21-XX	Reference Laboratory	150 J-	1,300 J+	660	110	1,700	30,000	8,900	3.6
13	BN-SO-25-XX	Reference Laboratory	82 J-	700 J	370 J-	64 J-	930 J-	16,000 J-	5,400 J-	3.8
13	BN-SO-33-XX	Reference Laboratory	100 J-	1,100	640	100	1,600	27,000	8,000	4
13	BN-SO-07-XC	Xcalibur XRF Services	100	1,133	601		1,456	23,645	8,902	
13	BN-SO-16-XC	Xcalibur XRF Services	99	1,134	561	74	1,457	23,360	8,699	
13	BN-SO-21-XC	Xcalibur XRF Services	105	1,120	586	57	1,466	24,645	8,942	
13	BN-SO-25-XC	Xcalibur XRF Services	103	1,016	592	105	1,369	22,952	8,762	
13	BN-SO-33-XC	Xcalibur XRF Services	100	1,243	599	39	1,527	25,053	8,705	
14	BN-SO-05-XX	Reference Laboratory	160 J-	1,600	850	86	2,200	26,000	12,000	5
14	BN-SO-19-XX	Reference Laboratory	150 J-	1,600	860	79	2,200	26,000	12,000	5
14	BN-SO-26-XX	Reference Laboratory	150 J-	1,700	900	82	2,400	27,000	12,000	5.4
14	BN-SO-29-XX	Reference Laboratory	150 J-	1,600	880	86	2,300	26,000	12,000	5.4
14	BN-SO-32-XX	Reference Laboratory	160 J-	1,600	860	84	2,300	26,000	12,000	5.4
14	BN-SO-05-XC	Xcalibur XRF Services	136	1,935	808	87	2,193	26,751	10,794	
14	BN-SO-19-XC	Xcalibur XRF Services	131	1,673	811	63	2,199	27,009	10,790	
14	BN-SO-26-XC	Xcalibur XRF Services	134	1,719	821	45	2,194	26,274	10,750	
14	BN-SO-29-XC	Xcalibur XRF Services	130	1,751	834		2,254	27,470	10,894	
14	BN-SO-32-XC	Xcalibur XRF Services	132	1,843	852	45	2,187	26,292	10,562	
15	CN-SO-01-XX	Reference Laboratory	13 J-	13	21	190	700	38,000	1,200	0.13
15	CN-SO-04-XX	Reference Laboratory	13 J-	11	21	200	680	37,000	1,200	0.14
15	CN-SO-08-XX	Reference Laboratory	15 J-	15	25	210	740	43,000	1,300	0.16
15	CN-SO-10-XX	Reference Laboratory	13 J-	13	22	200	760	39,000	1,200	0.12
15	CN-SO-11-XX	Reference Laboratory	17 J-	16	30	240	860	47,000	1,600	0.15
15	CN-SO-01-XC	Xcalibur XRF Services	16	99	20	158	731	34,316	1,738	
15	CN-SO-04-XC	Xcalibur XRF Services	19	111		179	741	34,495	1,901	
15	CN-SO-08-XC	Xcalibur XRF Services	19	40	19	179	737	32,236	1,790	
15	CN-SO-10-XC	Xcalibur XRF Services	14	83	20	170	752	33,088	1,755	
15	CN-SO-11-XC	Xcalibur XRF Services	21	93		155	729	33,039	1,806	
16	AS-SO-02-XX	Reference Laboratory	2.6 UJ	18	50	180	140	48,000	1,600	0.76
16	AS-SO-06-XX	Reference Laboratory	2.4 UJ	19	52	190	130	52,000	1,600	0.74
16	AS-SO-10-XX	Reference Laboratory	1.9 J-	18	48	180	110	45,000	1,400	0.78
16	AS-SO-11-XX	Reference Laboratory	3.7 J-	22	63	230	150	52,000	2,100	0.72
16	AS-SO-13-XX	Reference Laboratory	2.4 UJ	20	57	200	150	52,000	1,700	0.79
16	AS-SO-02-XC	Xcalibur XRF Services	14	85	64	146	311	33,843	2,497	10
16	AS-SO-06-XC	Xcalibur XRF Services	12	85	33	225	319	36,284	2,390	15
16	AS-SO-10-XC	Xcalibur XRF Services	12	34	63	172	330	36,503	2,495	17
16	AS-SO-11-XC	Xcalibur XRF Services	14	42	62	154	349	38,108	2,669	12
16	AS-SO-13-XC	Xcalibur XRF Services	13	41	47	175	350	36,984	2,579	8

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
13	BN-SO-07-XX	Reference Laboratory	120		26		70		41		4,000	
13	BN-SO-16-XX	Reference Laboratory	130		29		77		44		4,400	
13	BN-SO-21-XX	Reference Laboratory	160		35		88		52		5,100	
13	BN-SO-25-XX	Reference Laboratory	88	J-	19	J-	48	J-	28	J-	2,900	J-
13	BN-SO-33-XX	Reference Laboratory	150		34		81		48		5,100	
13	BN-SO-07-XC	Xcalibur XRF Services	100		47		75		31		5,643	
13	BN-SO-16-XC	Xcalibur XRF Services	71		15		73		41		5,547	
13	BN-SO-21-XC	Xcalibur XRF Services	103		20		76		0		5,498	
13	BN-SO-25-XC	Xcalibur XRF Services	99				66		0		5,524	
13	BN-SO-33-XC	Xcalibur XRF Services	117				84		0		5,746	
14	BN-SO-05-XX	Reference Laboratory	160		48		110		39		6,700	
14	BN-SO-19-XX	Reference Laboratory	160		48		120		39		6,700	
14	BN-SO-26-XX	Reference Laboratory	160		49		120		40		7,000	
14	BN-SO-29-XX	Reference Laboratory	160		48		120		41		6,800	
14	BN-SO-32-XX	Reference Laboratory	160		48		120		39		6,700	
14	BN-SO-05-XC	Xcalibur XRF Services	113		100		123		0		7,367	
14	BN-SO-19-XC	Xcalibur XRF Services	134		72		106		0		7,318	
14	BN-SO-26-XC	Xcalibur XRF Services	107				117		0		7,323	
14	BN-SO-29-XC	Xcalibur XRF Services	126				128		0		7,482	
14	BN-SO-32-XC	Xcalibur XRF Services	125				115		30		7,359	
15	CN-SO-01-XX	Reference Laboratory	240		2.2		12		21		3,100	
15	CN-SO-04-XX	Reference Laboratory	240		1.5		12		22		2,900	
15	CN-SO-08-XX	Reference Laboratory	280		1.3	U	15		26		3,200	
15	CN-SO-10-XX	Reference Laboratory	240		1.9		14		22		3,000	
15	CN-SO-11-XX	Reference Laboratory	320		1.3	U	16		27		3,500	
15	CN-SO-01-XC	Xcalibur XRF Services	198				18		48		3,910	
15	CN-SO-04-XC	Xcalibur XRF Services	183				23		0		3,742	
15	CN-SO-08-XC	Xcalibur XRF Services	184		55		3		51		3,732	
15	CN-SO-10-XC	Xcalibur XRF Services	185				15		56		3,451	
15	CN-SO-11-XC	Xcalibur XRF Services	217				3		0		3,451	
16	AS-SO-02-XX	Reference Laboratory	91		2.6	U	4.5		42		3,300	
16	AS-SO-06-XX	Reference Laboratory	93		2.6	U	4.8		44		3,500	
16	AS-SO-10-XX	Reference Laboratory	84		1.1	U	4.4		42		3,000	
16	AS-SO-11-XX	Reference Laboratory	120		1.1	U	5.6		54		3,800	
16	AS-SO-13-XX	Reference Laboratory	100		3		5.2		50		3,800	
16	AS-SO-02-XC	Xcalibur XRF Services	92				5		60		3,543	
16	AS-SO-06-XC	Xcalibur XRF Services	86						0		3,424	
16	AS-SO-10-XC	Xcalibur XRF Services	95				0		0		3,321	
16	AS-SO-11-XC	Xcalibur XRF Services	86				10		0		3,440	
16	AS-SO-13-XC	Xcalibur XRF Services	86				8		0		3,539	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
17	AS-SO-01-XX	Reference Laboratory	3.8 J-	26	100	420	250	100,000	3,200	1.4
17	AS-SO-04-XX	Reference Laboratory	6.4 UJ	22	110	480	260	110,000	3,300	1.3
17	AS-SO-07-XX	Reference Laboratory	3.6 J-	21	97	380	240	88,000	2,900	1.4
17	AS-SO-09-XX	Reference Laboratory	2.6 UJ	25 J-	100 J-	390 J-	250 J-	94,000 J-	3,200 J-	1.4
17	AS-SO-12-XX	Reference Laboratory	2.6 UJ	29	120	440	270	93,000	3,300	1.4
17	AS-SO-01-XC	Xcalibur XRF Services	4	74	67	371	591	74,049	6,123	53
17	AS-SO-04-XC	Xcalibur XRF Services		46	75	364	667	79,399	6,244	56
17	AS-SO-07-XC	Xcalibur XRF Services		45	105	415	623	78,896	6,402	51
17	AS-SO-09-XC	Xcalibur XRF Services	6	46	78	523	629	83,038	6,597	47
17	AS-SO-12-XC	Xcalibur XRF Services	6	85	84	313	686	82,623	6,547	44
18	SB-SO-03-XX	Reference Laboratory	1.2 UJ	9	0.51 U	150	48	38,000	18	62
18	SB-SO-06-XX	Reference Laboratory	1.7 J-	8	0.51 U	140	44	35,000	16	55
18	SB-SO-14-XX	Reference Laboratory	4.1 J-	9	0.51 U	150	46	37,000	17	55
18	SB-SO-38-XX	Reference Laboratory	1.3 UJ	10	0.51 U	150	57	37,000	18	56
18	SB-SO-41-XX	Reference Laboratory	1.3 UJ	9	0.51 U	160	58	40,000	19	54
18	SB-SO-47-XX	Reference Laboratory	1.3 UJ	8	0.51 U	140	44	34,000	16	58
18	SB-SO-51-XX	Reference Laboratory	1.3 UJ	9	0.51 U	160	50	40,000	18	54
18	SB-SO-03-XC	Xcalibur XRF Services	6	50	10	104	28	27,814	85	11
18	SB-SO-06-XC	Xcalibur XRF Services	0	63		115	20	27,713	88	15
18	SB-SO-14-XC	Xcalibur XRF Services	3	82		130	37	27,854	52	16
18	SB-SO-38-XC	Xcalibur XRF Services	4	72		148	27	27,016	75	19
18	SB-SO-41-XC	Xcalibur XRF Services	9	63		160	28	26,866	87	22
18	SB-SO-47-XC	Xcalibur XRF Services	15	77		183	29	27,284	0	9
18	SB-SO-51-XC	Xcalibur XRF Services	4	60	48	119	33	27,183	106	29
19	SB-SO-05-XX	Reference Laboratory	1.6 J-	9	0.51 U	140	46	35,000	16	540
19	SB-SO-18-XX	Reference Laboratory	1.2 UJ	10	0.51 U	150	46	38,000	17	280
19	SB-SO-30-XX	Reference Laboratory	3.2 J-	7	0.51 U	94	27	22,000	10	290
19	SB-SO-40-XX	Reference Laboratory	2.2 J-	9	0.51 U	120	40	33,000	15	280
19	SB-SO-53-XX	Reference Laboratory	1.2 UJ	10	0.51 U	140	44	37,000	17	270
19	SB-SO-05-XC	Xcalibur XRF Services	9	70	29	132	5	25,964	0	219
19	SB-SO-18-XC	Xcalibur XRF Services	0	82	34	68	23	23,181	68	155
19	SB-SO-30-XC	Xcalibur XRF Services	0	65	31	62	39	25,684	64	187
19	SB-SO-40-XC	Xcalibur XRF Services	0	72		153	37	25,849	0	205
19	SB-SO-53-XC	Xcalibur XRF Services	13	46		106	31	26,290	41	195

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
17	AS-SO-01-XX	Reference Laboratory	180	2.6 U	9.3	66	6,900
17	AS-SO-04-XX	Reference Laboratory	200	6.2 U	12	72	7,400
17	AS-SO-07-XX	Reference Laboratory	160	2.7	8.9	63	6,300
17	AS-SO-09-XX	Reference Laboratory	170 J-	2.6 U	9.6 J-	65 J-	6,800 J-
17	AS-SO-12-XX	Reference Laboratory	190	2.6 U	3.2	73	7,500
17	AS-SO-01-XC	Xcalibur XRF Services	130		3	88	2,484
17	AS-SO-04-XC	Xcalibur XRF Services	153		4	0	2,195
17	AS-SO-07-XC	Xcalibur XRF Services	159	24	9	0	2,196
17	AS-SO-09-XC	Xcalibur XRF Services	119		9	0	1,858
17	AS-SO-12-XC	Xcalibur XRF Services	133		7	37	2,034
18	SB-SO-03-XX	Reference Laboratory	210	1.3 U	1.3 U	67	90
18	SB-SO-06-XX	Reference Laboratory	200	1.3 U	1.3 U	63	82
18	SB-SO-14-XX	Reference Laboratory	210	1.3 U	1.3 U	66	95
18	SB-SO-38-XX	Reference Laboratory	210	1.3 U	1.3 U	68	91
18	SB-SO-41-XX	Reference Laboratory	230	1.3 U	1.3 U	71	96
18	SB-SO-47-XX	Reference Laboratory	200	1.3 U	1.3 U	62	82
18	SB-SO-51-XX	Reference Laboratory	230	1.3 U	1.3 U	74	93
18	SB-SO-03-XC	Xcalibur XRF Services	179	54	4	90	109
18	SB-SO-06-XC	Xcalibur XRF Services	197			36	98
18	SB-SO-14-XC	Xcalibur XRF Services	173		6	77	108
18	SB-SO-38-XC	Xcalibur XRF Services	196			35	114
18	SB-SO-41-XC	Xcalibur XRF Services	171			0	98
18	SB-SO-47-XC	Xcalibur XRF Services	168			0	114
18	SB-SO-51-XC	Xcalibur XRF Services	167		6	55	102
19	SB-SO-05-XX	Reference Laboratory	200	1.3 U	1.3 U	61	80
19	SB-SO-18-XX	Reference Laboratory	210	1.3 U	1.3 U	70	84
19	SB-SO-30-XX	Reference Laboratory	120	1.3 J+	1.3 U	43	50
19	SB-SO-40-XX	Reference Laboratory	180	1.3 U	1.3 U	58	74
19	SB-SO-53-XX	Reference Laboratory	200	1.3 U	1.3 U	64	81
19	SB-SO-05-XC	Xcalibur XRF Services	165			62	92
19	SB-SO-18-XC	Xcalibur XRF Services	139			0	89
19	SB-SO-30-XC	Xcalibur XRF Services	144	13	8	147	90
19	SB-SO-40-XC	Xcalibur XRF Services	154		16	42	106
19	SB-SO-53-XC	Xcalibur XRF Services	158		8	74	88

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
20	SB-SO-08-XX	Reference Laboratory	5.4 J-	13	0.51 U	120	39	32,000	17	730
20	SB-SO-11-XX	Reference Laboratory	5.7 J-	13	0.51 U	140	46	36,000	20	810
20	SB-SO-21-XX	Reference Laboratory	4.9 J	13	0.51 U	130	43	34,000	18	740
20	SB-SO-39-XX	Reference Laboratory	4.7 J-	13	0.51 U	140	46	34,000	19	790
20	SB-SO-42-XX	Reference Laboratory	4.6 J-	13	0.51 U	140	45	35,000	18	740
20	SB-SO-08-XC	Xcalibur XRF Services	10	63		109	25	24,137	0	656
20	SB-SO-11-XC	Xcalibur XRF Services	11	71		97	26	24,094	0	650
20	SB-SO-21-XC	Xcalibur XRF Services	0	72		51	18	22,026	0	485
20	SB-SO-39-XC	Xcalibur XRF Services	8	90		133	27	23,521	0	632
20	SB-SO-42-XC	Xcalibur XRF Services	13	120	26	132	5	24,055	0	617
21	SB-SO-22-XX	Reference Laboratory	10 J	18	0.51 U	120	37	29,000	22	3300
21	SB-SO-25-XX	Reference Laboratory	6.8 J+	18	0.51 U	120	37	29,000	22	3000
21	SB-SO-27-XX	Reference Laboratory	6.7 J+	18	0.51 U	120	37	29,000	22	3100
21	SB-SO-35-XX	Reference Laboratory	6 J+	17	0.51 U	110	35	28,000	21	3100
21	SB-SO-44-XX	Reference Laboratory	6.8 J+	18	0.51 U	120	37	29,000	22	3000
21	SB-SO-22-XC	Xcalibur XRF Services	10	124		51	16	18,480	0	1,057
21	SB-SO-25-XC	Xcalibur XRF Services	14	109	0	20	3	19,155	0	1,107
21	SB-SO-27-XC	Xcalibur XRF Services	15	112			20	19,141	0	1,128
21	SB-SO-35-XC	Xcalibur XRF Services	14	119		53	22	19,400	0	1,233
21	SB-SO-44-XC	Xcalibur XRF Services	20	116		61	25	19,543	0	1,283
22	SB-SO-23-XX	Reference Laboratory	48 J-	37	0.1 U	21	7	4,500	36	8500
22	SB-SO-28-XX	Reference Laboratory	42 J-	36	0.1 U	21	7	4,400	36	8800
22	SB-SO-32-XX	Reference Laboratory	46 J-	40	0.1 U	23	7.6	4,900	40	8900
22	SB-SO-43-XX	Reference Laboratory	40 J-	35	0.1 U	20	6.7	4,200	34	7600
22	SB-SO-48-XX	Reference Laboratory	39 J-	36	0.1 U	21	6.9	4,500	36	8200
22	SB-SO-23-XC	Xcalibur XRF Services	35	317	0	36	1	2,818	0	1,347
22	SB-SO-28-XC	Xcalibur XRF Services	39	347	12	25	0	2,961	0	1,494
22	SB-SO-32-XC	Xcalibur XRF Services	38	349	0		0	2,881	0	1,505
22	SB-SO-43-XC	Xcalibur XRF Services	34	425	40		1	2,850	0	1,494
22	SB-SO-48-XC	Xcalibur XRF Services	39	367	0		0	2,881	0	1,496
23	SB-SO-02-XX	Reference Laboratory	44 J-	23 J-	0.5 U	130	43	35,000	22 J-	130 J+
23	SB-SO-07-XX	Reference Laboratory	45 J	22	0.5 U	120	38	35,000	23	270
23	SB-SO-10-XX	Reference Laboratory	62 J	26	0.5 U	140	44	41,000	27	220
23	SB-SO-26-XX	Reference Laboratory	61 J	30	0.5 U	160	50	46,000	31	260
23	SB-SO-50-XX	Reference Laboratory	57 J	27	0.5 U	140	46	42,000	28	200
23	SB-SO-02-XC	Xcalibur XRF Services	49	99	35	115	30	28,994	49	74
23	SB-SO-07-XC	Xcalibur XRF Services	58	72		118	26	29,271	67	87
23	SB-SO-10-XC	Xcalibur XRF Services	53	52		122	38	29,497	74	69
23	SB-SO-26-XC	Xcalibur XRF Services	53	46	38	101	40	27,573	83	62
23	SB-SO-50-XC	Xcalibur XRF Services	50	74	24	134	39	28,740	0	74

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
20	SB-SO-08-XX	Reference Laboratory	180	1.3 U	1.3 U	57	70
20	SB-SO-11-XX	Reference Laboratory	200	1.3 U	1.3 U	66	84
20	SB-SO-21-XX	Reference Laboratory	190	1.3 U	1.3 U	58	75
20	SB-SO-39-XX	Reference Laboratory	200	1.3 U	1.3 U	62	77
20	SB-SO-42-XX	Reference Laboratory	200	1.3 U	1.3 U	65	78
20	SB-SO-08-XC	Xcalibur XRF Services	154			29	74
20	SB-SO-11-XC	Xcalibur XRF Services	134		1	96	72
20	SB-SO-21-XC	Xcalibur XRF Services	120			28	60
20	SB-SO-39-XC	Xcalibur XRF Services	135		18	53	77
20	SB-SO-42-XC	Xcalibur XRF Services	135			63	98
21	SB-SO-22-XX	Reference Laboratory	160	1.3 U	1.3 U	52	64 J-
21	SB-SO-25-XX	Reference Laboratory	160	1.3 U	1.3 U	54	63
21	SB-SO-27-XX	Reference Laboratory	170	1.3 U	1.3 U	54	65
21	SB-SO-35-XX	Reference Laboratory	160	1.3 U	1.3 U	50	62
21	SB-SO-44-XX	Reference Laboratory	170	1.3 U	1.3 U	53	64
21	SB-SO-22-XC	Xcalibur XRF Services	114			22	58
21	SB-SO-25-XC	Xcalibur XRF Services	119			121	67
21	SB-SO-27-XC	Xcalibur XRF Services	117	52		69	57
21	SB-SO-35-XC	Xcalibur XRF Services	121	25	12	96	60
21	SB-SO-44-XC	Xcalibur XRF Services	113		29	68	67
22	SB-SO-23-XX	Reference Laboratory	26	0.22 J	0.26 UJ	13	8
22	SB-SO-28-XX	Reference Laboratory	26	0.26 U	0.26 UJ	13	8
22	SB-SO-32-XX	Reference Laboratory	28	0.36	0.1 UJ	14	9
22	SB-SO-43-XX	Reference Laboratory	24	0.26 U	0.26 UJ	13	8
22	SB-SO-48-XX	Reference Laboratory	25	0.26 U	0.1 UJ	13	8
22	SB-SO-23-XC	Xcalibur XRF Services	6		7	147	18
22	SB-SO-28-XC	Xcalibur XRF Services	6		5	93	0
22	SB-SO-32-XC	Xcalibur XRF Services	6		15	156	0
22	SB-SO-43-XC	Xcalibur XRF Services	24		5	162	13
22	SB-SO-48-XC	Xcalibur XRF Services	6			135	0
23	SB-SO-02-XX	Reference Laboratory	180	1.2 U	1.2 UJ	59	88
23	SB-SO-07-XX	Reference Laboratory	170	1.4	1.6	53	86
23	SB-SO-10-XX	Reference Laboratory	200	2.8	1.8	59	100
23	SB-SO-26-XX	Reference Laboratory	220	3.4	1.8	68	110
23	SB-SO-50-XX	Reference Laboratory	200	2.9	1.8	61	100
23	SB-SO-02-XC	Xcalibur XRF Services	162		12	0	113
23	SB-SO-07-XC	Xcalibur XRF Services	160		18	0	114
23	SB-SO-10-XC	Xcalibur XRF Services	166	12	18	0	117
23	SB-SO-26-XC	Xcalibur XRF Services	173			43	103
23	SB-SO-50-XC	Xcalibur XRF Services	190		15	38	114

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
24	SB-SO-01-XX	Reference Laboratory	180 J	65	0.5 U	140	46	47,000	30	400
24	SB-SO-16-XX	Reference Laboratory	170 J	64	0.5 U	140	45	47,000	30	480
24	SB-SO-24-XX	Reference Laboratory	180 J	66	0.5 U	150	49	49,000	32	420
24	SB-SO-45-XX	Reference Laboratory	180 J	63	0.5 U	140	45	47,000	30	450
24	SB-SO-52-XX	Reference Laboratory	150 J	62	0.5 U	140	47	46,000	29	430
24	SB-SO-01-XC	Xcalibur XRF Services	123	83		132	44	32,072	67	158
24	SB-SO-16-XC	Xcalibur XRF Services	129	108	33	115	30	30,888	0	141
24	SB-SO-24-XC	Xcalibur XRF Services	105	103		76	25	28,359	0	111
24	SB-SO-45-XC	Xcalibur XRF Services	122	88	25	161	37	30,355	83	168
24	SB-SO-52-XC	Xcalibur XRF Services	131	94		110	38	29,892	76	153
25	SB-SO-13-XX	Reference Laboratory	430 J	160	1 U	140	46	61,000	36	850
25	SB-SO-19-XX	Reference Laboratory	310 J	100	0.5 U	100	32	42,000	25	740
25	SB-SO-33-XX	Reference Laboratory	350 J	110	0.5 U	100	33	45,000	28	870
25	SB-SO-37-XX	Reference Laboratory	340 J	130	1 U	120	39	51,000	31	790
25	SB-SO-55-XX	Reference Laboratory	340 J	120	0.5 U	120	37	49,000	29	900
25	SB-SO-13-XC	Xcalibur XRF Services	280	194	28	92	32	35,473	58	339
25	SB-SO-19-XC	Xcalibur XRF Services	210	174	24		22	31,909	0	220
25	SB-SO-33-XC	Xcalibur XRF Services	259	195	30	92	19	34,068	0	293
25	SB-SO-37-XC	Xcalibur XRF Services	258	172		170	35	36,020	76	310
25	SB-SO-55-XC	Xcalibur XRF Services	278	195		99	35	36,018	105	338
26	SB-SO-12-XX	Reference Laboratory	620 J	190	1 U	100	33	55,000	43	1,400
26	SB-SO-15-XX	Reference Laboratory	600 J-	170 J-	1 U	91 J-	30 J-	51,000 J-	40 J-	1,100
26	SB-SO-17-XX	Reference Laboratory	800 J+	210	1 U	110	37	61,000	48	1,200
26	SB-SO-46-XX	Reference Laboratory	740 J+	190	1 U	120	35	57,000	47	670
26	SB-SO-54-XX	Reference Laboratory	280	31	0.2 U	25	5.8	8,600	5 J-	560
26	SB-SO-12-XC	Xcalibur XRF Services	428	266	27	132	20	39,828	91	570
26	SB-SO-15-XC	Xcalibur XRF Services	419	298		133	28	39,562	0	524
26	SB-SO-17-XC	Xcalibur XRF Services	257	215		32	22	33,967	0	337
26	SB-SO-46-XC	Xcalibur XRF Services	417	259	29	133	24	39,119	80	504
26	SB-SO-54-XC	Xcalibur XRF Services	425	260	28	204	28	39,886	84	495
27	KP-SE-08-XX	Reference Laboratory	6.2	3	0.11 U	88	3.8	840	300 J-	0.089 U
27	KP-SE-11-XX	Reference Laboratory	5.6	3	0.11 U	96	4.1	940	310 J-	0.079 U
27	KP-SE-17-XX	Reference Laboratory	4.9	3	0.11 U	98	4.1	940	300 J-	0.082 U
27	KP-SE-25-XX	Reference Laboratory	6	3	0.11 U	99	4.3	960	310 J-	0.096 U
27	KP-SE-30-XX	Reference Laboratory	5.7	3	0.11 U	83	3.6	830	300 J-	0.1 U
27	KP-SE-08-XC	Xcalibur XRF Services	13	126		96	0	966	442	
27	KP-SE-11-XC	Xcalibur XRF Services	16	82		94	1	940	450	
27	KP-SE-17-XC	Xcalibur XRF Services	17	76	32	92	1	1,036	458	3
27	KP-SE-25-XC	Xcalibur XRF Services	12	0		113	1	1,039	512	3
27	KP-SE-30-XC	Xcalibur XRF Services	14	40	28	93	1	1,048	491	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
24	SB-SO-01-XX	Reference Laboratory	190	1.8	2.3	65	95
24	SB-SO-16-XX	Reference Laboratory	190	1.9	2.2	65	97
24	SB-SO-24-XX	Reference Laboratory	200	2.5	2.3	67	95
24	SB-SO-45-XX	Reference Laboratory	190	2.8	2.1 J-	63	93
24	SB-SO-52-XX	Reference Laboratory	190	1.8	2.2	64	90
24	SB-SO-01-XC	Xcalibur XRF Services	170		16	57	96
24	SB-SO-16-XC	Xcalibur XRF Services	139		6	24	84
24	SB-SO-24-XC	Xcalibur XRF Services	130		22	0	67
24	SB-SO-45-XC	Xcalibur XRF Services	149		4	59	91
24	SB-SO-52-XC	Xcalibur XRF Services	151		20	28	96
25	SB-SO-13-XX	Reference Laboratory	180	4.4	2.2 UJ	74	70
25	SB-SO-19-XX	Reference Laboratory	120	2.5	1.8	51	51
25	SB-SO-33-XX	Reference Laboratory	130	3	2 J	52	56
25	SB-SO-37-XX	Reference Laboratory	150	2.5 U	2 UJ	63	58
25	SB-SO-55-XX	Reference Laboratory	140	2.5	2.2 J	61	60
25	SB-SO-13-XC	Xcalibur XRF Services	125		24	64	56
25	SB-SO-19-XC	Xcalibur XRF Services	121		27	0	57
25	SB-SO-33-XC	Xcalibur XRF Services	100		13	148	57
25	SB-SO-37-XC	Xcalibur XRF Services	132		29	179	68
25	SB-SO-55-XC	Xcalibur XRF Services	128		37	48	57
26	SB-SO-12-XX	Reference Laboratory	110	2.5 U	2.1 UJ	59	42
26	SB-SO-15-XX	Reference Laboratory	100 J-	3.4	1.6 UJ	52 J-	36 J-
26	SB-SO-17-XX	Reference Laboratory	120	2.8	2.3 UJ	60	42
26	SB-SO-46-XX	Reference Laboratory	120	2.6	2.2 UJ	57	41
26	SB-SO-54-XX	Reference Laboratory	20	0.5 U	0.5 UJ	11	6
26	SB-SO-12-XC	Xcalibur XRF Services	122			0	41
26	SB-SO-15-XC	Xcalibur XRF Services	109		23	40	32
26	SB-SO-17-XC	Xcalibur XRF Services	56			0	35
26	SB-SO-46-XC	Xcalibur XRF Services	111		13	33	34
26	SB-SO-54-XC	Xcalibur XRF Services	110			9	37
27	KP-SE-08-XX	Reference Laboratory	42	0.27 U	0.27 UJ	4	5
27	KP-SE-11-XX	Reference Laboratory	46	0.43	0.27 UJ	4	6
27	KP-SE-17-XX	Reference Laboratory	47	0.27 U	0.27 UJ	4	5
27	KP-SE-25-XX	Reference Laboratory	47	0.26 U	0.27 UJ	4	5
27	KP-SE-30-XX	Reference Laboratory	39	0.24 U	0.27 UJ	4	5
27	KP-SE-08-XC	Xcalibur XRF Services	39			26	0
27	KP-SE-11-XC	Xcalibur XRF Services	36		12	0	35
27	KP-SE-17-XC	Xcalibur XRF Services	42	2	5	0	25
27	KP-SE-25-XC	Xcalibur XRF Services	46	41	2	0	25
27	KP-SE-30-XC	Xcalibur XRF Services	47	41		32	18

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
28	KP-SE-01-XX	Reference Laboratory	3.2	2	0.1 U	34	2.2	480	310 J-	0.053 U
28	KP-SE-12-XX	Reference Laboratory	3.1	2	0.1 U	42	2.5	510	320 J-	0.06 U
28	KP-SE-14-XX	Reference Laboratory	11 J-	2	0.1 U	46 J-	2.7 J+	520 J-	680 J-	0.065 U
28	KP-SE-19-XX	Reference Laboratory	3	2	0.1 U	44	2.3	510	330	0.044 U
28	KP-SE-28-XX	Reference Laboratory	3.3	2	0.1 U	45	2.3	520	320	0.056 U
28	KP-SE-01-XC	Xcalibur XRF Services		70	11	72	8	651	405	3
28	KP-SE-12-XC	Xcalibur XRF Services	4	27	26	59	1	509	343	2
28	KP-SE-14-XC	Xcalibur XRF Services	5	107		61	1	635	423	
28	KP-SE-19-XC	Xcalibur XRF Services	13	81		45	1	640	452	2
28	KP-SE-28-XC	Xcalibur XRF Services	5	36		70	1	646	453	
29	TL-SE-04-XX	Reference Laboratory	1.2 U	10	0.5 U	62	1,900	42,000	32	0.26 J-
29	TL-SE-10-XX	Reference Laboratory	1.2 U	10	0.5 U	64	2,000	43,000	35	0.19 J-
29	TL-SE-12-XX	Reference Laboratory	1.2 U	10	0.5 U	66	2,100	44,000	34	0.22 J-
29	TL-SE-15-XX	Reference Laboratory	1.2 U	9	0.5 U	54	1,800	36,000	28	0.28 J-
29	TL-SE-20-XX	Reference Laboratory	1.2 U	10	0.5 U	64	2,000	42,000	32	0.26 J-
29	TL-SE-24-XX	Reference Laboratory	1.2 U	11	0.5 U	67	2,100	43,000	37	0.26 J-
29	TL-SE-26-XX	Reference Laboratory	1.2 U	10	0.5 U	62	2,000	40,000	34	0.24 J-
29	TL-SE-04-XC	Xcalibur XRF Services	0	58			2,586	36,085	98	
29	TL-SE-10-XC	Xcalibur XRF Services	5	0		7	2,606	36,600	144	
29	TL-SE-12-XC	Xcalibur XRF Services	7	53			2,626	36,634	133	
29	TL-SE-15-XC	Xcalibur XRF Services	5	30		36	2,461	35,872	122	
29	TL-SE-20-XC	Xcalibur XRF Services	10	45	12		2,718	37,221	119	
29	TL-SE-24-XC	Xcalibur XRF Services	9	78			2,911	37,288	135	
29	TL-SE-26-XC	Xcalibur XRF Services	16	42		26	2,631	36,523	195	6
30	TL-SE-03-XX	Reference Laboratory	2.5 U	9	1 U	91	1,600	63,000	12	0.32 J-
30	TL-SE-19-XX	Reference Laboratory	2.5 U	10	1 U	96	1,700	66,000	13	0.32 J-
30	TL-SE-23-XX	Reference Laboratory	2.5 U	9	1 U	92	1,600	64,000	12	0.41 J-
30	TL-SE-25-XX	Reference Laboratory	2.5 U	10	1 U	91	1,600	62,000	11	0.44 J-
30	TL-SE-31-XX	Reference Laboratory	2.5 U	10	1 U	110	1,800	74,000	13	0.57 J-
30	TL-SE-03-XC	Xcalibur XRF Services	11	67	12		1,879	52,922	139	
30	TL-SE-19-XC	Xcalibur XRF Services	6	32			2,405	56,855	60	
30	TL-SE-23-XC	Xcalibur XRF Services	10	74			2,272	57,909	106	
30	TL-SE-25-XC	Xcalibur XRF Services	11	27			2,281	58,350	115	
30	TL-SE-31-XC	Xcalibur XRF Services	9	79	16		2,096	55,112	160	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn
28	KP-SE-01-XX	Reference Laboratory	16		0.26	U	0.26	UJ	2	J	6
28	KP-SE-12-XX	Reference Laboratory	20		0.26	U	0.26	UJ	2	J	8
28	KP-SE-14-XX	Reference Laboratory	23	J-	0.26	U	0.26	UJ	3	J	7
28	KP-SE-19-XX	Reference Laboratory	22		0.26	U	0.26	U	2	J	7
28	KP-SE-28-XX	Reference Laboratory	22		0.26	U	0.26	U	2	J	6
28	KP-SE-01-XC	Xcalibur XRF Services	27		11				0		33
28	KP-SE-12-XC	Xcalibur XRF Services	20		47				0		25
28	KP-SE-14-XC	Xcalibur XRF Services	24		27		3		0		30
28	KP-SE-19-XC	Xcalibur XRF Services	23						21		38
28	KP-SE-28-XC	Xcalibur XRF Services	18				3		0		35
29	TL-SE-04-XX	Reference Laboratory	71		1.2	U	1.3		95		160
29	TL-SE-10-XX	Reference Laboratory	72		1.2	U	1.2	U	95		160
29	TL-SE-12-XX	Reference Laboratory	75		1.2	U	1.2	U	100		170
29	TL-SE-15-XX	Reference Laboratory	63		1.2	U	1	U	84		140
29	TL-SE-20-XX	Reference Laboratory	74		1.2	U	1.2	U	100		160
29	TL-SE-24-XX	Reference Laboratory	77		1.2	U	1.3	U	100		170
29	TL-SE-26-XX	Reference Laboratory	70		1.2	U	1.2	U	96		160
29	TL-SE-04-XC	Xcalibur XRF Services	116		5		13		0		183
29	TL-SE-10-XC	Xcalibur XRF Services	115						0		180
29	TL-SE-12-XC	Xcalibur XRF Services	117				15		0		165
29	TL-SE-15-XC	Xcalibur XRF Services	123				25		0		179
29	TL-SE-20-XC	Xcalibur XRF Services	89						0		174
29	TL-SE-24-XC	Xcalibur XRF Services	107				17		0		168
29	TL-SE-26-XC	Xcalibur XRF Services	100						54		192
30	TL-SE-03-XX	Reference Laboratory	110		2.5	U	0.94	U	140		200
30	TL-SE-19-XX	Reference Laboratory	120		2.5	U	1.1	U	150		210
30	TL-SE-23-XX	Reference Laboratory	110		2.5	U	1.3	U	150		200
30	TL-SE-25-XX	Reference Laboratory	110		2.5	U	0.94	U	150		200
30	TL-SE-31-XX	Reference Laboratory	130		2.5	U	1.2	U	170		230
30	TL-SE-03-XC	Xcalibur XRF Services	154				6		0		113
30	TL-SE-19-XC	Xcalibur XRF Services	137				41		0		98
30	TL-SE-23-XC	Xcalibur XRF Services	121				23		0		95
30	TL-SE-25-XC	Xcalibur XRF Services	115		3		54		0		93
30	TL-SE-31-XC	Xcalibur XRF Services	115				15		66		97

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
31	TL-SE-01-XX	Reference Laboratory	1.2 UJ	9	0.5 U	110	1,400	19,000	48 J-	0.074 U
31	TL-SE-11-XX	Reference Laboratory	1.2 UJ	15	0.5 U	140	1,600	28,000	54 J-	0.021 U
31	TL-SE-14-XX	Reference Laboratory	1.2 UJ	10	0.27 J	110	1,500	18,000	50 J-	0.08 U
31	TL-SE-18-XX	Reference Laboratory	1.2 UJ	10	0.5 U	150	1,300	24,000	46 J-	0.025 U
31	TL-SE-22-XX	Reference Laboratory	1.2 UJ	11	0.5 U	150	1,700	26,000	54 J-	0.082 U
31	TL-SE-27-XX	Reference Laboratory	1.2 UJ	10	0.28 J	130	1,500	19,000	51 J-	0.02 U
31	TL-SE-29-XX	Reference Laboratory	1.2 UJ	11	0.22 J	140	1,600	23,000	51 J-	0.076 U
31	TL-SE-01-XC	Xcalibur XRF Services	0	68	19	124	1,462	26,263	154	5
31	TL-SE-11-XC	Xcalibur XRF Services	7	86		102	1,883	34,010	145	5
31	TL-SE-14-XC	Xcalibur XRF Services	3	58		87	1,900	33,304	141	3
31	TL-SE-18-XC	Xcalibur XRF Services	4	51		98	1,876	32,929	193	
31	TL-SE-22-XC	Xcalibur XRF Services	9	37		149	1,865	33,004	181	4
31	TL-SE-27-XC	Xcalibur XRF Services	12	16	13	141	1,932	32,418	187	18
31	TL-SE-29-XC	Xcalibur XRF Services	10	45	1	93	1,809	31,056	119	1
32	LV-SE-02-XX	Reference Laboratory	1.3 UJ	28	0.51 U	72	33	23,000	20 J-	0.02 U
32	LV-SE-10-XX	Reference Laboratory	1.3 UJ	34	0.51 U	84	42	28,000	25 J-	0.023 U
32	LV-SE-22-XX	Reference Laboratory	1.3 UJ	30	0.51 U	69	33	23,000	22 J-	1.1
32	LV-SE-25-XX	Reference Laboratory	1.3 UJ	31	0.51 U	74	36	25,000	23 J-	1
32	LV-SE-31-XX	Reference Laboratory	1.3 UJ	32	0.51 U	78	36	25,000	49 J-	1
32	LV-SE-35-XX	Reference Laboratory	1.3 UJ	31 J-	0.51 U	74 J-	35	24,000 J-	22 J-	1.4
32	LV-SE-50-XX	Reference Laboratory	2.5 U	29	1 U	74	34	24,000	24 J-	1.2
32	LV-SE-02-XC	Xcalibur XRF Services	5	74	33	39	6	19,902	80	13
32	LV-SE-10-XC	Xcalibur XRF Services	9	82	35		30	20,112	0	
32	LV-SE-22-XC	Xcalibur XRF Services	10	81		46	25	19,644	59	7
32	LV-SE-25-XC	Xcalibur XRF Services	5	85	11	44	23	20,308	125	6
32	LV-SE-31-XC	Xcalibur XRF Services	0	87	11		21	19,882	57	
32	LV-SE-35-XC	Xcalibur XRF Services	0	73		49	20	20,470	96	
32	LV-SE-50-XC	Xcalibur XRF Services	0	79	25		5	19,573	69	19
33	LV-SE-12-XX	Reference Laboratory	2.6 U	190	1 U	55	34	72,000	19 J-	5.6
33	LV-SE-26-XX	Reference Laboratory	2.6 U	220	1 U	64	39	83,000	25 J-	6
33	LV-SE-33-XX	Reference Laboratory	2.6 U	170	1 U	52	31	66,000	21 J-	6.8
33	LV-SE-39-XX	Reference Laboratory	2.6 U	190	1 U	58	35	74,000	22 J-	8
33	LV-SE-42-XX	Reference Laboratory	2.7 U	170	1.1 U	50	30	65,000	22 J-	4.3
33	LV-SE-12-XC	Xcalibur XRF Services		230		98	32	51,313	155	
33	LV-SE-26-XC	Xcalibur XRF Services	8	243		133	32	53,305	120	
33	LV-SE-33-XC	Xcalibur XRF Services	7	227			35	53,895	183	
33	LV-SE-39-XC	Xcalibur XRF Services	10	291			43	54,680	0	
33	LV-SE-42-XC	Xcalibur XRF Services	11	261		81	26	54,288	104	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
31	TL-SE-01-XX	Reference Laboratory	180		1.2	U	5.7	J-	75		130	
31	TL-SE-11-XX	Reference Laboratory	210		1.2	U	5.5	J-	85		140	
31	TL-SE-14-XX	Reference Laboratory	180		1.2	U	5.7	J-	73		140	
31	TL-SE-18-XX	Reference Laboratory	190		1.2	U	6.3	J-	70		120	
31	TL-SE-22-XX	Reference Laboratory	210		1.2	U	6.5	J-	80		150	
31	TL-SE-27-XX	Reference Laboratory	200		1.2	U	7.8	J-	67		140	
31	TL-SE-29-XX	Reference Laboratory	200		1.2	U	5.9	J-	80		140	
31	TL-SE-01-XC	Xcalibur XRF Services	156						0		97	
31	TL-SE-11-XC	Xcalibur XRF Services	220				33		0		104	
31	TL-SE-14-XC	Xcalibur XRF Services	198				21		64		107	
31	TL-SE-18-XC	Xcalibur XRF Services	199				24		54		93	
31	TL-SE-22-XC	Xcalibur XRF Services	206				26		0		107	
31	TL-SE-27-XC	Xcalibur XRF Services	232		64		23		65		109	
31	TL-SE-29-XC	Xcalibur XRF Services	209		37		26		44		104	
32	LV-SE-02-XX	Reference Laboratory	160		3.8		1.3	UJ	53		65	
32	LV-SE-10-XX	Reference Laboratory	200		4.7		1.3	UJ	66		77	
32	LV-SE-22-XX	Reference Laboratory	170		5.2		1.3	UJ	51		66	
32	LV-SE-25-XX	Reference Laboratory	170		5.1		1.3	UJ	56		70	
32	LV-SE-31-XX	Reference Laboratory	180		5.1		1.3	UJ	58		70	
32	LV-SE-35-XX	Reference Laboratory	170	J-	5		1.3	UJ	55	J-	67	J-
32	LV-SE-50-XX	Reference Laboratory	170		3.3		2.5	U	57		65	
32	LV-SE-02-XC	Xcalibur XRF Services	115				11		55		115	
32	LV-SE-10-XC	Xcalibur XRF Services	119				6		73		97	
32	LV-SE-22-XC	Xcalibur XRF Services	106				7		23		127	
32	LV-SE-25-XC	Xcalibur XRF Services	141				10		0		124	
32	LV-SE-31-XC	Xcalibur XRF Services	111				8		0		113	
32	LV-SE-35-XC	Xcalibur XRF Services	116				2		45		91	
32	LV-SE-50-XC	Xcalibur XRF Services	113				21		82		100	
33	LV-SE-12-XX	Reference Laboratory	71		3		2.6	U	72		66	
33	LV-SE-26-XX	Reference Laboratory	83		6.1		2.6	U	86		75	
33	LV-SE-33-XX	Reference Laboratory	66		2.8		2.6	U	67		59	
33	LV-SE-39-XX	Reference Laboratory	74		5.1		2.6	U	74		66	
33	LV-SE-42-XX	Reference Laboratory	67		3.4		2.7	U	64		57	
33	LV-SE-12-XC	Xcalibur XRF Services	71						25		48	
33	LV-SE-26-XC	Xcalibur XRF Services	72				3		27		37	
33	LV-SE-33-XC	Xcalibur XRF Services	72				4		0		40	
33	LV-SE-39-XC	Xcalibur XRF Services	102		22		5		0		31	
33	LV-SE-42-XC	Xcalibur XRF Services	99				2		0		41	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
34	LV-SE-09-XX	Reference Laboratory	6.7 U	450	2.7 U	48	34	150,000	14 J-	6
34	LV-SE-19-XX	Reference Laboratory	6.7 U	500	2.7 U	55	37	160,000	17 J-	7.2
34	LV-SE-27-XX	Reference Laboratory	6.7 U	530	2.7 U	56	39	180,000	16 J-	11
34	LV-SE-36-XX	Reference Laboratory	6.7 U	550	2.7 U	60	40	180,000	21 J-	8.5
34	LV-SE-38-XX	Reference Laboratory	6.7 U	480	2.7 U	52	36	160,000	15 J-	7.9
34	LV-SE-09-XC	Xcalibur XRF Services	0	513		270	40	105,797	0	
34	LV-SE-19-XC	Xcalibur XRF Services	11	517	24	384	2	104,569	166	
34	LV-SE-27-XC	Xcalibur XRF Services	10	488	18	86	3	103,211	257	
34	LV-SE-36-XC	Xcalibur XRF Services	15	545			36	107,069	0	
34	LV-SE-38-XC	Xcalibur XRF Services	0	503	0	92	4	105,744	0	
35	LV-SE-07-XX	Reference Laboratory	6.7 UJ	780	2.7 U	57	48	200,000	11	5.5
35	LV-SE-18-XX	Reference Laboratory	6.7 UJ	800	2.7 U	61	49	210,000	11	5.4
35	LV-SE-23-XX	Reference Laboratory	6.6 UJ	660	2.6 U	53	40	170,000	8	5
35	LV-SE-45-XX	Reference Laboratory	6.7 UJ	650	2.7 U	50	40	170,000	8	5.6
35	LV-SE-48-XX	Reference Laboratory	6.6 UJ	680	2.6 U	52	42	180,000	9	7.3
35	LV-SE-07-XC	Xcalibur XRF Services	12	692	12	314	47	132,559	270	
35	LV-SE-18-XC	Xcalibur XRF Services	11	670		702	2	133,997	356	
35	LV-SE-23-XC	Xcalibur XRF Services	22	705	9	346	28	129,948	192	
35	LV-SE-45-XC	Xcalibur XRF Services	35	642			54	120,254	0	
35	LV-SE-48-XC	Xcalibur XRF Services	28	585			1	124,294	445	109
36	LV-SE-01-XX	Reference Laboratory	1.5 UJ	6	0.76	4	18	1,100	17	0.098 U
36	LV-SE-14-XX	Reference Laboratory	1.5 UJ	5	0.74	4	16	980	14	0.056 U
36	LV-SE-21-XX	Reference Laboratory	1.5 UJ	7	0.84	4	19	970	18	0.048 U
36	LV-SE-24-XX	Reference Laboratory	1.5 UJ	5	0.68	4	15	840	14	0.053 U
36	LV-SE-32-XX	Reference Laboratory	1.4 UJ	6	0.87	4	16	860	14	0.052 U
36	LV-SE-01-XC	Xcalibur XRF Services	118	91	28		1	416	43	3
36	LV-SE-14-XC	Xcalibur XRF Services	80	70			14	446	22	
36	LV-SE-21-XC	Xcalibur XRF Services	98	72			0	455	22	
36	LV-SE-24-XC	Xcalibur XRF Services	98	66			0	437	27	2
36	LV-SE-32-XC	Xcalibur XRF Services	107	73			1	433	32	
37	LV-SE-08-XX	Reference Laboratory	1.3 UJ	30	0.52 U	54	23	23,000	55	5.2
37	LV-SE-16-XX	Reference Laboratory	1.3 UJ	29	0.52 U	53	22	22,000	53	5.4
37	LV-SE-28-XX	Reference Laboratory	1.3 UJ	31	0.52 U	59	25	25,000	59	5.4
37	LV-SE-30-XX	Reference Laboratory	1.3 UJ	30	0.52 U	58	25	24,000	58	6.3
37	LV-SE-47-XX	Reference Laboratory	1.3 UJ	31	0.52 U	56	23	23,000	57	4.9
37	LV-SE-08-XC	Xcalibur XRF Services	0	69	36		6	20,743	137	8
37	LV-SE-16-XC	Xcalibur XRF Services	1	83	52		5	20,064	177	12
37	LV-SE-28-XC	Xcalibur XRF Services	1	71		40	5	20,981	165	
37	LV-SE-30-XC	Xcalibur XRF Services	6	57	34	58	5	20,597	193	7
37	LV-SE-47-XC	Xcalibur XRF Services	0	93			23	19,257	145	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
34	LV-SE-09-XX	Reference Laboratory	55	6.7 U	6.7 U	100	51 J
34	LV-SE-19-XX	Reference Laboratory	65	5.9 J	6.7 U	110	55 J
34	LV-SE-27-XX	Reference Laboratory	64	6.7 U	6.7 U	120	58 J
34	LV-SE-36-XX	Reference Laboratory	70	11	6.7 U	120	60 J
34	LV-SE-38-XX	Reference Laboratory	75	6.7 U	6.7 U	100	54 J
34	LV-SE-09-XC	Xcalibur XRF Services	74		13	0	
34	LV-SE-19-XC	Xcalibur XRF Services	74			0	
34	LV-SE-27-XC	Xcalibur XRF Services	75	111	14	0	
34	LV-SE-36-XC	Xcalibur XRF Services	73		10	0	
34	LV-SE-38-XC	Xcalibur XRF Services	74	10		0	
35	LV-SE-07-XX	Reference Laboratory	58	10	6.7 U	130	24 J
35	LV-SE-18-XX	Reference Laboratory	60	12	6.7 U	140	52 J
35	LV-SE-23-XX	Reference Laboratory	50 J	9.6	6.6 U	120	18 J
35	LV-SE-45-XX	Reference Laboratory	50 J	8.2	6.7 U	120	19 J
35	LV-SE-48-XX	Reference Laboratory	50 J	7.6	6.6 U	120	30 J
35	LV-SE-07-XC	Xcalibur XRF Services	55			0	
35	LV-SE-18-XC	Xcalibur XRF Services	54			0	
35	LV-SE-23-XC	Xcalibur XRF Services	57	5		0	
35	LV-SE-45-XC	Xcalibur XRF Services	65	9		15	0
35	LV-SE-48-XC	Xcalibur XRF Services	111			0	
36	LV-SE-01-XX	Reference Laboratory	49	1.5 U	1.5 U	2 J	14 J
36	LV-SE-14-XX	Reference Laboratory	46	1.5 U	1.5 U	1 J	12 J
36	LV-SE-21-XX	Reference Laboratory	49	1.5 U	1.5 U	2 J	14 J
36	LV-SE-24-XX	Reference Laboratory	44	1.5 U	1.5 U	1 J	12 J
36	LV-SE-32-XX	Reference Laboratory	47	1.4 U	1.4 U	1 J	19
36	LV-SE-01-XC	Xcalibur XRF Services	21		9	0	22
36	LV-SE-14-XC	Xcalibur XRF Services	25			0	53
36	LV-SE-21-XC	Xcalibur XRF Services	17			0	0
36	LV-SE-24-XC	Xcalibur XRF Services	31		4	0	0
36	LV-SE-32-XC	Xcalibur XRF Services	15		19	0	29
37	LV-SE-08-XX	Reference Laboratory	110	4.8	1.3 U	44	61
37	LV-SE-16-XX	Reference Laboratory	110	5	1.3 U	42	59
37	LV-SE-28-XX	Reference Laboratory	120	5.8	1.3 U	48	65
37	LV-SE-30-XX	Reference Laboratory	120	5.6	1.3 U	48	66
37	LV-SE-47-XX	Reference Laboratory	120	4.2	1.3 U	45	65
37	LV-SE-08-XC	Xcalibur XRF Services	122	6	4	46	116
37	LV-SE-16-XC	Xcalibur XRF Services	84		0	32	103
37	LV-SE-28-XC	Xcalibur XRF Services	102		9	16	100
37	LV-SE-30-XC	Xcalibur XRF Services	108		15	36	92
37	LV-SE-47-XC	Xcalibur XRF Services	96		2	32	107

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg	Ni
38	LV-SE-11-XX	Reference Laboratory	1.4 UJ	150	6.6	120	270	42,000	7	2.8	870
38	LV-SE-29-XX	Reference Laboratory	1.4 UJ	150	6.3	120	260	42,000	7 J+	1.5 J-	860
38	LV-SE-44-XX	Reference Laboratory	1.4 U	140	6.1	120	250	40,000	8	1.5	830
38	LV-SE-46-XX	Reference Laboratory	0.88 U	110	5	92	200	32,000	6	1.4	660
38	LV-SE-52-XX	Reference Laboratory	1.4 U	160	6.8	130	280	44,000	8	21	910
38	LV-SE-11-XC	Xcalibur XRF Services	17	218		57	212	30,708	0		608
38	LV-SE-29-XC	Xcalibur XRF Services	13	228			202	30,725	0		636
38	LV-SE-44-XC	Xcalibur XRF Services		195			176	26,884			508
38	LV-SE-46-XC	Xcalibur XRF Services	9	199		41	200	27,483	0		567
38	LV-SE-52-XC	Xcalibur XRF Services	11	167			186	29,383	96		551
39	RF-SE-07-XX	Reference Laboratory	1.3 U	12	0.5 U	92	81	17,000	24	0.091 U	180
39	RF-SE-12-XX	Reference Laboratory	1.2 U	14	0.5 U	100	110	20,000	25	0.099 U	210
39	RF-SE-23-XX	Reference Laboratory	0.25 U	0 U	0.1 U	0 U	0.2 U	4 J	0 U	2.4	2 U
39	RF-SE-36-XX	Reference Laboratory	1.2 U	12	0.5 U	91	82	17,000	22	0.081 U	180
39	RF-SE-42-XX	Reference Laboratory	1.3 UJ	14	0.56	110	95	19,000	28	0.084 U	210
39	RF-SE-45-XX	Reference Laboratory	1.3 UJ	15	0.52 U	110	100	21,000	33	0.084 U	220
39	RF-SE-53-XX	Reference Laboratory	1.3 UJ	14	0.57 U	110	95	19,000	28	0.084 U	210
39	RF-SE-07-XC	Xcalibur XRF Services	0	63	10	48	56	15,548	108		132
39	RF-SE-12-XC	Xcalibur XRF Services	0	72		70	52	16,025	96	7	135
39	RF-SE-23-XC	Xcalibur XRF Services	0	54		80	72	16,718	97	7	155
39	RF-SE-36-XC	Xcalibur XRF Services	0	63		88	67	15,862	56	4	124
39	RF-SE-42-XC	Xcalibur XRF Services	4	34		51	75	17,031	60	8	165
39	RF-SE-45-XC	Xcalibur XRF Services	9	64	11	118	59	16,455	119	4	141
39	RF-SE-53-XC	Xcalibur XRF Services	0	73	30	70	55	16,043		86	144
40	RF-SE-03-XX	Reference Laboratory	1.2 UJ	27	1.3	93	200	17,000	88	0.48	150
40	RF-SE-28-XX	Reference Laboratory	1.2 UJ	31	1.5	100	220	18,000	99	0.57	160
40	RF-SE-38-XX	Reference Laboratory	1.2 UJ	27	1.2	90	190	16,000	83	0.41	140
40	RF-SE-49-XX	Reference Laboratory	1.2 UJ	31	1.5	100	220	18,000	97	0.43	170
40	RF-SE-55-XX	Reference Laboratory	1.2 UJ	24	1.1	91	180	15,000	75	0.42	140
40	RF-SE-03-XC	Xcalibur XRF Services	1	80	51		210	15,187	188	9	117
40	RF-SE-28-XC	Xcalibur XRF Services	5	53		106	211	15,903	204	7	116
40	RF-SE-38-XC	Xcalibur XRF Services	10	65	12	80	228	16,972	197		123
40	RF-SE-49-XC	Xcalibur XRF Services	1	64		73	200	15,568	178		117
40	RF-SE-55-XC	Xcalibur XRF Services	1	99		41	209	16,258	219	8	118

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
38	LV-SE-11-XX	Reference Laboratory	870	1.3 U	1.4 U	35	200
38	LV-SE-29-XX	Reference Laboratory	860	1.2 U	1.4 U	35	200
38	LV-SE-44-XX	Reference Laboratory	830	1.4 U	1.4 U	34	190
38	LV-SE-46-XX	Reference Laboratory	660	0.88 U	0.88 U	27	150
38	LV-SE-52-XX	Reference Laboratory	910	1.4 U	1.4 U	38	210
38	LV-SE-11-XC	Xcalibur XRF Services	608		7	0	191
38	LV-SE-29-XC	Xcalibur XRF Services	636		0	0	202
38	LV-SE-44-XC	Xcalibur XRF Services	508			0	202
38	LV-SE-46-XC	Xcalibur XRF Services	567			0	189
38	LV-SE-52-XC	Xcalibur XRF Services	551		0	61	187
39	RF-SE-07-XX	Reference Laboratory	180	1.3 U	1.3 U	34	130
39	RF-SE-12-XX	Reference Laboratory	210	1.2 U	1.2 U	38	140
39	RF-SE-23-XX	Reference Laboratory	2 U	0.25 U	0.37	3 U	1 U
39	RF-SE-36-XX	Reference Laboratory	180	1 U	1.2 U	34	120
39	RF-SE-42-XX	Reference Laboratory	210	1.3 U	1.3 U	40	140
39	RF-SE-45-XX	Reference Laboratory	220	1.3 U	1.3 U	43	150
39	RF-SE-53-XX	Reference Laboratory	210	1.3 U	1.3 U	40	140
39	RF-SE-07-XC	Xcalibur XRF Services	132		25	0	171
39	RF-SE-12-XC	Xcalibur XRF Services	135		4	35	190
39	RF-SE-23-XC	Xcalibur XRF Services	155	6		49	219
39	RF-SE-36-XC	Xcalibur XRF Services	124		4	0	227
39	RF-SE-42-XC	Xcalibur XRF Services	165	14		33	248
39	RF-SE-45-XC	Xcalibur XRF Services	141		14	0	217
39	RF-SE-53-XC	Xcalibur XRF Services	144		3	26	185
40	RF-SE-03-XX	Reference Laboratory	150	1.2 U	1.2 U	40	300
40	RF-SE-28-XX	Reference Laboratory	160	1.2 U	1.2 U	44	320
40	RF-SE-38-XX	Reference Laboratory	140	1.2 U	1.2 U	39	300
40	RF-SE-49-XX	Reference Laboratory	170	1.2 U	1.2 U	43	330
40	RF-SE-55-XX	Reference Laboratory	140	1.2 U	1.2 U	35	280
40	RF-SE-03-XC	Xcalibur XRF Services	117		6	42	481
40	RF-SE-28-XC	Xcalibur XRF Services	116			61	486
40	RF-SE-38-XC	Xcalibur XRF Services	123		3	0	552
40	RF-SE-49-XC	Xcalibur XRF Services	117		8	9	530
40	RF-SE-55-XC	Xcalibur XRF Services	118			0	534

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
41	RF-SE-06-XX	Reference Laboratory	1.3 UJ	70	3.6	90	490	20,000	230	1.1
41	RF-SE-13-XX	Reference Laboratory	1.3 UJ	76	3.7	92	530	21,000	230	1.2
41	RF-SE-27-XX	Reference Laboratory	1.3 UJ	64	3.1	78	440	18,000	200	1.2
41	RF-SE-31-XX	Reference Laboratory	1.3 UJ	39	1.8	63	250	12,000	120	1.1
41	RF-SE-58-XX	Reference Laboratory	1.3 UJ	71	3.6	89	500	21,000	230	1.2
41	RF-SE-06-XC	Xcalibur XRF Services	2	96			468	16,459	356	
41	RF-SE-13-XC	Xcalibur XRF Services	7	92	22	78	485	16,918	452	
41	RF-SE-27-XC	Xcalibur XRF Services		92	2	97	485	17,224	381	5
41	RF-SE-31-XC	Xcalibur XRF Services	2	123		59	523	17,807	392	
41	RF-SE-58-XC	Xcalibur XRF Services	6	89			520	17,583	501	
42	RF-SE-02-XX	Reference Laboratory	1.3 UJ	110	5.4	93	740	24,000	330	1.6
42	RF-SE-22-XX	Reference Laboratory	1.3 UJ	99	4.7	84	670	22,000	300	1.7
42	RF-SE-25-XX	Reference Laboratory	1.3 UJ	88	4	78	580	19,000	270	1.5
42	RF-SE-30-XX	Reference Laboratory	1.3 UJ	89	4.3	78	610	21,000	290	1.5
42	RF-SE-57-XX	Reference Laboratory	1.3 UJ	89	4.5	79	610	21,000	300	1.5
42	RF-SE-02-XC	Xcalibur XRF Services	2	143	4	58	599	17,633	478	
42	RF-SE-22-XC	Xcalibur XRF Services	10	179		79	649	17,877	467	7
42	RF-SE-25-XC	Xcalibur XRF Services	2	107	22	80	649	18,000	497	
42	RF-SE-30-XC	Xcalibur XRF Services	7	125	25	101	637	18,543	542	
42	RF-SE-57-XC	Xcalibur XRF Services	2	123			637	17,762	505	
43	RF-SE-15-XX	Reference Laboratory	1.3 UJ	120	6.2	72	820	23,000	390	2.6
43	RF-SE-24-XX	Reference Laboratory	1.3 UJ	130 J+	6.5 J+	74 J+	860 J+	24,000 J+	410 J+	2.3
43	RF-SE-32-XX	Reference Laboratory	1.3 UJ	120	5.1	64	770	20,000	330	2.8
43	RF-SE-43-XX	Reference Laboratory	1.3 UJ	130	5.7	68	840	22,000	350	2.7
43	RF-SE-59-XX	Reference Laboratory	1.3 UJ	140	5.9	73	890	23,000	380	0.085 U
43	RF-SE-15-XC	Xcalibur XRF Services	6	180		49	887	19,197	661	8
43	RF-SE-24-XC	Xcalibur XRF Services	3	207		75	889	19,447	654	
43	RF-SE-32-XC	Xcalibur XRF Services	3	148	21		901	19,415	703	
43	RF-SE-43-XC	Xcalibur XRF Services	10	168	38		938	19,857	678	
43	RF-SE-59-XC	Xcalibur XRF Services	12	152		36	928	19,383	686	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
41	RF-SE-06-XX	Reference Laboratory	150		1.3	U	1.3	U	44		740	
41	RF-SE-13-XX	Reference Laboratory	160		1.3	U	1.3		45		790	
41	RF-SE-27-XX	Reference Laboratory	130		1.3	U	1.3	U	39		670	
41	RF-SE-31-XX	Reference Laboratory	86		1.3	U	1.3	U	28		420	
41	RF-SE-58-XX	Reference Laboratory	150		1.3	U	1.3	U	46		770	
41	RF-SE-06-XC	Xcalibur XRF Services	108				24		50		1,092	
41	RF-SE-13-XC	Xcalibur XRF Services	122				22		0		1,173	
41	RF-SE-27-XC	Xcalibur XRF Services	100		24				14		1,090	
41	RF-SE-31-XC	Xcalibur XRF Services	115				5		33		1,156	
41	RF-SE-58-XC	Xcalibur XRF Services	107				25		23		1,231	
42	RF-SE-02-XX	Reference Laboratory	180		1.3	U	2.7		50		1,100	
42	RF-SE-22-XX	Reference Laboratory	160		1.3	U	2.3		44		990	
42	RF-SE-25-XX	Reference Laboratory	140		1.5		1.7		40		890	
42	RF-SE-30-XX	Reference Laboratory	150		1.3	U	1.9		44		960	
42	RF-SE-57-XX	Reference Laboratory	150		2		2.2		44		1,000	
42	RF-SE-02-XC	Xcalibur XRF Services	105				9		0		1,295	
42	RF-SE-22-XC	Xcalibur XRF Services	150		4				0		1,445	
42	RF-SE-25-XC	Xcalibur XRF Services	140		23				0		1,371	
42	RF-SE-30-XC	Xcalibur XRF Services	131				10		0		1,452	
42	RF-SE-57-XC	Xcalibur XRF Services	112				3		0		1,465	
43	RF-SE-15-XX	Reference Laboratory	160		1.4		3.6		45		1,300	
43	RF-SE-24-XX	Reference Laboratory	170	J+	1.3	U	3.8	J+	46	J+	1,400	J-
43	RF-SE-32-XX	Reference Laboratory	140		1.3	U	4.2		36		1,100	
43	RF-SE-43-XX	Reference Laboratory	150		1.3	U	4		40		1,200	
43	RF-SE-59-XX	Reference Laboratory	160		1.3	U	4.5		42		1,300	
43	RF-SE-15-XC	Xcalibur XRF Services	125				4		0		1,768	
43	RF-SE-24-XC	Xcalibur XRF Services	140		14				22		1,861	
43	RF-SE-32-XC	Xcalibur XRF Services	127				16		0		1,857	
43	RF-SE-43-XC	Xcalibur XRF Services	127				19		40		1,939	
43	RF-SE-59-XC	Xcalibur XRF Services	136				22		48		1,868	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
44	RF-SE-05-XX	Reference Laboratory	4.1 J+	160	9.1	69	1,000	26,000	450	2.6
44	RF-SE-26-XX	Reference Laboratory	2.2 J+	140	8.4	64	990	23,000	440	2.5
44	RF-SE-39-XX	Reference Laboratory	2.9 J+	160	9.3	73	1,100	26,000	490	2.2
44	RF-SE-44-XX	Reference Laboratory	2.7 J+	140	8.2	64	970	24,000	420	2.3
44	RF-SE-56-XX	Reference Laboratory	3.5 J+	180	9.6	75	1200	27,000	490	2.2
44	RF-SE-05-XC	Xcalibur XRF Services	3	163	15	40	1,115	20,304	729	7
44	RF-SE-26-XC	Xcalibur XRF Services	9	225	30	43	1,133	20,882	683	
44	RF-SE-39-XC	Xcalibur XRF Services	12	209	24	86	1,157	21,647	769	
44	RF-SE-44-XC	Xcalibur XRF Services	7	231		46	1,171	21,394	657	5
44	RF-SE-56-XC	Xcalibur XRF Services	12	212		45	1,138	20,900	694	
45	RF-SE-04-XX	Reference Laboratory	3.2 J+	230	12	42	1,500	27,000	730	4.2
45	RF-SE-14-XX	Reference Laboratory	4.4 J+	260	12	47	1,700	30,000	800	4.7
45	RF-SE-19-XX	Reference Laboratory	3.7 J+	250	13	48	1,700	30,000	800	3.9
45	RF-SE-34-XX	Reference Laboratory	2.9 J+	210	10	39	1,400	24,000	660	4.5
45	RF-SE-52-XX	Reference Laboratory	3.4 J+	220	11	42	1,500	26,000	720	4.1
45	RF-SE-04-XC	Xcalibur XRF Services	4	303			1,637	22,522	1,023	
45	RF-SE-14-XC	Xcalibur XRF Services	5	274	27	51	1,635	23,684	1,118	
45	RF-SE-19-XC	Xcalibur XRF Services	15	284		47	1,673	22,966	1,145	
45	RF-SE-34-XC	Xcalibur XRF Services	17	343	12		1,727	23,564	1,069	
45	RF-SE-52-XC	Xcalibur XRF Services	16	311	25	49	1,720	23,725		1,100
46	BN-SO-11-XX	Reference Laboratory	4 J-	2,900	720	820	120	23,000	56	24 J-
46	BN-SO-14-XX	Reference Laboratory	3.5 J-	2,800	690	800	120	22,000	51	26
46	BN-SO-23-XX	Reference Laboratory	1.2 UJ	2,800	700	800	120	23,000	52	31
46	BN-SO-11-XC	Xcalibur XRF Services	5	4,644	951	519	431	24,935	0	25
46	BN-SO-14-XC	Xcalibur XRF Services	8	4,470	850	507	406	24,994	61	43
46	BN-SO-23-XC	Xcalibur XRF Services	0	4,327	818	533	423	25,464	77	37
47	BN-SO-09-XX	Reference Laboratory	750 J-	97	2,700	2,900	100	22,000	4,700	0.39
47	BN-SO-12-XX	Reference Laboratory	750 J-	89	2,600	2,800	96	21,000	4,500	0.34
47	BN-SO-24-XX	Reference Laboratory	810 J-	97	2,900	3,000	100	23,000	4,900	0.37
47	BN-SO-09-XC	Xcalibur XRF Services	374	60	3,128	1,613	76	15,548	7,409	
47	BN-SO-12-XC	Xcalibur XRF Services	366	52	3,078	1,605	67	15,511	7,237	
47	BN-SO-24-XC	Xcalibur XRF Services	348	70	2,870	1,563	75	15,803	7,395	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag		V	Zn
44	RF-SE-05-XX	Reference Laboratory	150	3.1	7.4	J-	48	1,800
44	RF-SE-26-XX	Reference Laboratory	140	2.8	7.2	J-	42	1,700
44	RF-SE-39-XX	Reference Laboratory	150	2.6	8.2	J-	49	1,900
44	RF-SE-44-XX	Reference Laboratory	140	2.4	7.2	J-	44	1,600
44	RF-SE-56-XX	Reference Laboratory	160	1.8	8.3	J-	51	1,900
44	RF-SE-05-XC	Xcalibur XRF Services	105		11		50	2,313
44	RF-SE-26-XC	Xcalibur XRF Services	107	1			0	2,307
44	RF-SE-39-XC	Xcalibur XRF Services	94		17		12	2,279
44	RF-SE-44-XC	Xcalibur XRF Services	107		6		0	2,351
44	RF-SE-56-XC	Xcalibur XRF Services	112		7		40	2,222
45	RF-SE-04-XX	Reference Laboratory	130	2.8	12	J-	46	2,400
45	RF-SE-14-XX	Reference Laboratory	140	3	13	J-	51	2,600
45	RF-SE-19-XX	Reference Laboratory	140	4.1	14	J-	52	2,700
45	RF-SE-34-XX	Reference Laboratory	120	1.9	10	J-	42	2,200
45	RF-SE-52-XX	Reference Laboratory	130	2	11	J-	47	2,300
45	RF-SE-04-XC	Xcalibur XRF Services	117		42		48	2,884
45	RF-SE-14-XC	Xcalibur XRF Services	105		25		34	3,004
45	RF-SE-19-XC	Xcalibur XRF Services	126		33		0	2,922
45	RF-SE-34-XC	Xcalibur XRF Services	125		12		0	2,971
45	RF-SE-52-XC	Xcalibur XRF Services	137		9		0	2,916
46	BN-SO-11-XX	Reference Laboratory	2,900	140	140	J-	150	3,900
46	BN-SO-14-XX	Reference Laboratory	2,800	130	140	J-	150	3,800
46	BN-SO-23-XX	Reference Laboratory	2,800	130	130	J-	150	3,800
46	BN-SO-11-XC	Xcalibur XRF Services	2,533	260	166		48	5,392
46	BN-SO-14-XC	Xcalibur XRF Services	2,429	208	142		75	5,294
46	BN-SO-23-XC	Xcalibur XRF Services	2,460	206	135		76	5,306
47	BN-SO-09-XX	Reference Laboratory	1,500	290	100	J-	340	81
47	BN-SO-12-XX	Reference Laboratory	1,400	290	210	J-	310	74
47	BN-SO-24-XX	Reference Laboratory	1,600	300	140	J-	350	81
47	BN-SO-09-XC	Xcalibur XRF Services	1,158	717	520		99	104
47	BN-SO-12-XC	Xcalibur XRF Services	1,194	624	512		40	110
47	BN-SO-24-XC	Xcalibur XRF Services	1,169	611	447		75	116

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
48	SB-SO-09-XX	Reference Laboratory	1.3 UJ	9	0.51 U	130	120	35,000	19	30
48	SB-SO-20-XX	Reference Laboratory	1.3 UJ	11	0.51 U	170	150	44,000	24	10
48	SB-SO-31-XX	Reference Laboratory	1.3 UJ	8 J-	0.51 U	140	130	38,000	21	32
48	SB-SO-09-XC	Xcalibur XRF Services	3	67	10	160	414	38,474	32	34
48	SB-SO-20-XC	Xcalibur XRF Services	11	62	13	64	379	32,246	0	
48	SB-SO-31-XC	Xcalibur XRF Services	0	68		104	393	35,657	0	
49	SB-SO-29-XX	Reference Laboratory	1.2 U	9	0.5 U	140	130	41,000	19	7.9 J
49	SB-SO-36-XX	Reference Laboratory	1.2 U	8	0.5 U	120	100	33,000	15	36
49	SB-SO-56-XX	Reference Laboratory	1.2 U	10	0.5 U	150	140	42,000	20	9
49	SB-SO-29-XC	Xcalibur XRF Services	0	74	9	75	408	36,179	41	15
49	SB-SO-36-XC	Xcalibur XRF Services	0	52	28	150	424	37,462	55	19
49	SB-SO-56-XC	Xcalibur XRF Services	0	95	17	144	419	38,650	40	26
50	SB-SO-04-XX	Reference Laboratory	940	13	2,800	2,800	100	38,000	21	40
50	SB-SO-34-XX	Reference Laboratory	980	12	2,500	2,500	91	34,000	18	36
50	SB-SO-49-XX	Reference Laboratory	700	12	2,500	2,400	89	33,000	18	36
50	SB-SO-04-XC	Xcalibur XRF Services	522	121	3,100	1,800	80	24,853	68	34
50	SB-SO-34-XC	Xcalibur XRF Services		121	2,907	1,691	72	23,843	20	484
50	SB-SO-49-XC	Xcalibur XRF Services	528	85	3,174	1,770	71	24,585	51	44
51	WS-SO-07-XX	Reference Laboratory	3.8	53	1.9	640	4,400	25,000	1,700	0.26
51	WS-SO-11-XX	Reference Laboratory	1.2 U	46	1.4	570	3,900	19,000	1,500	0.27
51	WS-SO-25-XX	Reference Laboratory	1.2 U	59	3.1	730	4,900	24,000	1,900	0.25
51	WS-SO-07-XC	Xcalibur XRF Services	16	95		499	9,740	20,630	5,119	
51	WS-SO-11-XC	Xcalibur XRF Services	17	138		476	9,332	19,876	4,887	
51	WS-SO-25-XC	Xcalibur XRF Services	13	44		379	6,663	16,469	3,193	9
52	WS-SO-10-XX	Reference Laboratory	1.3 U	83	1.8	67	76	19,000	1,900	0.058 U
52	WS-SO-20-XX	Reference Laboratory	1.3 U	100	1.9	81	90	23,000	2,300	0.06 U
52	WS-SO-23-XX	Reference Laboratory	1.3 U	110	2.1	82	96	23,000	2,500	0.05 U
52	WS-SO-10-XC	Xcalibur XRF Services	22	80	11	93	286	26,434	5,426	
52	WS-SO-20-XC	Xcalibur XRF Services	23	56	40	52	288	25,411	4,817	20
52	WS-SO-23-XC	Xcalibur XRF Services	21	44	50	80	271	24,797	4,721	18
53	AS-SO-03-XX	Reference Laboratory	1.2 U	14	1,300	33	6,200	15,000	160	3.7 J-
53	AS-SO-05-XX	Reference Laboratory	1.2 U	9	900	23	4,500	11,000	110	2.5 J-
53	AS-SO-08-XX	Reference Laboratory	1.2 U	10	930	24	4,600	11,000	120	2.5 J-
53	AS-SO-03-XC	Xcalibur XRF Services	4	101	1,167	59	8,843	11,957	208	
53	AS-SO-05-XC	Xcalibur XRF Services	1	67	1,184		9,977	12,322	211	
53	AS-SO-08-XC	Xcalibur XRF Services	1	55	1,217		9,488	12,255	218	5

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
48	SB-SO-09-XX	Reference Laboratory	2900	26	160 J-	120	3,600
48	SB-SO-20-XX	Reference Laboratory	3700	30	140 J-	160	4,500
48	SB-SO-31-XX	Reference Laboratory	3200 J-	28 J-	160 J-	140	3,900 J-
48	SB-SO-09-XC	Xcalibur XRF Services	2,715	28	387	41	4,097
48	SB-SO-20-XC	Xcalibur XRF Services	2,375	31	300	0	3,934
48	SB-SO-31-XC	Xcalibur XRF Services	2,499	21	335	144	4,005
49	SB-SO-29-XX	Reference Laboratory	200	160	1.2 UJ	400	3,900
49	SB-SO-36-XX	Reference Laboratory	160	130	1.2 UJ	320	3,200
49	SB-SO-56-XX	Reference Laboratory	210	160	1.2 UJ	410	4,100
49	SB-SO-29-XC	Xcalibur XRF Services	146	381	3	199	4,175
49	SB-SO-36-XC	Xcalibur XRF Services	166	369	4	225	4,103
49	SB-SO-56-XC	Xcalibur XRF Services	169	383	5	97	4,111
50	SB-SO-04-XX	Reference Laboratory	3,300	390	1.3 UJ	58	86
50	SB-SO-34-XX	Reference Laboratory	3,000	360	1.3 UJ	52	77
50	SB-SO-49-XX	Reference Laboratory	2,800	330	1.2 UJ	52	72
50	SB-SO-04-XC	Xcalibur XRF Services	2,718	912		0	70
50	SB-SO-34-XC	Xcalibur XRF Services	2,565	855		76	62
50	SB-SO-49-XC	Xcalibur XRF Services	2,648	960	65	96	63
51	WS-SO-07-XX	Reference Laboratory	260	1.2 U	400 J-	48	180
51	WS-SO-11-XX	Reference Laboratory	240	1.2 U	340 J-	43	160
51	WS-SO-25-XX	Reference Laboratory	300	1.2 U	450 J-	54	200
51	WS-SO-07-XC	Xcalibur XRF Services	437	9	542	39	306
51	WS-SO-11-XC	Xcalibur XRF Services	420		537	9	327
51	WS-SO-25-XC	Xcalibur XRF Services	310		378	0	279
52	WS-SO-10-XX	Reference Laboratory	290	280	1.3 UJ	260	1,900
52	WS-SO-20-XX	Reference Laboratory	350	340	1.3 UJ	320	2,300
52	WS-SO-23-XX	Reference Laboratory	380	360	1.3 UJ	330	2,500
52	WS-SO-10-XC	Xcalibur XRF Services	328	1,175	19	151	3,742
52	WS-SO-20-XC	Xcalibur XRF Services	312	998	8	106	3,763
52	WS-SO-23-XC	Xcalibur XRF Services	319	1,017	16	117	3,736
53	AS-SO-03-XX	Reference Laboratory	520	200	480 J-	29	350
53	AS-SO-05-XX	Reference Laboratory	370	140	330 J-	23	250
53	AS-SO-08-XX	Reference Laboratory	380	140	280 J-	23	260
53	AS-SO-03-XC	Xcalibur XRF Services	607	334	366	13	532
53	AS-SO-05-XC	Xcalibur XRF Services	675	307	544	0	599
53	AS-SO-08-XC	Xcalibur XRF Services	605	339	377	0	556

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
54	LV-SO-03-XX	Reference Laboratory	1.6	42	590	600	130	24,000	94	48 J-
54	LV-SO-40-XX	Reference Laboratory	2.7	42	580	590	130	24,000	92	46 J-
54	LV-SO-49-XX	Reference Laboratory	7.4	43	600	610	130	25,000	98	52 J-
54	LV-SO-03-XC	Xcalibur XRF Services	6	61	670	346	451	34,877	173	
54	LV-SO-40-XC	Xcalibur XRF Services	4	75	730	393	449	35,004	153	13
54	LV-SO-49-XC	Xcalibur XRF Services	1	65	604	346	413	30,730	197	
55	LV-SO-04-XX	Reference Laboratory	860	120	2,400	2,300	98	22,000	4,000	130 J-
55	LV-SO-34-XX	Reference Laboratory	870 J-	110 J-	2,300 J-	2,200 J-	87	20,000 J-	3,700 J-	130 J-
55	LV-SO-37-XX	Reference Laboratory	590	84	1,700	1,600	66	16,000	2,800	130 J-
55	LV-SO-04-XC	Xcalibur XRF Services	347	90	3,054	1,351	61	20,656	8,958	37
55	LV-SO-34-XC	Xcalibur XRF Services	335	52	2,985	1,443	48	20,387	8,837	38
55	LV-SO-37-XC	Xcalibur XRF Services	344	39	3,095	1,423	72	20,591	8,918	21
56	CN-SO-03-XX	Reference Laboratory	22	87	63	17	72	15,000	130	34 J-
56	CN-SO-06-XX	Reference Laboratory	20	91	64	18	74	16,000	130	40 J-
56	CN-SO-07-XX	Reference Laboratory	20	90	63	19	72	17,000	130	36 J-
56	CN-SO-03-XC	Xcalibur XRF Services	11	169	77		93	16,552	272	10
56	CN-SO-06-XC	Xcalibur XRF Services	12	201	68		82	15,546	256	7
56	CN-SO-07-XC	Xcalibur XRF Services	8	163	94		93	17,267	294	15
57	CN-SO-02-XX	Reference Laboratory	230	19	820	290	140	22,000	490	270 J-
57	CN-SO-05-XX	Reference Laboratory	130	6	630	26	160	23,000	25	280 J-
57	CN-SO-09-XX	Reference Laboratory	120	6	580	21	140	19,000	23	260 J-
57	CN-SO-02-XC	Xcalibur XRF Services	74	79	677	38	336	22,137	0	98
57	CN-SO-05-XC	Xcalibur XRF Services	78	80	695	35	308	20,389	0	110
57	CN-SO-09-XC	Xcalibur XRF Services	78	51	667		296	20,290	43	98
58	LV-SE-06-XX	Reference Laboratory	30	23	160	540	30	18,000	1,600	610 J-
58	LV-SE-13-XX	Reference Laboratory	31	24	160	540	30	18,000	1,600	640 J-
58	LV-SE-41-XX	Reference Laboratory	30	21	150	480	26	16,000	1,500	610 J-
58	LV-SE-06-XC	Xcalibur XRF Services	40	87	269	341	3	17,343	3,778	245
58	LV-SE-13-XC	Xcalibur XRF Services	38	54	234	379	4	17,202	3,596	225
58	LV-SE-41-XC	Xcalibur XRF Services	45	74	235	365	16	18,059	3,754	249
59	LV-SE-05-XX	Reference Laboratory	92	20	440	840	39	16,000	14	2.6 J-
59	LV-SE-20-XX	Reference Laboratory	140 J+	31	680	1,400	60	22,000	21	2.8
59	LV-SE-43-XX	Reference Laboratory	160 J+	24	550	1,100	47	19,000	17	2.8
59	LV-SE-05-XC	Xcalibur XRF Services	90	93	657	652	225	25,232	101	
59	LV-SE-20-XC	Xcalibur XRF Services	93	74	745	714	231	24,839	63	
59	LV-SE-43-XC	Xcalibur XRF Services	103	59	743	709	233	24,954	107	8

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
54	LV-SO-03-XX	Reference Laboratory	2,000		120		210	J-	120		3,700	
54	LV-SO-40-XX	Reference Laboratory	1,900		120		210	J-	120		3,700	
54	LV-SO-49-XX	Reference Laboratory	2,000		120		220	J-	120		3,800	
54	LV-SO-03-XC	Xcalibur XRF Services	1,795		320		204		41		4,677	
54	LV-SO-40-XC	Xcalibur XRF Services	1,815		291		223		99		4,664	
54	LV-SO-49-XC	Xcalibur XRF Services	1,650		270		191		81		4,653	
55	LV-SO-04-XX	Reference Laboratory	2,000		230		1.2	UJ	260		53	
55	LV-SO-34-XX	Reference Laboratory	1,900	J-	220	J-	1.2	UJ	230	J-	48	J-
55	LV-SO-37-XX	Reference Laboratory	1,400		170		1.2	U	180		37	
55	LV-SO-04-XC	Xcalibur XRF Services	1,834		502				107		57	
55	LV-SO-34-XC	Xcalibur XRF Services	1,791		549		19		105		38	
55	LV-SO-37-XC	Xcalibur XRF Services	1,875		586		22		133		63	
56	CN-SO-03-XX	Reference Laboratory	74		36		90		30		58	
56	CN-SO-06-XX	Reference Laboratory	76		38		94		32		59	
56	CN-SO-07-XX	Reference Laboratory	75		37		91		33		58	
56	CN-SO-03-XC	Xcalibur XRF Services	81		22		114		33		132	
56	CN-SO-06-XC	Xcalibur XRF Services	78		46		99		23		140	
56	CN-SO-07-XC	Xcalibur XRF Services	102		41		108		29		140	
57	CN-SO-02-XX	Reference Laboratory	530		190		68		160		1,900	
57	CN-SO-05-XX	Reference Laboratory	360		190		78		160		2,200	
57	CN-SO-09-XX	Reference Laboratory	330		170		74		140		2,100	
57	CN-SO-02-XC	Xcalibur XRF Services	327		406		82		75		3,552	
57	CN-SO-05-XC	Xcalibur XRF Services	308		385		73		67		3,580	
57	CN-SO-09-XC	Xcalibur XRF Services	289		388		92		78		3,451	
58	LV-SE-06-XX	Reference Laboratory	360		160		110		480		52	
58	LV-SE-13-XX	Reference Laboratory	360		160		110		470		51	
58	LV-SE-41-XX	Reference Laboratory	320		150		99		420		46	
58	LV-SE-06-XC	Xcalibur XRF Services	299		406		148		194		64	
58	LV-SE-13-XC	Xcalibur XRF Services	305		343		139		207		79	
58	LV-SE-41-XC	Xcalibur XRF Services	331		329		139		217		64	
59	LV-SE-05-XX	Reference Laboratory	400		340		49		340		1,800	
59	LV-SE-20-XX	Reference Laboratory	660		500		75	J-	530		2,800	
59	LV-SE-43-XX	Reference Laboratory	530		420		60	J-	430		2,300	
59	LV-SE-05-XC	Xcalibur XRF Services	411		1,189		65		151		3,269	
59	LV-SE-20-XC	Xcalibur XRF Services	427		1,255		74		211		3,354	
59	LV-SE-43-XC	Xcalibur XRF Services	420		1,210		77		173		3,300	

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
60	LV-SE-15-XX	Reference Laboratory	290 J+	32	1,300	83	2,300	22,000	18	500
60	LV-SE-17-XX	Reference Laboratory	280 J+	31	1,300	79	2,200	21,000	17 J-	490
60	LV-SE-51-XX	Reference Laboratory	210 J+	26	1,100	72	2,000	19,000	15	470
60	LV-SE-15-XC	Xcalibur XRF Services	150	70	1,365	69	2,440	18,435	47	247
60	LV-SE-17-XC	Xcalibur XRF Services	152	72	1,362		2,459	18,517	80	236
60	LV-SE-51-XC	Xcalibur XRF Services	119	129	1,186	45	2,275	17,652	0	212
61	TL-SE-05-XX	Reference Laboratory	100 J+	34	0.34 J	40	4,900	24,000	1,200	980
61	TL-SE-09-XX	Reference Laboratory	100 J+	33	0.24 J	39	4,800	23,000	1,200	820
61	TL-SE-13-XX	Reference Laboratory	95 J+	31	0.45 J	36 J+	4,400 J+	22,000 J+	1,100 J+	990
61	TL-SE-05-XC	Xcalibur XRF Services	134	53			6,233	22,153	2,327	424
61	TL-SE-09-XC	Xcalibur XRF Services	128	65			6,272	21,976	2,295	434
61	TL-SE-13-XC	Xcalibur XRF Services	125	87			6,013	22,026	2,354	450
62	TL-SE-06-XX	Reference Laboratory	1.2 U	86	350	34	2000	22,000	1,700	2.2
62	TL-SE-17-XX	Reference Laboratory	1.2 U	85	340	33	2100	21,000	1,700	2.6
62	TL-SE-28-XX	Reference Laboratory	1.2 U	89	360	34	2100	22,000	1,700	2.8
62	TL-SE-06-XC	Xcalibur XRF Services	16	67	463		2,895	22,639	4,143	
62	TL-SE-17-XC	Xcalibur XRF Services	17	63	501		2,729	21,993	4,379	
62	TL-SE-28-XC	Xcalibur XRF Services	21	126	520	20	2,659	21,638	4,711	
63	TL-SE-07-XX	Reference Laboratory	30	11	48	66	2200	37,000	13	40
63	TL-SE-21-XX	Reference Laboratory	33	13	51	73	2300	44,000	15	120
63	TL-SE-30-XX	Reference Laboratory	31	11	47	64	2200	36,000	14	100
63	TL-SE-07-XC	Xcalibur XRF Services	48	74	58	16	3,700	53,232	73	41
63	TL-SE-21-XC	Xcalibur XRF Services	50	69	42	13	3,736	53,772	117	19
63	TL-SE-30-XC	Xcalibur XRF Services	52	54	44		3,573	52,769	129	23
64	TL-SE-02-XX	Reference Laboratory	77	15	160	64	3,100	32,000	12	400
64	TL-SE-08-XX	Reference Laboratory	66	10	180	74	3,200	45,000	11	350
64	TL-SE-16-XX	Reference Laboratory	73	15	170	69	3,100	38,000	13	420
64	TL-SE-02-XC	Xcalibur XRF Services	101	55	159	43	4,258	45,180	144	152
64	TL-SE-08-XC	Xcalibur XRF Services	123	71	214		5,452	54,821	73	163
64	TL-SE-16-XC	Xcalibur XRF Services	120	33	213	12	5,324	52,189	43	192

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag		V		Zn
60	LV-SE-15-XX	Reference Laboratory	230	92	300	J-	180		62
60	LV-SE-17-XX	Reference Laboratory	220	89	200	J-	170		58
60	LV-SE-51-XX	Reference Laboratory	200	76	250	J-	160		54
60	LV-SE-15-XC	Xcalibur XRF Services	187	76	513		94		27
60	LV-SE-17-XC	Xcalibur XRF Services	184	95	577		127		53
60	LV-SE-51-XC	Xcalibur XRF Services	166	70	437		69		0
61	TL-SE-05-XX	Reference Laboratory	54	130	180	J-	66		100
61	TL-SE-09-XX	Reference Laboratory	53	130	170	J-	63		100
61	TL-SE-13-XX	Reference Laboratory	49	120	160	J	59	J+	96
61	TL-SE-05-XC	Xcalibur XRF Services	116	190	224		0		86
61	TL-SE-09-XC	Xcalibur XRF Services	120	216	254		0		104
61	TL-SE-13-XC	Xcalibur XRF Services	103	180	227		0		99
62	TL-SE-06-XX	Reference Laboratory	44	45	56		78		83
62	TL-SE-17-XX	Reference Laboratory	43	44	56		78		81
62	TL-SE-28-XX	Reference Laboratory	44	45	57		81		83
62	TL-SE-06-XC	Xcalibur XRF Services	90	83	86		0		105
62	TL-SE-17-XC	Xcalibur XRF Services	71	31	85		0		116
62	TL-SE-28-XC	Xcalibur XRF Services	65	50	89		13		117
63	TL-SE-07-XX	Reference Laboratory	94	120	63		110		160
63	TL-SE-21-XX	Reference Laboratory	100	140	67		120		170
63	TL-SE-30-XX	Reference Laboratory	93	120	62		100		160
63	TL-SE-07-XC	Xcalibur XRF Services	152	404	117		0		95
63	TL-SE-21-XC	Xcalibur XRF Services	118	421	99		0		94
63	TL-SE-30-XC	Xcalibur XRF Services	122	483	130		58		115
64	TL-SE-02-XX	Reference Laboratory	99	44	120		110		160
64	TL-SE-08-XX	Reference Laboratory	100	39	130		120		170
64	TL-SE-16-XX	Reference Laboratory	100	44	120		110		160
64	TL-SE-02-XC	Xcalibur XRF Services	181	77	171		65		91
64	TL-SE-08-XC	Xcalibur XRF Services	155	92	227		0		93
64	TL-SE-16-XC	Xcalibur XRF Services	176	66	227		0		91

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
65	RF-SE-01-XX	Reference Laboratory	12	230	40	280	63	14,000	22	47
65	RF-SE-09-XX	Reference Laboratory	10	260	45	310	71	16,000	26	45
65	RF-SE-11-XX	Reference Laboratory	11	240	43	300	72	15,000	25	52
65	RF-SE-17-XX	Reference Laboratory	11	250	43	300	67	15,000	26	20
65	RF-SE-29-XX	Reference Laboratory	13	280	49	330	75	17,000	26	20
65	RF-SE-37-XX	Reference Laboratory	11	260	45	320	72	16,000	27	22
65	RF-SE-50-XX	Reference Laboratory	8.9	230	40	280	65	14,000	23	19
65	RF-SE-01-XC	Xcalibur XRF Services	8	406	49	158	205	15,474	33	7
65	RF-SE-09-XC	Xcalibur XRF Services	11	424	69	232	216	15,814	77	19
65	RF-SE-11-XC	Xcalibur XRF Services	10	433	52	213	209	15,982	27	7
65	RF-SE-17-XC	Xcalibur XRF Services	11	406	59	270	220	16,378	82	16
65	RF-SE-29-XC	Xcalibur XRF Services	0	418	69	192	212	16,296	70	17
65	RF-SE-37-XC	Xcalibur XRF Services	0	447	66	219	216	16,332	32	6
65	RF-SE-50-XC	Xcalibur XRF Services	4	437	62	219	211	16,381	37	14
66	RF-SE-08-XX	Reference Laboratory	14	460	67	510	1,800	18,000	580	29
66	RF-SE-10-XX	Reference Laboratory	12	400	58	440	1,500	16,000	510	27
66	RF-SE-33-XX	Reference Laboratory	13	440	64	490	1,700	18,000	570	28
66	RF-SE-08-XC	Xcalibur XRF Services	21	737	65	337	2,066	12,810	1,023	16
66	RF-SE-10-XC	Xcalibur XRF Services	21	784	75	355	2,205	13,872	987	8
66	RF-SE-33-XC	Xcalibur XRF Services	19	709	73	324	2,138	13,548	1,068	11
67	RF-SE-16-XX	Reference Laboratory	85 J-	72 J-	310 J-	820 J-	73 J-	16,000 J-	24 J-	260
67	RF-SE-41-XX	Reference Laboratory	100	82	360	950	85	18,000	25	230
67	RF-SE-48-XX	Reference Laboratory	100	87	380	1,000	90	19,000	27	250
67	RF-SE-16-XC	Xcalibur XRF Services	52	114	421	664	112	14,873	44	102
67	RF-SE-41-XC	Xcalibur XRF Services	52	109	463	617	97	14,638	74	107
67	RF-SE-48-XC	Xcalibur XRF Services	57	142	432	640	104	14,899	36	111
68	RF-SE-18-XX	Reference Laboratory	320	810	770	950	78	16,000	860	600
68	RF-SE-35-XX	Reference Laboratory	300	740	700	860	70	15,000	780	650
68	RF-SE-54-XX	Reference Laboratory	320	880	840	1,000	86	18,000	920	670
68	RF-SE-18-XC	Xcalibur XRF Services	160	1,208	899	615	30	11,932	1,267	221
68	RF-SE-35-XC	Xcalibur XRF Services	164	1,239	942	681	44	12,298	1,329	242
68	RF-SE-54-XC	Xcalibur XRF Services	165	1,238	918	621	37	12,444	1,346	243

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
65	RF-SE-01-XX	Reference Laboratory	200	21	37	29	1,700
65	RF-SE-09-XX	Reference Laboratory	220	23	42	32	1,900
65	RF-SE-11-XX	Reference Laboratory	210	20	40	29	1,800
65	RF-SE-17-XX	Reference Laboratory	210	22	40	30	1,800
65	RF-SE-29-XX	Reference Laboratory	240	26	44	35	2,100
65	RF-SE-37-XX	Reference Laboratory	220	23	44	32	1,900
65	RF-SE-50-XX	Reference Laboratory	200	20	38	29	1,700
65	RF-SE-01-XC	Xcalibur XRF Services	169		46	0	3,293
65	RF-SE-09-XC	Xcalibur XRF Services	182		42	48	3,500
65	RF-SE-11-XC	Xcalibur XRF Services	192		27	0	3,405
65	RF-SE-17-XC	Xcalibur XRF Services	189	31	49	0	3,473
65	RF-SE-29-XC	Xcalibur XRF Services	186		62	0	3,421
65	RF-SE-37-XC	Xcalibur XRF Services	178		43	0	3,423
65	RF-SE-50-XC	Xcalibur XRF Services	183	59	52	0	3,463
66	RF-SE-08-XX	Reference Laboratory	250	42	0.39 U	120	120
66	RF-SE-10-XX	Reference Laboratory	220	39	0.34 U	100	110
66	RF-SE-33-XX	Reference Laboratory	240	41	0.33 U	120	130
66	RF-SE-08-XC	Xcalibur XRF Services	233			59	81
66	RF-SE-10-XC	Xcalibur XRF Services	252	28	13	61	107
66	RF-SE-33-XC	Xcalibur XRF Services	247	6	5	37	105
67	RF-SE-16-XX	Reference Laboratory	1,700 J-	1.2 U	130 J-	32 J-	760 J-
67	RF-SE-41-XX	Reference Laboratory	1,900	1.2 U	140	39	830
67	RF-SE-48-XX	Reference Laboratory	2,000	2.2	150	40	880
67	RF-SE-16-XC	Xcalibur XRF Services	1,663		156	21	1,373
67	RF-SE-41-XC	Xcalibur XRF Services	1,618		166	0	1,401
67	RF-SE-48-XC	Xcalibur XRF Services	1,608		169	54	1,377
68	RF-SE-18-XX	Reference Laboratory	390	140	140	390	120
68	RF-SE-35-XX	Reference Laboratory	350	140	150	340	110
68	RF-SE-54-XX	Reference Laboratory	420	160	180	410	120
68	RF-SE-18-XC	Xcalibur XRF Services	276	265	258	142	164
68	RF-SE-35-XC	Xcalibur XRF Services	340	257	268	142	176
68	RF-SE-54-XC	Xcalibur XRF Services	307	268	260	132	153

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
69	RF-SE-20-XX	Reference Laboratory	550	1300	540	94	93	20,000	28	0.48
69	RF-SE-46-XX	Reference Laboratory	270	590	240	44	40	8,900	13	0.45
69	RF-SE-51-XX	Reference Laboratory	480	1100	450	77	77	17,000	23	0.48
69	RF-SE-20-XC	Xcalibur XRF Services	219	1,760	576	43	144	16,036		13
69	RF-SE-46-XC	Xcalibur XRF Services	232	1,858	619	73	139	16,115	89	19
69	RF-SE-51-XC	Xcalibur XRF Services	232	1,774	595	76	136	16,229	76	12
70	RF-SE-21-XX	Reference Laboratory	1.3 U	62	1,700	76	1,000	16,000	2,100	320
70	RF-SE-40-XX	Reference Laboratory	1.3 U	70	1,900	85	1,100	18,000	2,400	280
70	RF-SE-47-XX	Reference Laboratory	1.3 U	72	1,900	90	1,200	19,000	2,400	320
70	RF-SE-21-XC	Xcalibur XRF Services	19	85	2,180	50	1,060	13,404	3,814	95
70	RF-SE-40-XC	Xcalibur XRF Services	20	122	2,314	91	1,101	13,281	3,973	93
70	RF-SE-47-XC	Xcalibur XRF Services	19	116	2,295	53	1,093	13,555	3,841	92

Appendix D. Analytical Data Summary, Xcalibur ElvaX and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
69	RF-SE-20-XX	Reference Laboratory	1,400	380	59	36	1,400
69	RF-SE-46-XX	Reference Laboratory	650	170	26	16	650
69	RF-SE-51-XX	Reference Laboratory	1,200	320	48	30	1,200
69	RF-SE-20-XC	Xcalibur XRF Services	1,079	828	80	0	2,094
69	RF-SE-46-XC	Xcalibur XRF Services	1,056	912	69	0	2,115
69	RF-SE-51-XC	Xcalibur XRF Services	1,075	818	84	0	2,054
70	RF-SE-21-XX	Reference Laboratory	220	440	120	130	100
70	RF-SE-40-XX	Reference Laboratory	250	480	100	150	120
70	RF-SE-47-XX	Reference Laboratory	250	510	120	150	120
70	RF-SE-21-XC	Xcalibur XRF Services	179	1,232	305	58	107
70	RF-SE-40-XC	Xcalibur XRF Services	193	1,299	325	52	61
70	RF-SE-47-XC	Xcalibur XRF Services	211	1,321	312	59	117

Notes:
All concentrations reported in milligrams per kilogram (mg/kg), or parts per million (ppm).
Sample results for which “0” or no value was reported were considered nondetections as reported by Xcalibur.
Reference laboratory data qualifiers were as follows:

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

APPENDIX E

STATISTICAL DATA SUMMARIES

Figure E-1: Linear Correlation Plot for Antimony

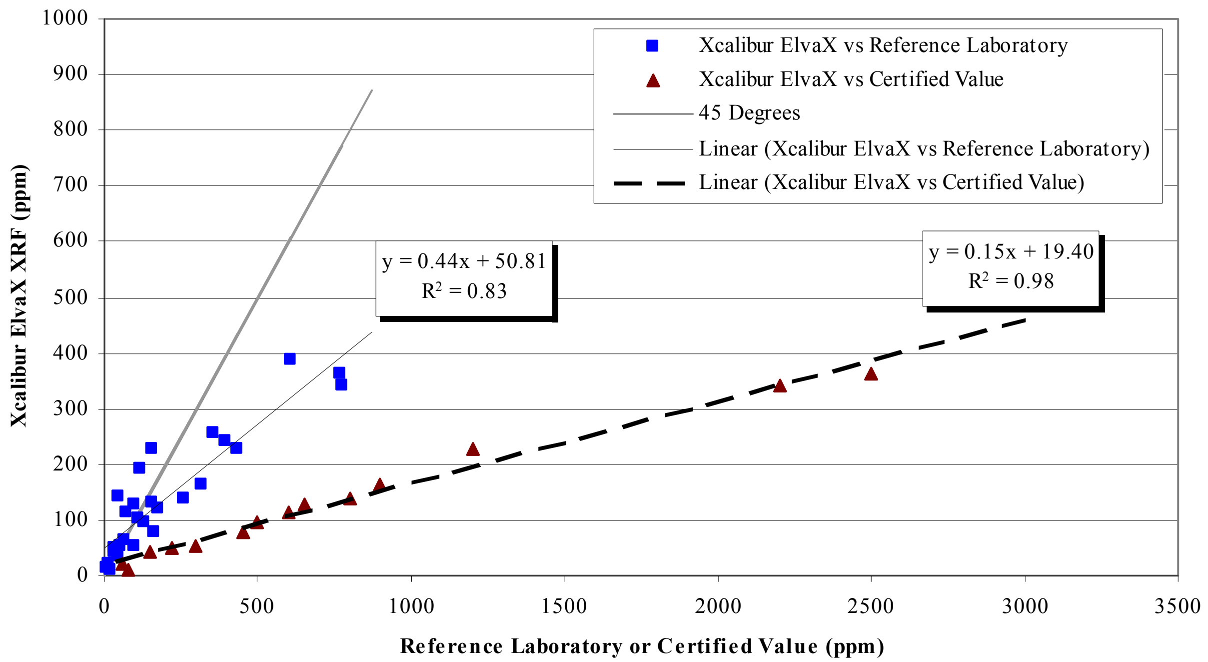


Figure E-2: Linear Correlation Plot for Arsenic

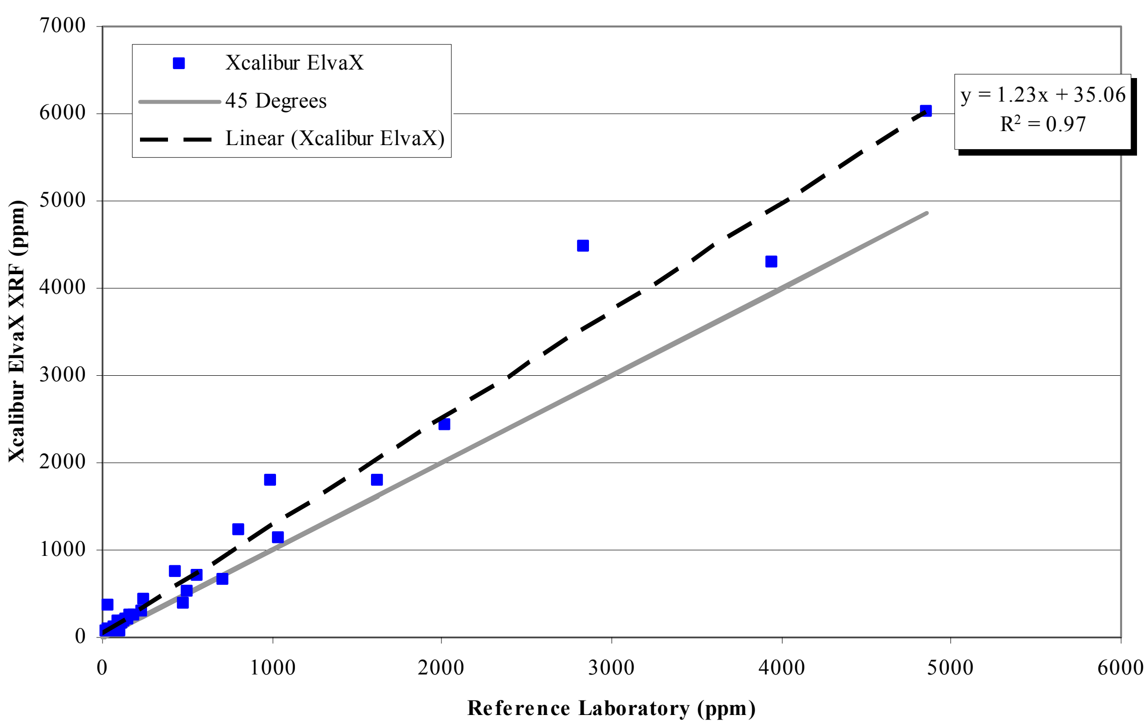


Figure E-3: Linear Correlation Plot for Cadmium

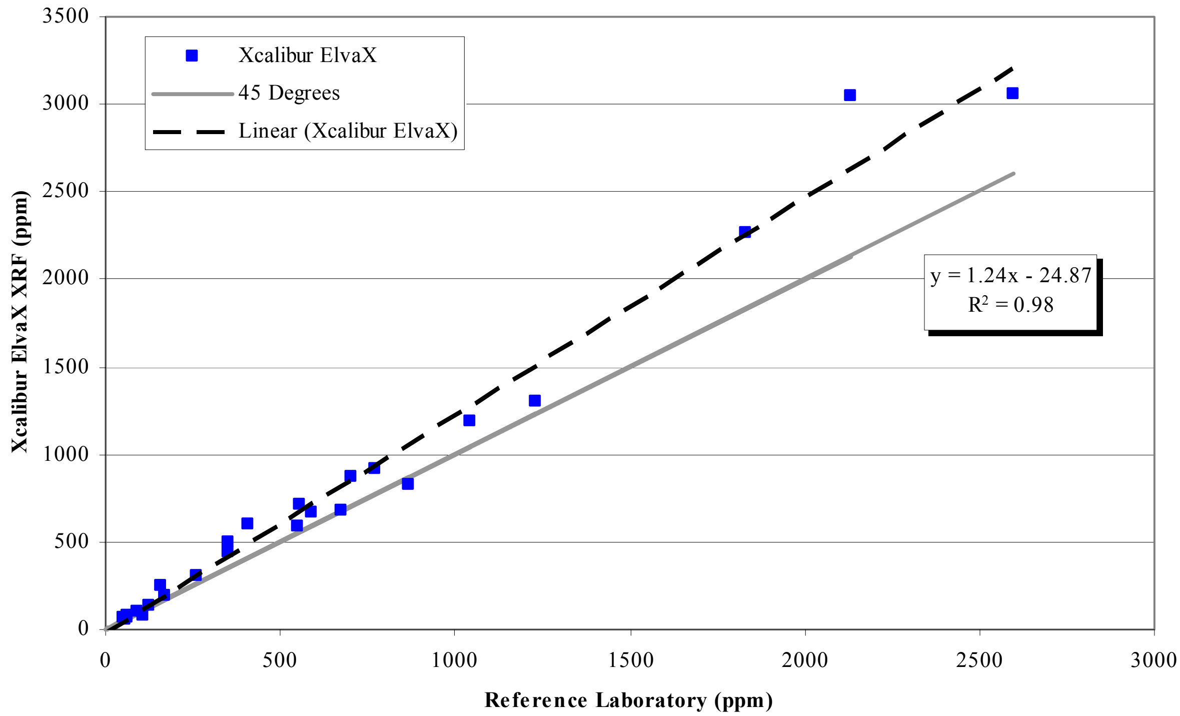


Figure E-4: Linear Correlation Plot for Chromium

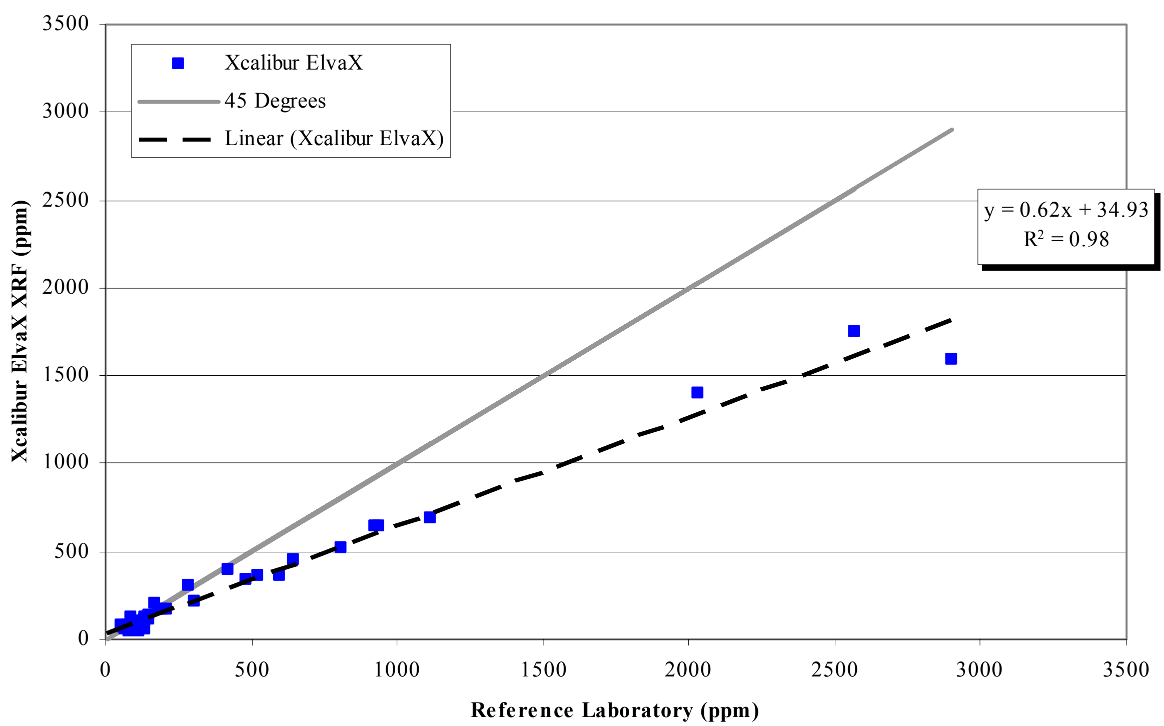


Figure E-5: Linear Correlation Plot for Copper

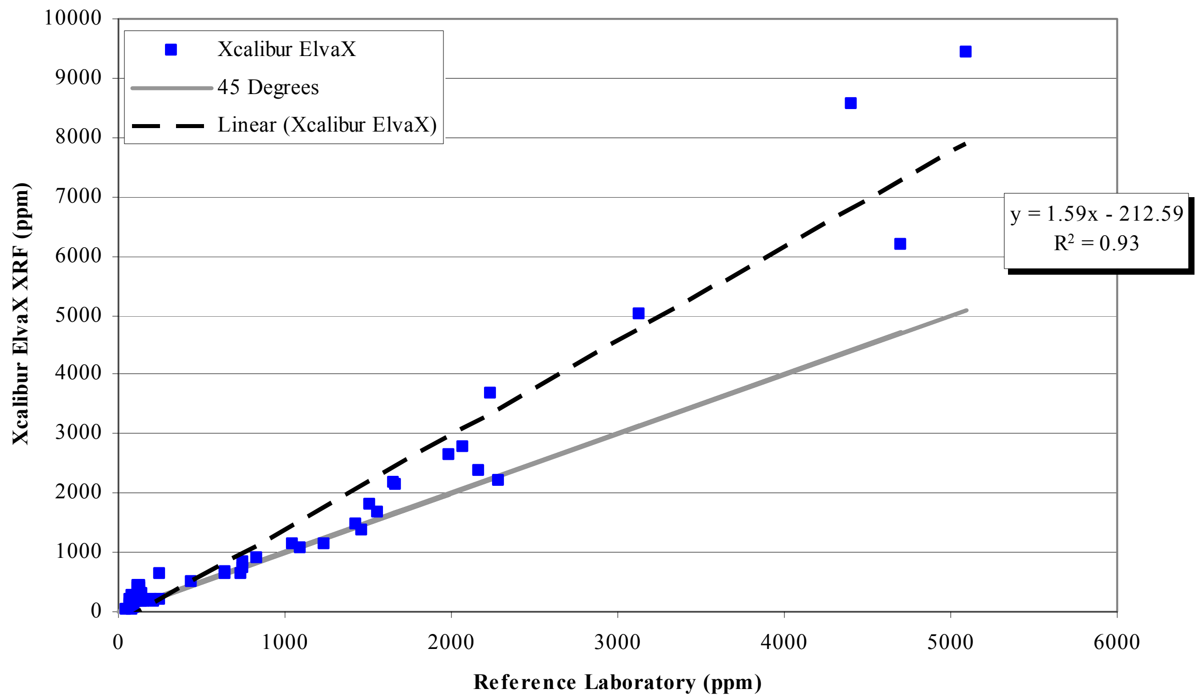
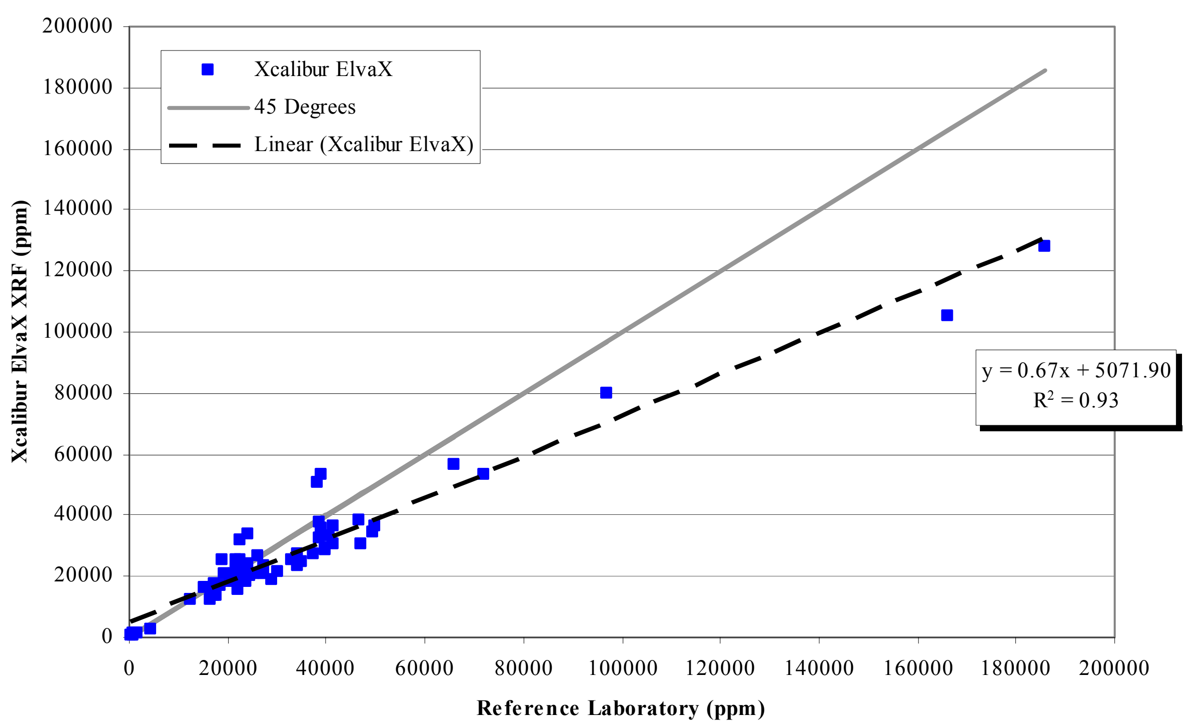


Figure E-6: Linear Correlation Plot for Iron



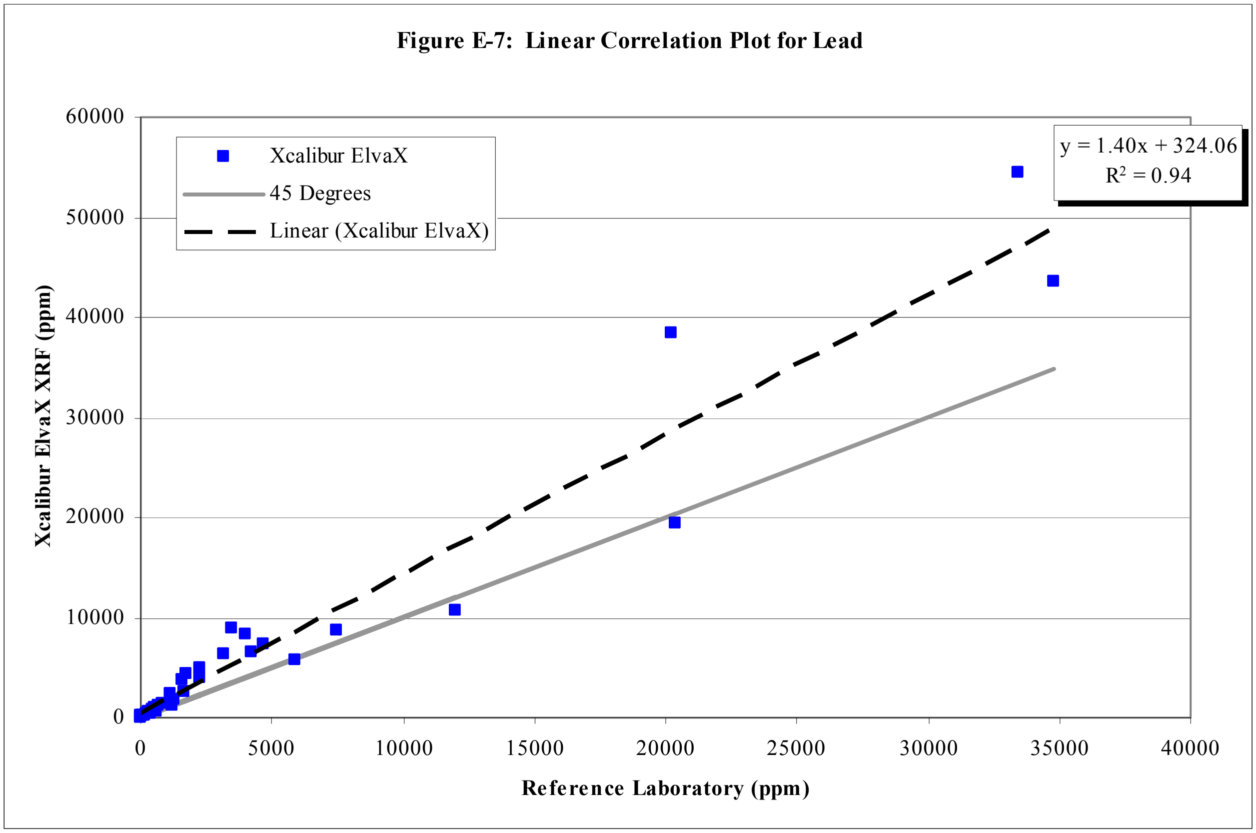


Figure E-7: Linear Correlation Plot for Lead
Xcalibur ElvaX
45 Degrees
Linear (Xcalibur ElvaX)
y = 1.40x + 324.06
R² = 0.94
Xcalibur ElvaX XRF (ppm)
Reference Laboratory (ppm)
0
10000
20000
30000
40000
50000
60000
0
5000
10000
15000
20000
25000
30000
35000
40000

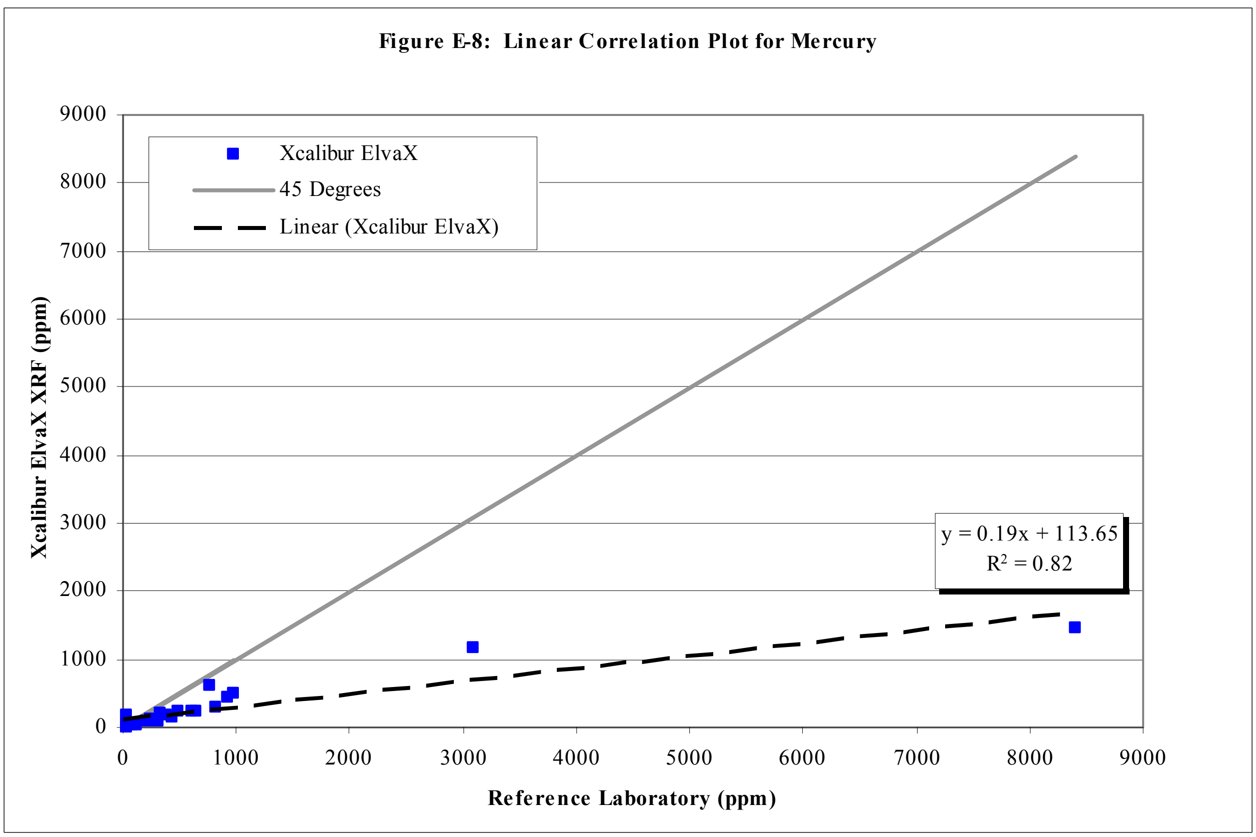


Figure E-8: Linear Correlation Plot for Mercury
Xcalibur ElvaX
45 Degrees
Linear (Xcalibur ElvaX)
y = 0.19x + 113.65
R² = 0.82
Xcalibur ElvaX XRF (ppm)
Reference Laboratory (ppm)
0
1000
2000
3000
4000
5000
6000
7000
8000
9000

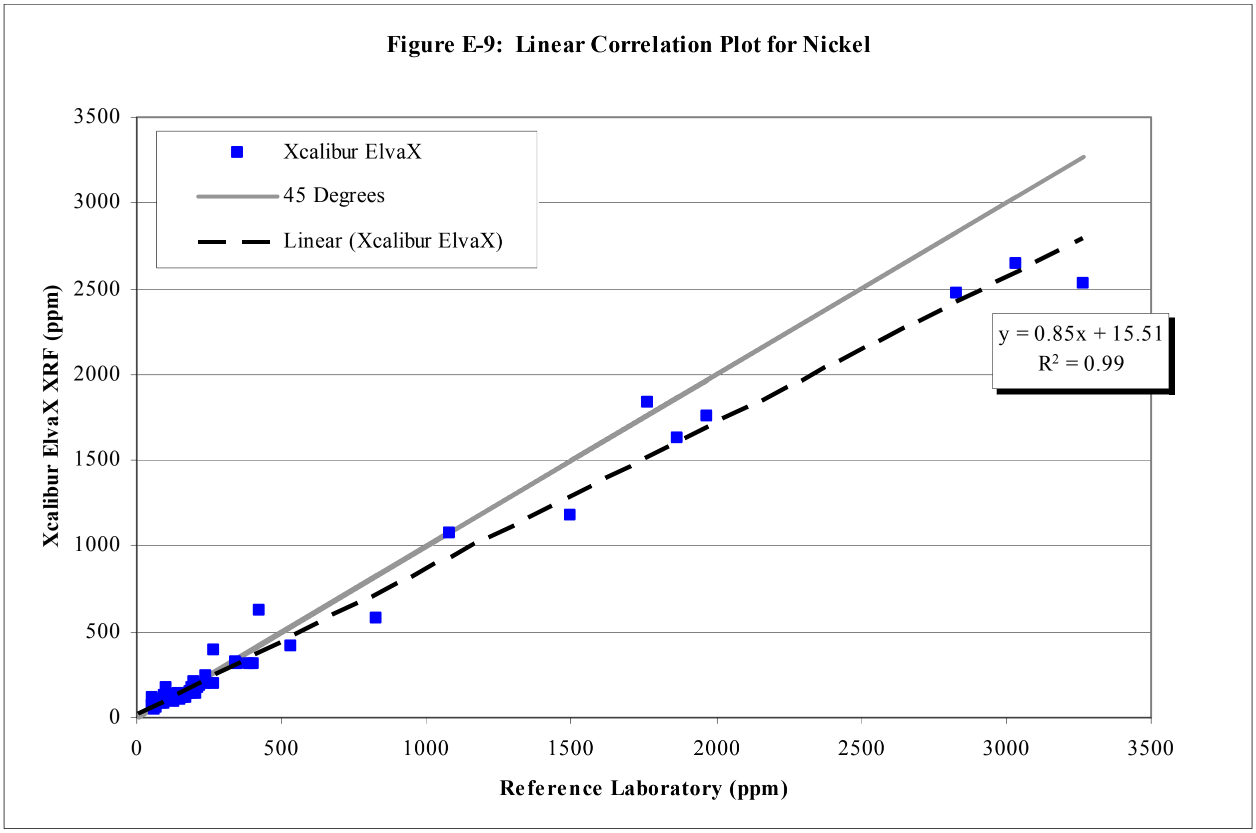


Figure E-9: Linear Correlation Plot for Nickel
Xcalibur ElvaX
45 Degrees
Linear (Xcalibur ElvaX)
y = 0.85x + 15.51
R² = 0.99
Xcalibur ElvaX XRF (ppm)
Reference Laboratory (ppm)
0
500
1000
1500
2000
2500
3000
3500

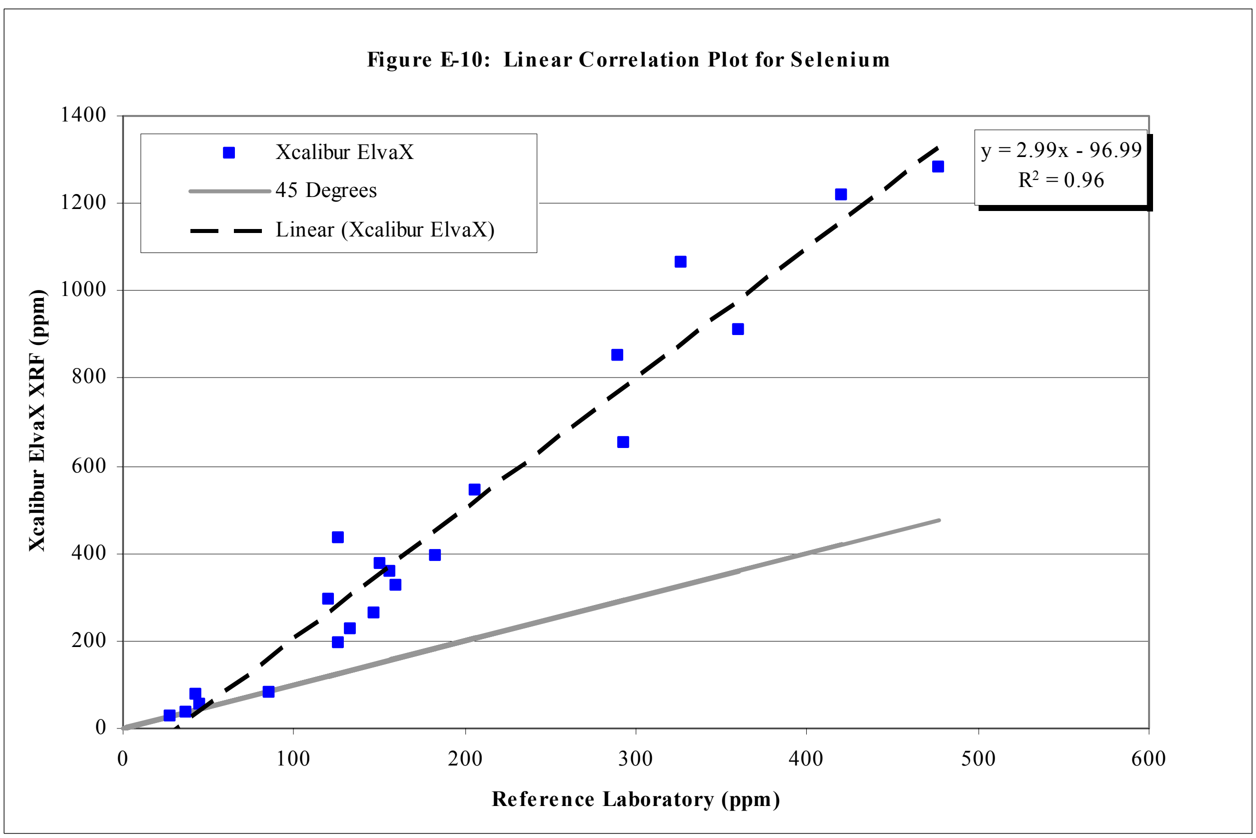


Figure E-10: Linear Correlation Plot for Selenium
Xcalibur ElvaX
45 Degrees
Linear (Xcalibur ElvaX)
y = 2.99x - 96.99
R² = 0.96
Xcalibur ElvaX XRF (ppm)
Reference Laboratory (ppm)
0
200
400
600
800
1000
1200
1400
100
300
500

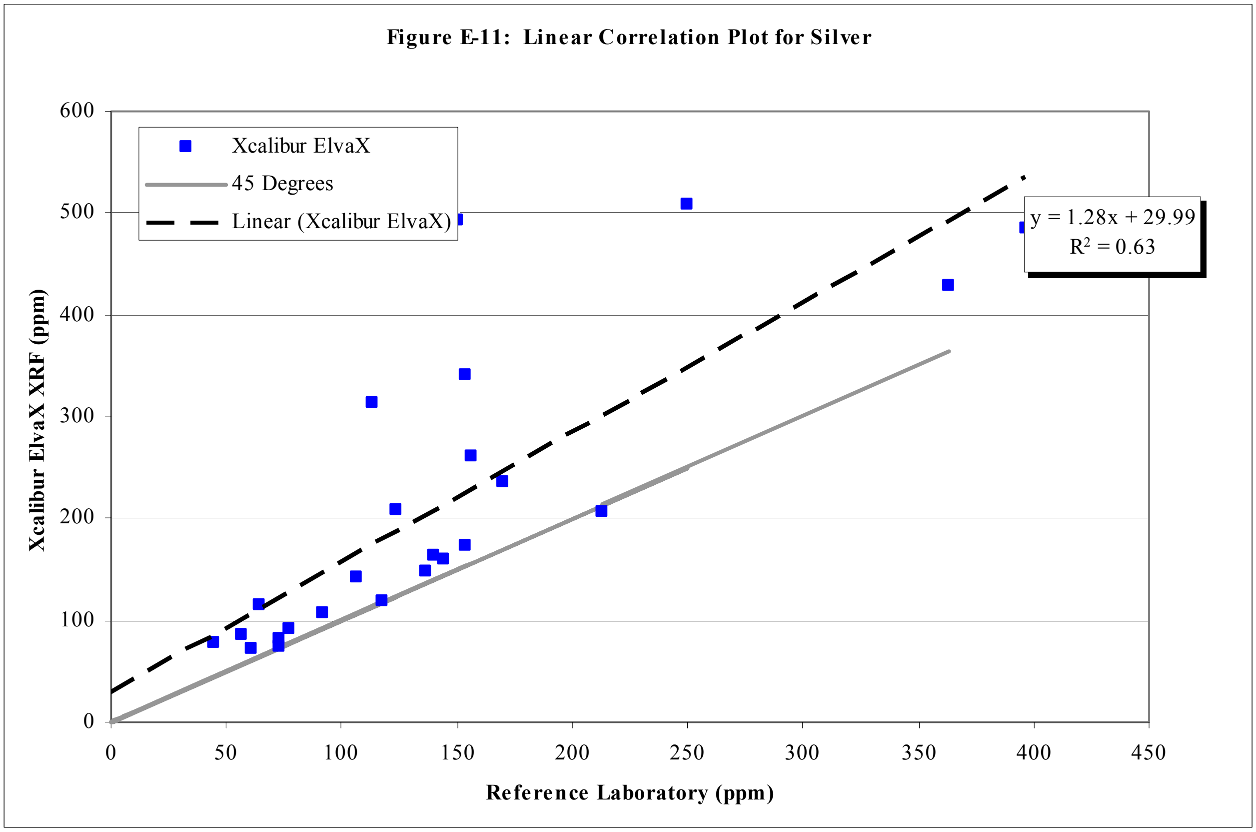


Figure E-11: Linear Correlation Plot for Silver
Xcalibur ElvaX
45 Degrees
Linear (Xcalibur ElvaX)
y = 1.28x + 29.99
R² = 0.63
Xcalibur ElvaX XRF (ppm)
Reference Laboratory (ppm)
0
100
200
300
400
500
600
0
50
100
150
200
250
300
350
400
450

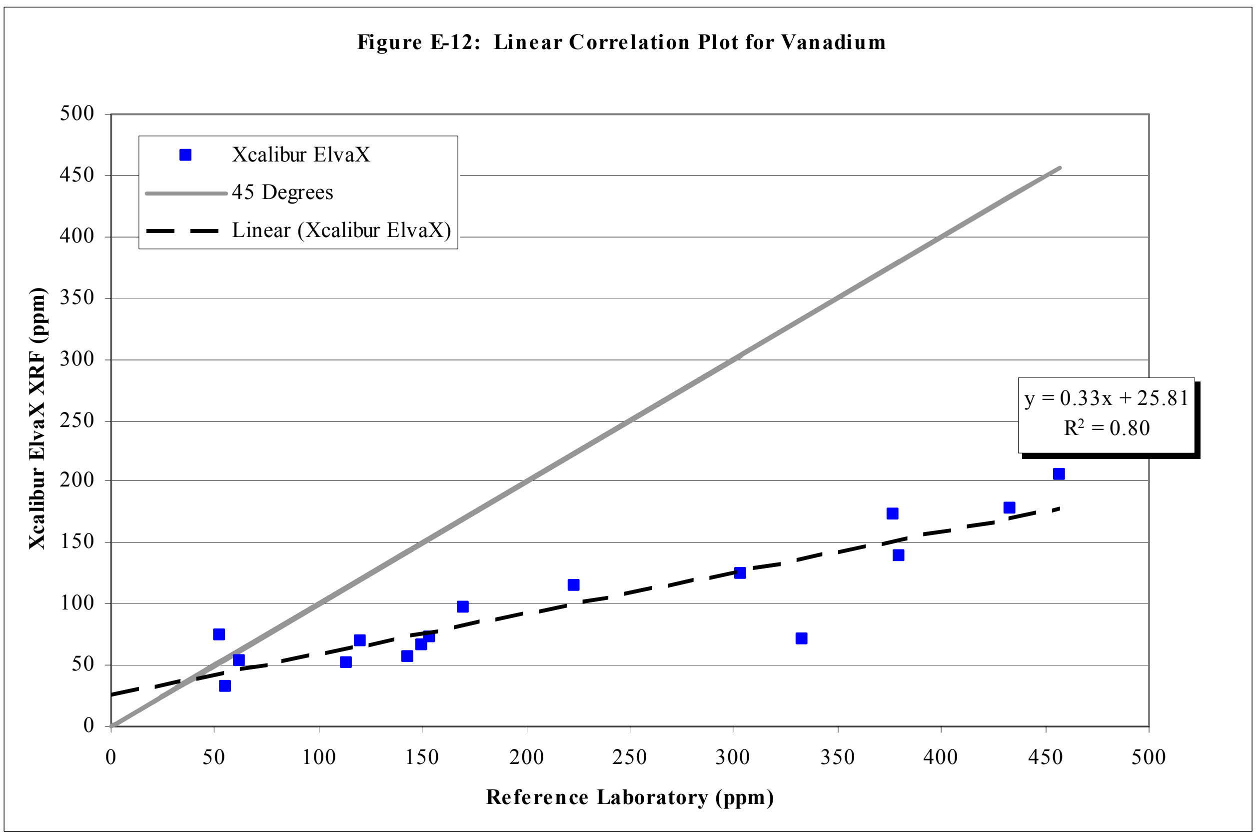


Figure E-12: Linear Correlation Plot for Vanadium
Xcalibur ElvaX
45 Degrees
Linear (Xcalibur ElvaX)
y = 0.33x + 25.81
R² = 0.80
Xcalibur ElvaX XRF (ppm)
Reference Laboratory (ppm)
0
50
100
150
200
250
300
350
400
450
500

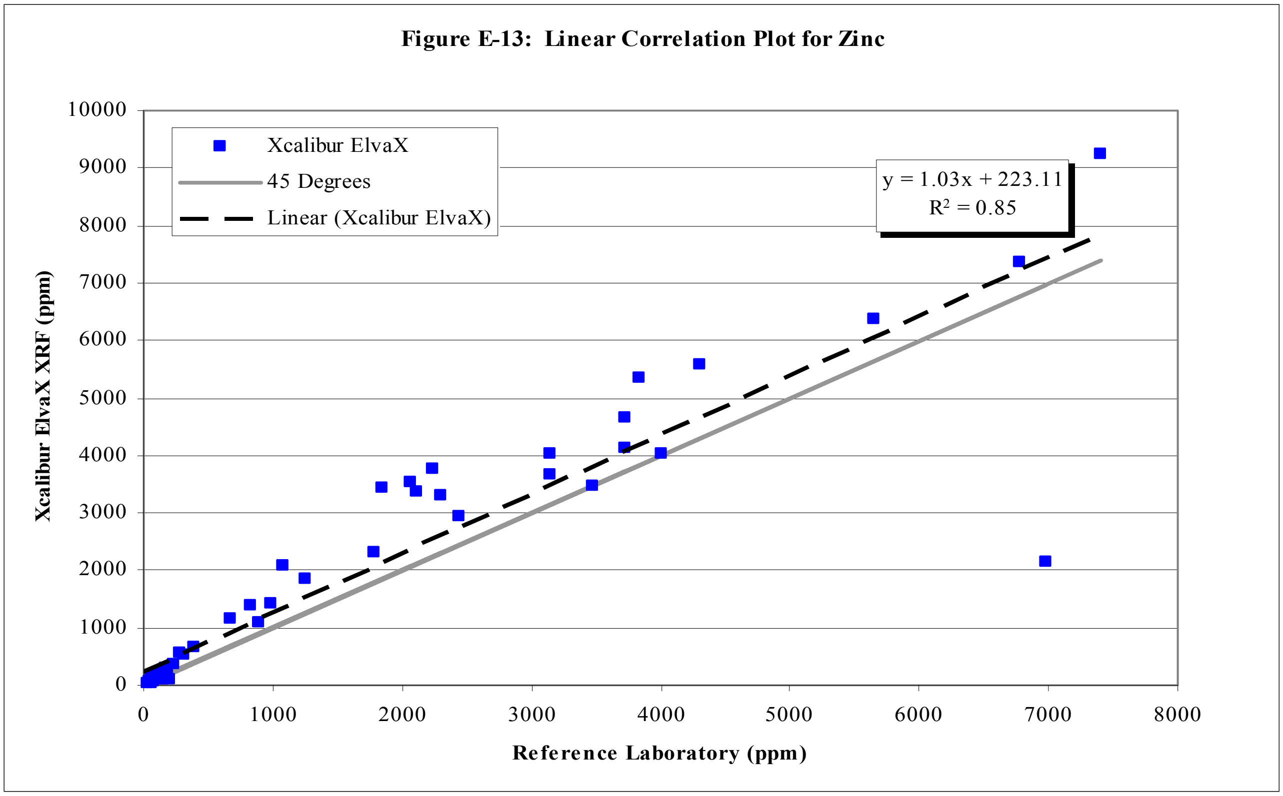
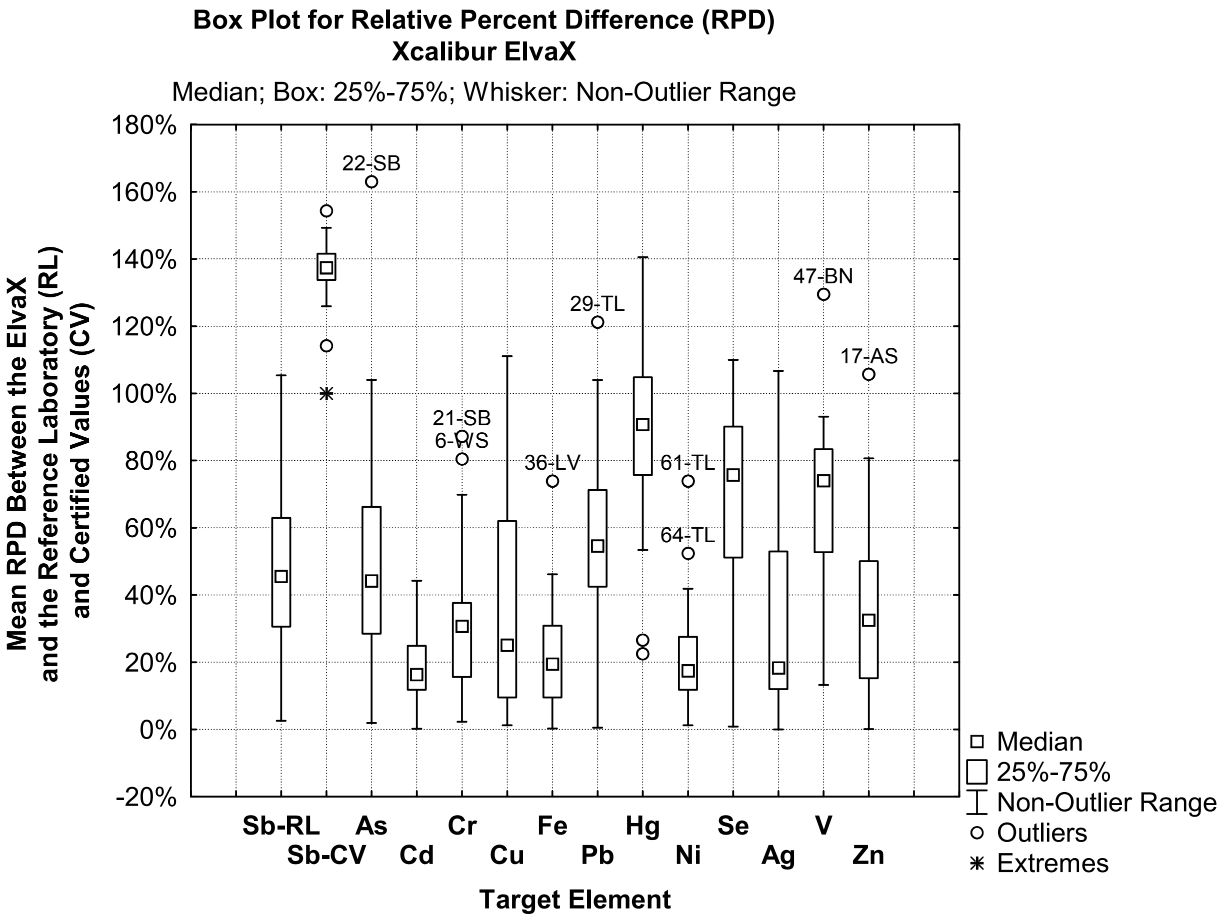


Figure E-13: Linear Correlation Plot for Zinc
Xcalibur ElvaX
45 Degrees
Linear (Xcalibur ElvaX)
y = 1.03x + 223.11
R² = 0.85
Xcalibur ElvaX XRF (ppm)
Reference Laboratory (ppm)
0
1000
2000
3000
4000
5000
6000
7000
8000
9000
10000



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 results and sampling site associated with each Blend number.

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

Table E-1. Evaluation of Accuracy - Relative Percent Difference versus Reference Laboratory Data Calculated for the Xcalibur ElvaX

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
			Ref Lab	ERA Spike							
Soil	Level 1	Number	9	1	15	7	23	16	5	7	5
		Minimum	2.6%	154.4%	33.7%	0.2%	2.3%	9.9%	0.3%	8.3%	26.6%
		Maximum	105.4%	154.4%	163.0%	29.4%	87.2%	111.1%	46.2%	75.3%	133.5%
		Mean	43.7%	154.4%	62.6%	17.3%	28.4%	61.2%	24.7%	51.3%	99.0%
		Median	47.0%	154.4%	53.8%	16.4%	21.2%	62.7%	25.5%	50.5%	107.9%
	Level 2	Number	5	1	4	7	4	8	13	4	7
		Minimum	10.2%	6.2%	8.4%	0.4%	35.6%	1.3%	1.1%	0.5%	22.5%
		Maximum	70.2%	6.2%	22.2%	35.2%	49.6%	15.7%	33.9%	88.5%	99.6%
		Mean	32.2%	6.2%	15.6%	13.5%	41.2%	7.0%	13.8%	40.4%	75.2%
		Median	31.7%	6.2%	15.9%	12.4%	39.9%	5.7%	10.3%	36.2%	90.4%
	Level 3	Number	3	2	4	2	2	2	13	8	2
		Minimum	43.9%	0.7%	8.4%	10.1%	37.6%	59.7%	2.1%	2.2%	91.0%
		Maximum	77.4%	6.3%	45.0%	16.3%	58.1%	64.4%	40.3%	87.1%	140.5%
		Mean	64.4%	3.5%	23.4%	13.2%	47.9%	62.0%	24.6%	50.9%	115.7%
		Median	71.9%	3.5%	20.1%	13.2%	47.9%	62.0%	29.1%	55.5%	115.7%
	Level 4	Number	--	--	--	--	--	--	7	5	--
		Minimum	--	--	--	--	--	--	18.9%	5.0%	--
		Maximum	--	--	--	--	--	--	43.6%	62.3%	--
		Mean	--	--	--	--	--	--	28.5%	29.6%	--
		Median	--	--	--	--	--	--	30.9%	22.3%	--
	All Soil	Number	17	4	23	16	29	26	38	24	14
		Minimum	2.6%	0.7%	8.4%	0.2%	2.3%	1.3%	0.3%	0.5%	22.5%
		Maximum	105.4%	154.4%	163.0%	35.2%	87.2%	111.1%	46.2%	88.5%	140.5%
		Mean	44.0%	41.9%	47.6%	15.1%	31.5%	44.6%	21.6%	44.8%	89.5%
		Median	43.9%	6.2%	45.0%	13.9%	29.4%	26.6%	24.1%	47.0%	96.2%

Table E-1. Evaluation of Accuracy - Relative Percent Difference versus Reference Laboratory Data Calculated for the Xcalibur ElvaX (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	24	2	3	3	20
		Minimum	1.5%	0.9%	2.6%	13.2%	0.1%
		Maximum	35.4%	3.7%	16.2%	50.4%	80.6%
		Mean	17.7%	2.3%	10.1%	33.0%	30.9%
		Median	16.7%	2.3%	11.6%	35.4%	21.6%
	Level 2	Number	5	5	3	3	6
		Minimum	6.1%	51.2%	0.0%	47.6%	0.8%
		Maximum	39.1%	86.3%	15.4%	77.4%	52.2%
		Mean	28.2%	72.5%	7.7%	65.2%	31.5%
		Median	30.9%	72.7%	7.6%	70.6%	35.2%
	Level 3	Number	6	4	7	4	9
		Minimum	3.7%	75.7%	3.6%	64.2%	0.3%
		Maximum	25.4%	106.0%	106.7%	129.5%	105.7%
		Mean	15.4%	89.6%	35.1%	87.7%	26.6%
		Median	13.6%	88.3%	16.5%	78.6%	22.2%
	Level 4	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Soil	Number	35	11	13	10	35
		Minimum	1.5%	0.9%	0.0%	13.2%	0.1%
		Maximum	39.1%	106.0%	106.7%	129.5%	105.7%
		Mean	18.8%	66.0%	23.0%	64.6%	29.9%
		Median	17.0%	75.7%	12.4%	67.4%	22.3%

Table E-1. Evaluation of Accuracy - Relative Percent Difference versus Reference Laboratory Data Calculated for the Xcalibur ElvaX (Continued)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
			Ref Lab	ERA Spike							
Sediment	Level 1	Number	3	3	17	3	6	8	3	12	3
		Minimum	30.6%	4.1%	1.9%	11.8%	4.8%	4.6%	10.9%	5.7%	80.5%
		Maximum	45.5%	100.0%	104.0%	44.3%	30.7%	101.8%	73.9%	121.2%	103.7%
		Mean	39.9%	38.5%	55.9%	23.4%	19.4%	43.0%	34.7%	67.0%	91.3%
		Median	43.8%	11.5%	42.5%	14.0%	20.6%	33.0%	19.2%	68.1%	89.8%
	Level 2	Number	4	4	4	4	3	4	19	3	4
		Minimum	27.0%	2.6%	2.2%	22.5%	34.0%	1.2%	1.2%	42.5%	70.9%
		Maximum	55.7%	18.8%	52.7%	37.1%	35.9%	8.3%	34.7%	66.4%	106.7%
		Mean	39.9%	9.9%	28.7%	29.7%	34.8%	4.8%	15.0%	56.2%	84.0%
		Median	38.4%	9.2%	29.9%	29.6%	34.6%	4.8%	14.0%	59.9%	79.2%
	Level 3	Number	3	3	2	3	3	10	4	3	3
		Minimum	59.9%	0.1%	41.1%	5.6%	36.2%	7.3%	16.2%	51.0%	72.3%
		Maximum	63.0%	4.2%	57.3%	21.0%	46.8%	48.7%	30.9%	88.7%	92.4%
		Mean	61.7%	2.3%	49.2%	14.8%	40.2%	26.7%	23.3%	73.7%	84.4%
		Median	62.3%	2.7%	49.2%	17.7%	37.8%	27.2%	23.0%	81.2%	88.5%
	Level 4	Number	--	--	--	--	--	--	6	--	--
		Minimum	--	--	--	--	--	--	12.4%	--	--
		Maximum	--	--	--	--	--	--	44.8%	--	--
		Mean	--	--	--	--	--	--	28.5%	--	--
		Median	--	--	--	--	--	--	30.6%	--	--
	All Sediment	Number	10	10	23	10	12	22	32	18	10
		Minimum	27.0%	0.1%	1.9%	5.6%	4.8%	1.2%	1.2%	5.7%	70.9%
		Maximum	63.0%	100.0%	104.0%	44.3%	46.8%	101.8%	73.9%	121.2%	106.7%
		Mean	46.4%	16.2%	50.6%	23.3%	28.5%	28.7%	20.4%	66.3%	86.3%
		Median	45.6%	6.2%	42.5%	21.7%	32.4%	25.1%	17.6%	66.9%	84.5%

Table E-1. Evaluation of Accuracy - Relative Percent Difference versus Reference Laboratory Data Calculated for the Xcalibur ElvaX (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Sediment	Level 1	Number	18	3	4	0	17
		Minimum	3.6%	6.5%	16.1%	NC	2.1%
		Maximum	73.9%	59.5%	57.1%	NC	70.9%
		Mean	26.3%	28.7%	42.6%	NC	34.6%
		Median	22.1%	20.0%	48.6%	NC	33.9%
	Level 2	Number	6	4	4	3	5
		Minimum	2.9%	42.6%	15.5%	55.1%	34.9%
		Maximum	22.8%	110.0%	51.1%	87.0%	63.4%
		Mean	15.3%	72.0%	31.9%	72.1%	47.8%
		Median	17.4%	67.7%	30.4%	74.2%	50.8%
	Level 3	Number	4	3	3	3	4
		Minimum	1.2%	91.7%	50.3%	75.8%	18.6%
		Maximum	36.0%	98.5%	93.9%	93.1%	60.1%
		Mean	18.5%	95.9%	70.8%	84.1%	35.0%
		Median	18.5%	97.4%	68.3%	83.3%	30.6%
	Level 4	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Sediment	Number	28	10	11	6	26
		Minimum	1.2%	6.5%	15.5%	55.1%	2.1%
		Maximum	73.9%	110.0%	93.9%	93.1%	70.9%
		Mean	22.8%	66.2%	46.4%	78.1%	37.2%
		Median	20.0%	69.1%	50.3%	79.5%	37.2%

Table E-1. Evaluation of Accuracy - Relative Percent Difference versus Reference Laboratory Data Calculated for the Xcalibur ElvaX (Continued)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
			Ref Lab	ERA Spike							
All Samples	Xcalibur ElvaX	Number	27	14	46	26	41	48	70	42	24
		Minimum	2.6%	0.1%	1.9%	0.2%	2.3%	1.2%	0.3%	0.5%	22.5%
		Maximum	105.4%	154.4%	163.0%	44.3%	87.2%	111.1%	73.9%	121.2%	140.5%
		Mean	44.9%	23.6%	49.1%	18.3%	30.6%	37.3%	21.1%	54.0%	88.2%
		Median	45.5%	6.2%	44.2%	16.3%	30.7%	25.1%	19.5%	54.6%	90.7%
All Samples	All Instruments	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 Difference versus Reference Laboratory Data Calculated for the Xcalibur ElvaX (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
All Samples	Xcalibur ElvaX	Number	63	21	24	16	61
		Minimum	1.2%	0.9%	0.0%	13.2%	0.1%
		Maximum	73.9%	110.0%	106.7%	129.5%	105.7%
		Mean	20.6%	66.1%	33.7%	69.6%	33.0%
		Median	17.5%	75.7%	18.3%	74.0%	32.5%
All Samples	All Instruments	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%

Table E-1. Evaluation of Accuracy - Relative Percent Difference versus Reference Laboratory Data Calculated for the Xcalibur ElvaX (Continued)

Notes:

All RPDs presented in this table are absolute values.

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

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for the Xcalibur ElvaX

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
Soil	Low	Number	9	15	7	23	16	5	7	5
		Minimum	3.0%	6.4%	4.7%	6.5%	2.0%	1.8%	1.9%	26.2%
		Maximum	24.2%	51.3%	35.2%	48.5%	20.5%	7.7%	25.2%	137.1%
		Mean	10.7%	21.4%	20.8%	23.4%	7.4%	4.8%	9.3%	54.3%
		Median	6.7%	16.1%	20.5%	20.9%	6.2%	4.4%	7.0%	37.7%
	Medium	Number	5	4	7	4	8	13	4	7
		Minimum	1.6%	5.9%	1.8%	2.5%	1.2%	0.7%	3.5%	7.1%
		Maximum	11.0%	34.7%	9.4%	14.1%	11.3%	11.7%	23.9%	18.1%
		Mean	5.3%	14.0%	4.1%	6.9%	5.2%	3.5%	9.0%	13.3%
		Median	3.0%	7.7%	2.2%	5.5%	3.8%	2.7%	4.2%	12.6%
	High	Number	3	4	2	2	2	13	8	2
		Minimum	1.8%	3.5%	4.5%	1.7%	6.0%	1.5%	0.7%	4.6%
		Maximum	19.0%	21.8%	4.5%	3.2%	19.5%	15.5%	19.3%	8.0%
		Mean	8.1%	14.3%	4.5%	2.4%	12.8%	5.5%	5.0%	6.3%
		Median	3.6%	16.0%	4.5%	2.4%	12.8%	3.8%	2.6%	6.3%
	Very High	Number	--	--	--	--	--	7	5	--
		Minimum	--	--	--	--	--	2.8%	1.1%	--
		Maximum	--	--	--	--	--	12.2%	15.8%	--
		Mean	--	--	--	--	--	5.7%	8.7%	--
		Median	--	--	--	--	--	4.5%	10.4%	--
	All Soil	Number	17	23	16	29	26	38	24	14
		Minimum	1.6%	3.5%	1.8%	1.7%	1.2%	0.7%	0.7%	4.6%
		Maximum	24.2%	51.3%	35.2%	48.5%	20.5%	15.5%	25.2%	137.1%
		Mean	8.6%	18.9%	11.4%	19.7%	7.1%	4.8%	7.7%	26.9%
		Median	6.5%	14.8%	6.3%	17.7%	6.0%	4.1%	4.6%	15.7%

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

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Low	Number	24	2	3	3	20
		Minimum	2.9%	18.9%	8.6%	43.4%	1.8%
		Maximum	37.7%	35.1%	21.4%	52.3%	24.4%
		Mean	14.3%	27.0%	13.8%	48.4%	9.4%
		Median	10.3%	27.0%	11.5%	49.4%	8.1%
	Medium	Number	5	5	3	3	6
		Minimum	2.6%	2.0%	7.2%	7.7%	0.4%
		Maximum	17.7%	13.6%	10.9%	40.8%	7.3%
		Mean	8.1%	6.5%	8.5%	23.9%	3.2%
		Median	6.3%	5.2%	7.3%	23.3%	2.3%
	High	Number	6	4	7	4	9
		Minimum	1.6%	5.8%	7.9%	13.5%	0.3%
		Maximum	6.8%	9.1%	23.8%	42.0%	10.8%
		Mean	3.5%	7.9%	16.8%	28.3%	4.0%
		Median	2.6%	8.3%	19.2%	28.9%	1.8%
	Very High	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Soil	Number	35	11	13	10	35
		Minimum	1.6%	2.0%	7.2%	7.7%	0.3%
		Maximum	37.7%	35.1%	23.8%	52.3%	24.4%
		Mean	11.6%	10.7%	14.2%	33.0%	7.0%
		Median	7.9%	8.5%	11.5%	39.9%	6.4%

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

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
Sediment	Low	Number	3	17	3	6	8	3	12	3
		Minimum	3.8%	6.3%	7.4%	9.0%	2.3%	3.4%	2.9%	35.5%
		Maximum	9.0%	41.3%	16.0%	37.2%	18.1%	9.8%	47.0%	44.0%
		Mean	6.0%	19.2%	10.5%	27.0%	7.8%	6.0%	16.2%	40.8%
		Median	5.1%	18.5%	8.1%	30.0%	6.1%	4.9%	12.5%	42.8%
	Medium	Number	4	4	4	3	4	19	3	4
		Minimum	3.5%	3.7%	3.6%	4.7%	1.9%	0.4%	1.3%	1.9%
		Maximum	10.7%	7.2%	7.0%	16.0%	3.3%	8.2%	3.9%	12.1%
		Mean	6.7%	5.0%	5.4%	8.7%	2.4%	2.5%	2.8%	6.5%
		Median	6.3%	4.6%	5.5%	5.4%	2.3%	2.2%	3.2%	5.9%
	High	Number	3	2	3	3	10	4	3	3
		Minimum	1.6%	1.4%	2.3%	3.7%	2.3%	0.9%	2.2%	3.0%
		Maximum	13.2%	2.9%	7.9%	5.7%	13.1%	9.8%	6.5%	5.2%
		Mean	6.1%	2.2%	4.5%	4.8%	5.6%	3.9%	3.8%	4.5%
		Median	3.4%	2.2%	3.2%	5.0%	4.3%	2.4%	2.7%	5.2%
	Very High	Number	--	--	--	--	--	6	--	--
		Minimum	--	--	--	--	--	1.4%	--	--
		Maximum	--	--	--	--	--	6.2%	--	--
		Mean	--	--	--	--	--	3.3%	--	--
		Median	--	--	--	--	--	3.2%	--	--
	All Sediment	Number	10	23	10	12	22	32	18	10
		Minimum	1.6%	1.4%	2.3%	3.7%	1.9%	0.4%	1.3%	1.9%
		Maximum	13.2%	41.3%	16.0%	37.2%	18.1%	9.8%	47.0%	44.0%
		Mean	6.3%	15.2%	6.6%	16.9%	5.8%	3.1%	11.9%	16.1%
		Median	5.2%	14.1%	6.5%	12.5%	4.3%	2.5%	6.4%	6.5%

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

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Sediment	Low	Number	18	3	4	0	17
		Minimum	0.9%	16.0%	2.2%	NC	1.2%
		Maximum	35.4%	48.6%	13.6%	NC	31.5%
		Mean	11.1%	27.3%	8.6%	NC	10.2%
		Median	9.9%	17.3%	9.4%	NC	8.3%
	Medium	Number	6	4	4	3	5
		Minimum	4.0%	2.3%	3.4%	7.2%	1.1%
		Maximum	10.5%	11.4%	15.5%	30.0%	5.2%
		Mean	6.4%	8.2%	7.5%	21.0%	3.2%
		Median	5.9%	9.6%	5.6%	25.7%	3.3%
	High	Number	4	3	3	3	4
		Minimum	1.1%	2.8%	2.0%	4.4%	1.3%
		Maximum	8.7%	6.1%	13.8%	16.9%	2.1%
		Mean	3.4%	4.2%	6.3%	9.0%	1.7%
		Median	1.8%	3.6%	3.2%	5.6%	1.8%
	Very High	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Sediment	Number	28	10	11	6	26
		Minimum	0.9%	2.3%	2.0%	4.4%	1.1%
		Maximum	35.4%	48.6%	15.5%	30.0%	31.5%
		Mean	9.0%	12.7%	7.6%	15.0%	7.6%
		Median	8.2%	9.6%	7.0%	12.1%	5.8%

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

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
All Samples	Xcalibur ElvaX	Number	27	46	26	41	48	70	42	24
		Minimum	1.6%	1.4%	1.8%	1.7%	1.2%	0.4%	0.7%	1.9%
		Maximum	24.2%	51.3%	35.2%	48.5%	20.5%	15.5%	47.0%	137.1%
		Mean	7.8%	17.1%	9.6%	18.9%	6.5%	4.0%	9.5%	22.4%
		Median	5.4%	14.1%	6.5%	17.2%	4.9%	3.2%	6.0%	12.5%
All Samples	All Instruments	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 the Xcalibur ElvaX (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
All Samples	Xcalibur ElvaX	Number	63	21	24	16	61
		Minimum	0.9%	2.0%	2.0%	4.4%	0.3%
		Maximum	37.7%	48.6%	23.8%	52.3%	31.5%
		Mean	10.4%	11.7%	11.2%	26.2%	7.2%
		Median	8.0%	8.9%	9.4%	24.5%	6.1%
All Samples	All Instruments	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.
Conc	Concentration.
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 Interferent Elements on RPDs (Accuracy) of Other Target Elements[1]

Parameter	Statistic	Lead Effects on Arsenic			Copper Effects on Nickel			Nickel Effects on Copper		
Interferent/Element Ratio		<5	5 - 10	>10	<5	5 - 10	>10	<5	5 - 10	>10
Number of Samples		29	7	10	44	5	14	39	1	8
RPD of Target Element[2]	Minimum	-163.0%	-33.7%	-104.0%	-24.1%	-3.6%	-73.9%	-108.7%	70.7%	-111.1%
	Maximum	8.1%	22.2%	53.8%	39.1%	33.9%	27.7%	53.0%	70.7%	32.4%
	Mean	-55.0%	-11.7%	-22.9%	14.0%	12.1%	-17.3%	-22.2%	70.7%	-40.3%
	Median	-45.1%	-9.7%	-31.9%	16.7%	15.7%	-21.7%	-11.2%	70.7%	-44.9%
RPD of Target Element (Absolute Value)[2]	Minimum	2.2%	8.4%	1.9%	1.2%	2.9%	4.0%	1.2%	70.7%	22.8%
	Maximum	163.0%	33.7%	104.0%	39.1%	33.9%	73.9%	108.7%	70.7%	111.1%
	Mean	55.5%	18.0%	52.2%	18.4%	14.7%	29.6%	31.4%	70.7%	61.7%
	Median	45.1%	21.5%	50.6%	17.2%	15.7%	27.6%	21.0%	70.7%	49.4%
Interferent Concentration Range	Minimum	ND	1163	2326	ND	852	682	ND	307	1070
	Maximum	1314	54373	38476	1085	2137	9436	629	307	2644
	Mean	299	19071	8482	193	1372	3473	163	307	1888
	Median	152	8802	4699	73	1143	2298	128	307	1793
Target Element Concentration Range	Minimum	67	163	59	48	78	58	29	37	60
	Maximum	4480	6033	2434	2644	244	629	9436	37	438
	Mean	452	2068	310	476	152	172	1669	37	213
	Median	178	1129	70	146	131	122	852	37	122

Table E-4. Evaluation of the Effects of Interferent Elements on RPDs (Accuracy) of Other Target Elements[1] (Continued)

Parameter	Statistic	Zinc Effects on Copper			Copper Effects on Zinc		
Interferent/Element Ratio		<5	5 - 10	>10	<5	5 - 10	>10
Number of Samples		35	2	11	49	3	9
RPD of Target Element[2]	Minimum	-64.4%	-9.9%	-111.1%	-80.6%	-47.1%	-65.0%
	Maximum	70.7%	6.1%	-23.3%	105.7%	70.9%	56.2%
	Mean	-4.2%	-1.9%	-87.9%	-24.4%	13.8%	-0.4%
	Median	-7.3%	-1.9%	-99.1%	-25.3%	17.6%	2.3%
RPD of Target Element (Absolute Value)[2]	Minimum	1.2%	6.1%	23.3%	0.1%	17.6%	2.3%
	Maximum	70.7%	9.9%	111.1%	105.7%	70.9%	65.0%
	Mean	23.0%	8.0%	87.9%	32.0%	45.2%	34.7%
	Median	21.0%	8.0%	99.1%	32.5%	47.1%	31.3%
Interferent Concentration Range	Minimum	ND	1085	1384	ND	634	1818
	Maximum	7370	9253	5331	2205	2187	9436
	Mean	1267	5169	3447	386	1302	4692
	Median	177	5169	3528	177	1085	3670
Target Element Concentration Range	Minimum	29	177	104	36	95	92
	Maximum	9436	1374	639	9253	165	562
	Mean	1760	775	336	1944	120	183
	Median	909	775	332	1085	99	103

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		Cadmium	
				Reference Laboratory		Certified Value		Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	--	--	1	1	3	3
			Minimum	--	--	--	--	-82.6%	82.6%	-13.1%	0.2%
			Maximum	--	--	--	--	-82.6%	82.6%	25.0%	25.0%
			Mean	--	--	--	--	-82.6%	82.6%	4.0%	12.8%
			Median	--	--	--	--	-82.6%	82.6%	0.2%	13.1%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	1	1	7	7	5	5
			Minimum	-2.7%	2.7%	6.3%	6.3%	-63.7%	8.4%	-21.6%	5.3%
			Maximum	71.9%	71.9%	6.3%	6.3%	43.3%	63.7%	5.3%	21.6%
			Mean	23.6%	24.9%	6.3%	6.3%	-19.9%	32.3%	-9.8%	11.9%
			Median	12.6%	12.6%	6.3%	6.3%	-22.1%	33.7%	-10.1%	10.1%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	2	2	1	1	2	2
			Minimum	67.6%	67.6%	6.2%	6.2%	-66.2%	66.2%	-23.0%	0.4%
			Maximum	70.2%	70.2%	154.4%	154.4%	-66.2%	66.2%	-0.4%	23.0%
			Mean	68.9%	68.9%	80.3%	80.3%	-66.2%	66.2%	-11.7%	11.7%
			Median	68.9%	68.9%	80.3%	80.3%	-66.2%	66.2%	-11.7%	11.7%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges.)	Number	1	1	--	--	--	--	--	--
			Minimum	48.2%	48.2%	--	--	--	--	--	--
			Maximum	48.2%	48.2%	--	--	--	--	--	--
			Mean	48.2%	48.2%	--	--	--	--	--	--
			Median	48.2%	48.2%	--	--	--	--	--	--

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

Matrix	Site	Matrix Description	Statistic	Chromium Reference Laboratory RPD	Chromium Reference Laboratory RPD ABS Val	Copper Reference Laboratory RPD	Copper Reference Laboratory RPD ABS Val	Iron Reference Laboratory RPD	Iron Reference Laboratory RPD ABS Val	Lead Reference Laboratory RPD	Lead Reference Laboratory RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	2	2	3	3	3	3	3	3
			Minimum	6.1%	6.1%	-86.3%	59.7%	1.3%	1.3%	-67.0%	40.2%
			Maximum	11.6%	11.6%	-59.7%	86.3%	31.2%	31.2%	-40.2%	67.0%
			Mean	8.8%	8.8%	-76.5%	76.5%	17.4%	17.4%	-51.8%	51.8%
			Median	8.8%	8.8%	-83.7%	83.7%	19.7%	19.7%	-48.1%	48.1%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	5	5	6	6	7	7	6	6
			Minimum	31.6%	31.6%	-111.1%	2.0%	-10.3%	1.1%	-50.5%	10.9%
			Maximum	58.1%	58.1%	30.4%	111.1%	33.9%	33.9%	10.9%	50.5%
			Mean	42.1%	42.1%	-17.3%	28.5%	11.3%	14.8%	-31.7%	35.3%
			Median	43.2%	43.2%	-6.6%	12.1%	5.7%	10.3%	-43.8%	43.8%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	3	3	3	3	2	2
			Minimum	21.2%	21.2%	-72.5%	1.3%	-2.8%	1.9%	-71.2%	32.2%
			Maximum	21.2%	21.2%	1.3%	72.5%	19.8%	19.8%	-32.2%	71.2%
			Mean	21.2%	21.2%	-30.7%	31.6%	6.3%	8.2%	-51.7%	51.7%
			Median	21.2%	21.2%	-21.0%	21.0%	1.9%	2.8%	-51.7%	51.7%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges.)	Number	4	4	2	2	6	6	6	6
			Minimum	-20.1%	4.8%	15.7%	15.7%	-19.2%	0.3%	-43.0%	0.5%
			Maximum	8.9%	20.1%	22.8%	22.8%	46.2%	46.2%	8.3%	43.0%
			Mean	-5.5%	10.0%	19.2%	19.2%	8.2%	18.3%	-5.6%	10.8%
			Median	-5.4%	7.5%	19.2%	19.2%	3.9%	15.1%	0.8%	5.3%

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

Matrix	Site	Matrix Description	Statistic	Mercury Reference Laboratory RPD	Mercury Reference Laboratory RPD ABS Val	Nickel Reference Laboratory RPD	Nickel Reference Laboratory RPD ABS Val	Selenium Reference Laboratory RPD	Selenium Reference Laboratory RPD ABS Val	Silver Reference Laboratory RPD	Silver Reference Laboratory RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	3	3	1	1	1	1
			Minimum	--	--	-39.1%	9.3%	-68.5%	68.5%	-16.5%	16.5%
			Maximum	--	--	25.8%	39.1%	-68.5%	68.5%	-16.5%	16.5%
			Mean	--	--	-1.3%	24.7%	-68.5%	68.5%	-16.5%	16.5%
			Median	--	--	9.3%	25.8%	-68.5%	68.5%	-16.5%	16.5%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	1	1	6	6	2	2	4	4
			Minimum	-26.6%	26.6%	-9.8%	9.8%	-75.7%	51.2%	-106.7%	0.0%
			Maximum	-26.6%	26.6%	27.7%	27.7%	-51.2%	75.7%	0.0%	106.7%
			Mean	-26.6%	26.6%	16.5%	19.8%	-63.4%	63.4%	-29.2%	29.2%
			Median	-26.6%	26.6%	20.1%	20.1%	-63.4%	63.4%	-5.1%	5.1%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	3	3	2	2	2	2
			Minimum	90.4%	90.4%	-14.8%	14.8%	-72.7%	0.9%	-15.4%	11.6%
			Maximum	107.9%	107.9%	30.9%	30.9%	0.9%	72.7%	-11.6%	15.4%
			Mean	99.2%	99.2%	14.6%	24.4%	-35.9%	36.8%	-13.5%	13.5%
			Median	99.2%	99.2%	27.6%	27.6%	-35.9%	36.8%	-13.5%	13.5%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges.)	Number	--	--	3	3	--	--	--	--
			Minimum	--	--	-9.8%	1.5%	--	--	--	--
			Maximum	--	--	12.0%	12.0%	--	--	--	--
			Mean	--	--	0.2%	7.8%	--	--	--	--
			Median	--	--	-1.5%	9.8%	--	--	--	--

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

Matrix	Site	Matrix Description	Statistic	Vanadium Reference Laboratory RPD	Vanadium Reference Laboratory RPD ABS Val	Zinc Reference Laboratory RPD	Zinc Reference Laboratory RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	3	3
			Minimum	--	--	-65.0%	0.8%
			Maximum	--	--	105.7%	105.7%
			Mean	--	--	13.8%	57.1%
			Median	--	--	0.8%	65.0%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	2	2	7	7
			Minimum	77.4%	77.4%	-61.6%	8.3%
			Maximum	129.5%	129.5%	-8.3%	61.6%
			Mean	103.4%	103.4%	-36.8%	36.8%
			Median	103.4%	103.4%	-33.4%	33.4%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	3	3
			Minimum	70.6%	70.6%	-80.6%	15.2%
			Maximum	70.6%	70.6%	-15.2%	80.6%
			Mean	70.6%	70.6%	-49.4%	49.4%
			Median	70.6%	70.6%	-52.2%	52.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges.)	Number	--	--	2	2
			Minimum	--	--	-57.3%	47.1%
			Maximum	--	--	-47.1%	57.3%
			Mean	--	--	-52.2%	52.2%
			Median	--	--	-52.2%	52.2%

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

Matrix	Site	Matrix Description	Statistic	Antimony Reference Laboratory RPD	Antimony Reference Laboratory RPD ABS Val	Antimony Certified Value RPD	Antimony Certified Value RPD ABS Val	Arsenic Reference Laboratory RPD	Arsenic Reference Laboratory RPD ABS Val	Cadmium Reference Laboratory RPD	Cadmium Reference Laboratory RPD ABS Val
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	4	4	12	12	4	4
			Minimum	35.9%	35.9%	-108.4%	9.9%	-31.8%	1.2%	-97.3%	59.2%
			Maximum	49.6%	49.6%	32.4%	108.4%	73.9%	73.9%	-59.2%	97.3%
			Mean	42.2%	42.2%	-15.1%	44.0%	16.5%	27.5%	-81.2%	81.2%
			Median	41.6%	41.6%	7.8%	28.9%	17.3%	28.4%	-84.2%	84.2%
Sediment	RF	Silty fine sand (tailings)	Number	4	4	4	4	12	12	5	5
			Minimum	-43.8%	43.8%	-2.6%	0.1%	-88.4%	25.7%	-37.1%	11.8%
			Maximum	63.0%	63.0%	98.9%	98.9%	-25.7%	88.4%	-11.8%	37.1%
			Mean	34.3%	56.2%	24.8%	26.1%	-44.8%	44.8%	-22.0%	22.0%
			Median	59.0%	59.0%	1.4%	2.7%	-41.8%	41.8%	-21.0%	21.0%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	6	6	--	--	5	5	1	1
			Minimum	-68.5%	2.6%	--	--	-163.0%	39.2%	-16.3%	16.3%
			Maximum	43.9%	68.5%	--	--	-39.2%	163.0%	-16.3%	16.3%
			Mean	9.7%	32.5%	--	--	-76.5%	76.5%	-16.3%	16.3%
			Median	23.0%	32.9%	--	--	-48.6%	48.6%	-16.3%	16.3%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	3	3	3	2	2	2	2
			Minimum	-45.7%	27.0%	10.2%	10.2%	-70.4%	1.9%	-34.3%	14.0%
			Maximum	-27.0%	45.7%	18.8%	18.8%	1.9%	70.4%	-14.0%	34.3%
			Mean	-39.4%	39.4%	13.5%	13.5%	-34.3%	36.2%	-24.1%	24.1%
			Median	-45.5%	45.5%	11.5%	11.5%	-34.3%	36.2%	-24.1%	24.1%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	3	--	--	7	7	3	3
			Minimum	-105.4%	37.6%	--	--	-66.4%	8.4%	-29.4%	12.2%
			Maximum	-37.6%	105.4%	--	--	47.4%	66.4%	-12.2%	29.4%
			Mean	-63.3%	63.3%	--	--	-14.3%	34.2%	-18.7%	18.7%
			Median	-47.0%	47.0%	--	--	-18.6%	22.2%	-14.7%	14.7%
	All	Samples	Number	27	27	14	14	46	46	26	26
			Minimum	-105.4%	2.6%	-2.6%	0.1%	-163.0%	1.9%	-44.3%	0.2%
			Maximum	77.4%	105.4%	154.4%	154.4%	53.8%	163.0%	25.0%	44.3%
			Mean	11.3%	44.9%	23.0%	23.5%	-41.4%	49.1%	-15.9%	18.3%
			Median	14.9%	45.5%	6.2%	6.2%	-41.0%	44.2%	-15.5%	16.3%

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

Matrix	Site	Matrix Description	Statistic	Chromium Reference Laboratory RPD	Chromium Reference Laboratory RPD ABS Val	Copper Reference Laboratory RPD	Copper Reference Laboratory RPD ABS Val	Iron Reference Laboratory RPD	Iron Reference Laboratory RPD ABS Val	Lead Reference Laboratory RPD	Lead Reference Laboratory RPD ABS Val
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	3	3	11	11	5	5	4	4
			Minimum	70.9%	70.9%	-24.1%	3.7%	-97.4%	6.5%	-68.3%	3.6%
			Maximum	121.0%	121.0%	39.1%	39.1%	6.5%	97.4%	3.6%	68.3%
			Mean	93.5%	93.5%	8.9%	18.7%	-68.8%	71.3%	-27.4%	29.1%
			Median	88.5%	88.5%	11.5%	14.3%	-84.1%	84.1%	-22.4%	22.4%
Sediment	RF	Silty fine sand (tailings)	Number	8	8	13	13	13	13	10	10
			Minimum	11.7%	11.7%	-101.8%	1.2%	-5.3%	5.0%	-76.3%	42.5%
			Maximum	37.8%	37.8%	70.7%	101.8%	28.8%	28.8%	-42.5%	76.3%
			Mean	29.8%	29.8%	-11.0%	28.5%	13.9%	15.5%	-58.6%	58.6%
			Median	32.4%	32.4%	-8.3%	11.6%	16.2%	16.2%	-59.0%	59.0%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	10	10	4	4	12	12	--	--
			Minimum	-34.8%	9.0%	-108.7%	22.8%	3.2%	3.2%	--	--
			Maximum	87.2%	87.2%	53.0%	108.7%	44.0%	44.0%	--	--
			Mean	21.8%	28.8%	-33.0%	70.9%	29.7%	29.7%	--	--
			Median	19.5%	22.9%	-38.1%	76.1%	33.7%	33.7%	--	--
Sediment	TL	Silt and clay (slag-enriched)	Number	1	1	7	7	7	7	4	4
			Minimum	15.6%	15.6%	-48.7%	18.2%	-34.7%	1.9%	-121.2%	66.4%
			Maximum	15.6%	15.6%	-18.2%	48.7%	15.7%	34.7%	-66.4%	121.2%
			Mean	15.6%	15.6%	-32.1%	32.1%	-9.0%	18.2%	-95.1%	95.1%
			Median	15.6%	15.6%	-28.6%	28.6%	-1.9%	15.7%	-96.4%	96.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	6	6	6	6	7	7	7	7
			Minimum	-41.9%	2.3%	-105.3%	5.4%	-16.4%	16.4%	-88.5%	22.3%
			Maximum	80.4%	80.4%	9.2%	105.3%	32.4%	32.4%	-22.3%	88.5%
			Mean	28.5%	42.4%	-28.3%	33.4%	19.3%	24.0%	-63.3%	63.3%
			Median	30.1%	38.7%	-7.6%	9.5%	23.8%	23.8%	-70.5%	70.5%
	All	Samples	Number	41	41	48	48	70	70	42	42
			Minimum	-41.9%	2.3%	-111.1%	1.2%	-34.7%	0.3%	-121.2%	0.5%
			Maximum	87.2%	87.2%	70.7%	111.1%	73.9%	73.9%	10.9%	121.2%
			Mean	25.3%	30.6%	-23.3%	37.3%	14.4%	21.1%	-52.8%	54.0%
			Median	28.1%	30.7%	-11.4%	25.1%	17.6%	19.5%	-54.6%	54.6%

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

Matrix	Site	Matrix Description	Statistic	Mercury		Nickel		Selenium		Silver	
				Reference Laboratory		Reference Laboratory		Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	3	3	11	11	5	5	4	4
			Minimum	70.9%	70.9%	-24.1%	3.7%	-97.4%	6.5%	-68.3%	3.6%
			Maximum	121.0%	121.0%	39.1%	39.1%	6.5%	97.4%	3.6%	68.3%
			Mean	93.5%	93.5%	8.9%	18.7%	-68.8%	71.3%	-27.4%	29.1%
			Median	88.5%	88.5%	11.5%	14.3%	-84.1%	84.1%	-22.4%	22.4%
Sediment	RF	Silty fine sand (tailings)	Number	5	5	13	13	3	3	4	4
			Minimum	79.3%	79.3%	-2.9%	1.2%	-98.5%	56.8%	-93.9%	15.5%
			Maximum	106.7%	106.7%	34.5%	34.5%	-56.8%	98.5%	-15.5%	93.9%
			Mean	89.7%	89.7%	17.7%	18.1%	-82.3%	82.3%	-53.6%	53.6%
			Median	89.8%	89.8%	19.9%	19.9%	-91.7%	91.7%	-52.6%	52.6%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	10	10	11	11	3	3	1	1
			Minimum	-133.5%	22.5%	-7.5%	7.5%	-86.5%	3.7%	-75.8%	75.8%
			Maximum	140.5%	140.5%	35.4%	35.4%	3.7%	86.5%	-75.8%	75.8%
			Mean	64.0%	90.7%	19.0%	20.4%	-56.4%	58.8%	-75.8%	75.8%
			Median	92.4%	96.2%	17.5%	17.5%	-86.3%	86.3%	-75.8%	75.8%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	3	6	6	4	4	4	4
			Minimum	72.3%	72.3%	-73.9%	3.6%	-110.0%	20.0%	-57.1%	32.1%
			Maximum	103.7%	103.7%	-3.6%	73.9%	-20.0%	110.0%	-32.1%	57.1%
			Mean	85.0%	85.0%	-35.5%	35.5%	-58.0%	58.0%	-45.7%	45.7%
			Median	79.1%	79.1%	-36.4%	36.4%	-51.1%	51.1%	-46.8%	46.8%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	--	7	7	1	1	4	4
			Minimum	--	--	-37.3%	4.0%	-106.0%	106.0%	-20.2%	10.5%
			Maximum	--	--	23.2%	37.3%	-106.0%	106.0%	-10.5%	20.2%
			Mean	--	--	-4.4%	18.6%	-106.0%	106.0%	-14.8%	14.8%
			Median	--	--	4.0%	16.3%	-106.0%	106.0%	-14.3%	14.3%
	All	Samples	Number	24	24	63	63	21	21	24	24
			Minimum	-133.5%	22.5%	-73.9%	1.2%	-110.0%	0.9%	-106.7%	0.0%
			Maximum	140.5%	140.5%	39.1%	73.9%	6.5%	110.0%	3.6%	106.7%
			Mean	74.8%	88.2%	6.9%	20.6%	-65.0%	66.1%	-33.4%	33.7%
			Median	90.1%	90.7%	13.6%	17.5%	-75.7%	75.7%	-18.3%	18.3%

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

Matrix	Site	Matrix Description	Statistic	Vanadium Reference Laboratory		Zinc Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	5	5	8	8
			Minimum	47.6%	47.6%	-48.4%	2.1%
			Maximum	83.3%	83.3%	48.8%	48.8%
			Mean	65.2%	65.2%	-19.0%	31.2%
			Median	64.2%	64.2%	-27.4%	34.2%
Sediment	RF	Silty fine sand (tailings)	Number	3	3	13	13
			Minimum	74.2%	74.2%	-63.4%	17.6%
			Maximum	93.1%	93.1%	20.3%	63.4%
			Mean	84.8%	84.8%	-33.2%	39.0%
			Median	87.0%	87.0%	-38.4%	38.4%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	3	3	11	11
			Minimum	-35.4%	13.2%	-22.8%	0.1%
			Maximum	73.8%	73.8%	18.9%	22.8%
			Mean	17.2%	40.8%	-3.8%	9.3%
			Median	13.2%	35.4%	-0.3%	7.8%
Sediment	TL	Silt and clay (slag-enriched)	Number	--	--	7	7
			Minimum	--	--	-31.3%	2.3%
			Maximum	--	--	70.9%	70.9%
			Mean	--	--	23.3%	35.2%
			Median	--	--	28.5%	31.3%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	2	2	7	7
			Minimum	50.4%	50.4%	-51.3%	11.8%
			Maximum	83.4%	83.4%	-11.8%	51.3%
			Mean	66.9%	66.9%	-32.5%	32.5%
			Median	66.9%	66.9%	-24.9%	24.9%
	All	Samples	Number	16	16	61	61
			Minimum	-35.4%	13.2%	-80.6%	0.1%
			Maximum	129.5%	129.5%	105.7%	105.7%
			Mean	65.2%	69.6%	-19.0%	33.0%
			Median	74.0%	74.0%	-22.8%	32.5%

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

Site Abbreviations:

AS	Alton Steel Mill
BN	Burlington Northern Railroad/ASARCO East
CN	Naval Surface Warfare Center, Crane Division
KP	KARS Park – Kennedy Space Center
LV	Leviathan Mine/Aspen Creek
RF	Ramsey Flats – Silver Bow Creek
SB	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).