United States
Environmental Protection
Agency

Office of Research and
Development
Washington, DC 20460

EPA/540/R-06/005
February 2006

Innovative Technology Verification Report

XRF Technologies for Measuring Trace Elements in Soil and Sediment

Rontec PicoTAX XRF Analyzer

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Rontec PicoTAX XRF Analyzer

Prepared by

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Contract No. 68-C-00-181
Task Order No. 42

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Notice

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Foreword

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

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

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

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

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

Gary Foley, Ph.D.
Director
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Abstract

The Rontec PicoTAX x-ray fluorescence (XRF) analyzer was demonstrated under the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) Program. The field portion of the demonstration was conducted in January 2005 at the Kennedy Athletic, Recreational and Social Park (KARS) at Kennedy Space Center on Merritt Island, Florida. The demonstration was designed to collect reliable performance and cost data for the PicoTAX 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 PicoTAX 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 PicoTAX is a transportable bench-top device that provides quantitative and semi-quantitative multi-element microanalysis of soils and sediments using total reflection XRF spectroscopy. The spectrometer includes a 40-watt metal-ceramic x-ray tube excitation source and a thermoelectrically cooled silicon drift (Si Drift) x-ray detector. The PicoTAX is capable of detecting up to 75 elements from aluminum to yttrium and from palladium to uranium.

The PicoTAX uses an internal standard for instrument calibration; thus, initial calibration is not required. A solution of internal standard that contains a project-specific element is added to each sample to establish response factors (determined by the software). Element quantitation is determined by comparing the response to the unknown element to the response of the internal standard with a known concentration.

A laptop computer is used to monitor and control all aspects of PicoTAX system operation. Rontec's Quantum software, which is loaded into the laptop computer, calibrates the instrument, handles measurement data and methods, controls all hardware functions, and provides statistical functions, reporting functions, and data and spectra export.

This report describes the results of the evaluation of the PicoTAX 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 PicoTAX 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

Acknowledgements

This report was co-authored by Dr. Greg Swanson and Dr. Mark Colsman of Tetra Tech EM Inc. The authors acknowledge the advice and support of the following individuals in preparing this report: Dr. Stephen Billets and Mr. George Brilis of the U.S. Environmental Protection Agency's National Exposure Research Laboratory; Dr. Hagen Stosnach, Dr. Armin Gross and Ulrick Waldschläger of RÖNTEC AG in Germany, Paul Smith of RÖNTEC USA; and Dr. Jackie Quinn of the National Aeronautics and Space Administration (NASA), Kennedy Space Center (KSC). The demonstration team also acknowledges the field support of Michael Deliz of NASA KSC and Mark Speranza of Tetra Tech NUS, the consultant program manager for NASA.

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 Rontec PicoTAX XRF analyzer based on the results of the demonstration.

1.1 Organization of this Report

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

The second part of this report provides information relevant to the specific instrument that is the subject of this ITVR. This information includes a description of the instrument (Chapter 6), a performance

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 Analytcal, 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 Brooker AXS AXS, 11/2005)	RÖNTEC USA	Rontec	PicoTAX	PicoTAX

evaluation (Chapter 7), a cost analysis (Chapter 8), and a summary of the demonstration results (Chapter 9). References are provided in Chapter 10.

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

1.2 Description of the SITE Program

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

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

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

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

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

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

1.3 Scope of the Demonstration

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

EPA-sponsored demonstrations of XRF technologies have been under way for more than a decade. The first SITE MMT demonstration of XRF occurred in 1995, when six instruments were evaluated for their ability to analyze 10 target elements. The results of this demonstration were published in individual

reports for each instrument (EPA 1996a, 1996b, 1998a, 1998b, 1998c, and 1998d). In 2003, two XRF instruments were included in a demonstration of field methods for analysis of mercury in soil and sediment. Individual ITVRs were also prepared for each of these two instruments (EPA 2004a, 2004b).

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

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

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

1.4 General Description of XRF Technology

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

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

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

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

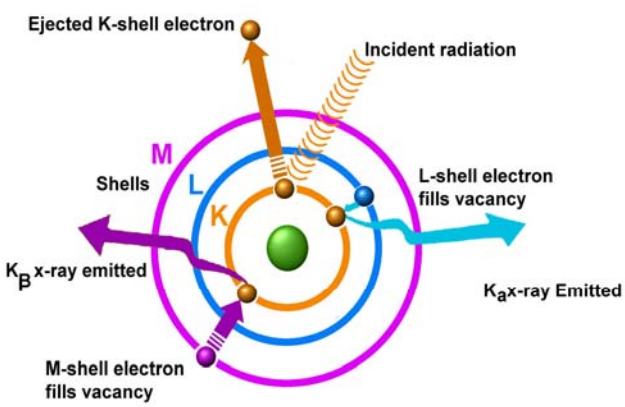

Figure 1-1. The XRF process.

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

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

1. Physical matrix effects (variations in the physical character of the sample).

2. Chemical matrix effects (absorption and enhancement phenomena) and Spectral interferences (peak overlaps).

3. Moisture content above 10 percent, which affects x-ray transmission.

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

1.5 Properties of the Target Elements

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

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

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

1.5.1 Antimony

Naturally occurring antimony in surface soils is typically found at less than 1 to 4 milligrams per kilogram (mg/kg). 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. Concentra-tions of cadmium that exceed 37 mg/kg may result in adverse effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 50 mg/kg. Elevated concentrations of cadmium are associated with mine wastes and industrial facilities. Cadmium is successfully analyzed by both ICP-AES and field-portable XRF; however, action levels for cadmium may be lower than the detection limits of field-portable XRF instruments.

1.5.4 Chromium

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

1.5.5 Copper

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

1.5.6 Iron

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

often used as a target reference element in XRF analysis.

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

1.5.7 Lead

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

1.5.8 Mercury

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

1.5.9 Nickel

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

1.5.10 Selenium

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

Analytical results for selenium using ICP-AES and XRF are expected to be comparable.

1.5.11 Silver

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

1.5.12 Vanadium

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

1.5.13 Zinc

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

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Chapter 2
Field Sample Collection Locations

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

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

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

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

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

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

2.1 Alton Steel Mill Site

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

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

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

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

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

Notes (in order of appearance in table):

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

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

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

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

2.2 Burlington Northern-ASARCO Smelter Site

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

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

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

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

2.3 Kennedy Athletic, Recreational and Social Park Site

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

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

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

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

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

2.4 Leviathan Mine Site

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

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

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

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

2.5 Navy Surface Warfare Center, Crane Division Site

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

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

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

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

2.6 Ramsay Flats-Silver Bow Creek Site

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

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

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

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

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

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

2.7 Sulphur Bank Mercury Mine

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.

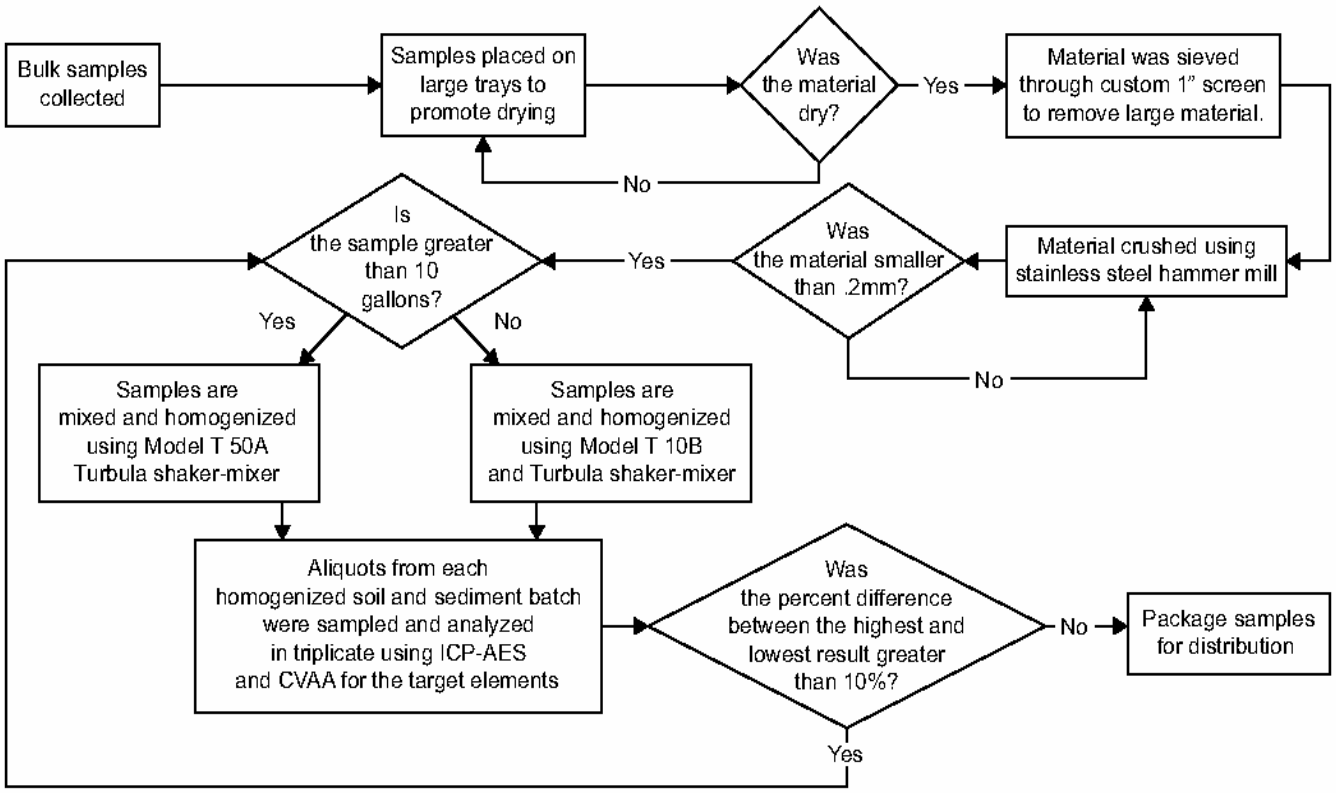

Figure 3-1. Bulk sample processing diagram.

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 PicoTAX XRF analyzer is manufactured by RÖNTEC AG, Berlin, Germany and distributed in the United States by RÖNTEC USA (Rontec). This chapter provides a technical description of the PicoTAX based on information obtained from Rontec and the observation of this analyzer during the field demonstration. This chapter also identifies a Rontec company contact, where additional technical information may be obtained.

6.1 General Description

The PicoTAX is a portable bench-top device that provides quantitative and semi-quantitative multi-element microanalysis of soils and sediments using total reflection x-ray fluorescence spectroscopy. The spectrometer includes a 40-watt metal-ceramic x-ray tube excitation source and a thermoelectrically cooled silicon drift (Si Drift) x-ray detector. The PicoTAX is capable of detecting up to 75 elements from aluminum to yttrium and from palladium to uranium.

The PicoTAX uses an internal standard for instrument calibration; thus, initial calibration is not required. A solution of internal standard that contains a project-specific element is added to each sample to establish response factors (determined by the software). Element quantitation is determined by comparing the response of the unknown element to the response of the internal standard with a known concentration.

A laptop computer is used to monitor and control all aspects of PicoTAX system operation. Rontec's Quantum software, which is loaded into the laptop computer, calibrates the instrument, handles measurement data and methods, controls all hardware functions, and provides statistical functions, reporting functions, and data and spectra export.

Technical specifications for the PicoTAX are presented in Table 6-1. The PicoTAX is shown in the standard bench-top configuration in Figure 6-1.

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

6.2 Instrument Operations during the Demonstration

The PicoTAX spectrometer and accessories were shipped to the demonstration site from Rontec headquarters in Berlin, Germany in packaging that complied with international and customs regulations. A heavy-duty crate with an inner metal liner contained the instrument, necessary tools, and an analytical balance. According to Rontec, a smaller metal and wood box would typically be used for shipment within the United States. The tools and balance would typically be shipped separately. The total weight of the analyzer and accessories was approximately 45 kg.

6.2.1 Set up and Calibration

The PicoTAX was set on a vibration free bench and plugged into a 110-volt (V) electrical outlet. After connecting the instrument to an accompanying laptop personal computer (PC), the PicoTAX software was initialized. The XRF detector was allowed to warm up for 20 to 25 minutes and the optical path of the instrument was inspected. Sample preparation equipment consisted of the analytical balance, a mortar and pestle for sample grinding, test tube rack to hold sample tubes, and reagents for preparation

Table 6-1. Rontec PicoTAX XRF Analyzer Technical Specifications

Weight:	37 kg.
Dimensions:	420 x 590 x 300 mm.
Excitation Source:	40W metal ceramic x-ray tube, Mo-target, air cooled.
X-ray Optics:	Ni/C multilayer, 17.5 keV, 80% reflectivity.
Detector:	XFlash Detector, 10 mm^2, 160 eV FWHM.
Signal Processing:	Digital signal processing unit, data interchange, and control via RS232 interface.
Software:	Modular Quantum software package for instrument control, spectra accumulation, calibration, and quantification.
Element Range:	Elements from aluminum to yttrium and from palladium to uranium (niobium to rhodium are not detectable).
Sample Container:	30 mm quartz disk.
Variants:	PicoTAX Basic with single sample changer. PicoTAX Automatic with automatic changer for 25 sample disks.
Power:	110-220 volts, 50 hertz, 180 watts.

and analysis. The total time for setting up the XRF analyzer and sample preparation equipment was about 30 minutes.

Gallium was selected as the internal standard for this effort. The gallium internal standard solution was added to each sample to establish response factors and quantitatively determine the concentrations of elements in each sample.

6.2.2 Demonstration Sample Processing

Rontec provided a team of three technical staff members from their headquarters in Berlin, Germany to process samples. One staff member prepared samples for analysis, one operated the PicoTAX analyzer, and the third assisted with data management or other tasks.

The typical daily routine for this demonstration involved analyzing 3 batches of 25 samples daily. Of the 25 samples in a batch, 22 were actual samples and the other 3 for QC and performance check samples. Thus, Rontec was able to analyze 66 samples in a 24-hour period using an autosampler to assist in the analysis once operations were standardized. At the beginning of each day, Rontec reviewed the sample analysis completed during the previous night's run. The quartz disks used to contain each sample in the autosampler were cleaned and reused. All samples were prepared and analyzed in accordance with the procedures listed in the PicoTAX instrument manual and application note that was available during the demonstration.

The procedure for preparing soil samples for analysis is described briefly below but is provided in detail in their instrument manual and application note:

- Approximately 150 milligrams (mg) of soil was finely ground to less than 75 microns.
- An amount of approximately 25 mg of the finely ground soil sample was mixed with 2.5 milliliters of Triton X solution to form a soil suspension and then with 40 microliters of gallium standard solution to incorporate the internal standard.
- Cleaned quartz disks were prepared for analytical use by adding a drop of silicon solution to the center of each disk and warming on a hot plate to about 60 °C for about 10 minutes. This procedural step leaves a surface residue of silicon that helps contain the soil suspension for XRF analysis.
- Ten microliters of the soil suspension was dispensed on top of the silicon residue and the disks returned to the hot plate for an additional 10 minutes to dry (Figure 6-2). The final samples for analysis contained circular soil residues on the quartz disks.

Figure 6-2. Quartz disks drying on a hot plate.

- Cooled disks were placed into the autosampler tray, which feeds into the PicoTAX analyzer. Each disk was marked for identification and recorded on a log sheet (Figure 6-3).

Figure 6-3. Rontec technicians recording identification numbers.

An autosampler was used to allow overnight processing of samples through the PicoTAX analyzer. The autosampler had 25 slots for samples; thus, samples were analyzed in a batch of 25 samples that included 22 demonstration samples and 3 QC samples. Each sample was analyzed for 10 minutes for the demonstration, thus requiring over 4 hours to analyze each batch. After the analysis was complete, the software automatically calculated the element concentrations from raw data and provided the results in tabular format (text files or Microsoft Excel® data files). The quartz disks were cleaned and reused for this demonstration; however, disposable acrylic disks may also be used instead.

6.3 General Demonstration Results

The unique sample preparation required for the PicoTAX analyzer took about 5 minutes to complete and occupied one member of the field team essentially full time at the demonstration site. The analysis time in the XRF analyzer was set at 10 minutes for the demonstration, although Rontec indicated that sufficient precision and accuracy could be obtained for soil and sediment samples using shorter analysis times. These factors limited the number of samples that could be processed to three batches of 22 samples (66 samples) per day once the instrument had been set up and overall efficiency had been optimized.

The three-person Rontec field team completed the analysis of 260 samples during 5 full days at the demonstration site (Monday through Friday). Because some supplies did not arrive on Monday, and the team needed to catch an international flight on Saturday morning, the Rontec field team was allowed to take the remaining 66 samples in the demonstration sample set back to Germany for analysis. At the rate the Rontec field team was processing samples during the field demonstration, these remaining samples would have taken 1 full day to process.

6.4 Contact Information

In November 2005, Rontec was acquired by Bruker AXS Inc. Additional information on Rontec's PicoTAX XRF analyzer is available from the following source:

BRUKER AXS Inc.
5465 East Cheryl Parkway
Madison, WI 53711-5373, USA
Telephone: (800) 234-XRAY
Telephone: (608) 276-3000
Fax: (608) 276-3006
Email: info@bruker-axs.com

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Chapter 7
Performance Evaluation

As discussed in Chapter 6, Rontec analyzed 260 of the 326 demonstration samples of soil and sediment at the field demonstration site between January 24 and 28, 2005. Weather delayed Rontec's arrival at the field demonstration on Monday, January 24, and further delayed the receipt of some supplies until Tuesday, January 25. In addition, Rontec's detailed sample preparation process affected sample throughput during the field demonstration and the field team had to catch an international plane flight on Saturday morning, January 29. For these reasons, EPA allowed Rontec to analyze the remaining 66 samples the following week at Rontec's Berlin laboratories. A complete set of electronic data for the PicoTAX in Excel spreadsheet format was delivered to Tetra Tech on March 3, 2005. Because data quality for antimony was anticipated to be poor due to the x-ray tube used in the demonstration (molybdenum), no data were reported for antimony by Rontec. Although results were reported for silver and cadmium, Rontec also anticipated poor accuracy and precision for these two elements because of the x-ray tube used. All the data provided by Rontec are tabulated and compared with the reference laboratory data and the ERA-certified spike concentrations in Appendix D.

The PicoTAX 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. The evaluation and selection of data for the MDL calculation also addressed results reported as "not detected" by Rontec. For many of the MDL blend results, element concentrations were below the statistical lower limits of detection (LLD) calculated by the PicoTAX's instrument algorithms. Non-detect values were reported by Rontec as "<LLD" without additional clarification as to the concentration represented by the LLD. 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 "<LLD" were generally not used. In essence, this meant that all seven replicates had to have detected concentrations, as reported by Rontec, to calculate an MDL for a blend. However, this approach produced very limited or no MDL results for a few elements, so blends for which at least six of the seven replicates were detections were included in the evaluation. In addition, the fourth replicate for MDL Blend 8 and the second replicate for MDL Blend 10 were removed from the evaluation because all results from these two replicates differed significantly from the results for the other replicates of the blend. These replicates may have been inadvertently switched with replicates from other blends. Iron was not included in the MDL evaluation, as was discussed in Section 4.2.1.

The MDLs calculated for the PicoTAX are presented in Table 7-1. As shown, only a single MDL could be calculated for silver, and only three or fewer MDLs could be calculated for cadmium, mercury, and selenium. Eight 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): selenium.
- Low (20 to 50 ppm): arsenic, copper, and vanadium.
- Medium (50 to 100 ppm): mercury, nickel, and zinc.
- High (greater than 100 ppm): cadmium, chromium, lead, and silver.

Mean MDLs greater than 500 ppm were calculated for cadmium and silver; no other mean MDLs were above 110 ppm. The MDLs for cadmium and silver were based on very limited data (one to two blends each) and are somewhat uncertain; however, these findings are in keeping with the developer's prediction of poor performance for these elements. High biases in a few PicoTAX results produced some extremely high MDLs

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

Matrix	Blend No.	Antimony			Arsenic			Cadmium			Chromium		
		Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]
Soil	2	NA	NA	17	NC	ND	1.5	NC	ND	ND	**113**	230	167
Soil	5	NA	NA	ND	**38**	59	47	NC	ND	1.9	**99**	145	121
Soil	6	NA	NA	8	NC	343	477	NC	ND	12	**43**	111	133
Soil	8[5]	NA	NA	118	NC	3596	3,943	**939**	963	91	**153**	56	55
Soil	10[5]	NA	NA	ND	**19**	43	39	NC	ND	0.96	**94**	129	116
Soil	12	NA	NA	62	NC	590	559	**120**	48	263	**79**	120	101
Soil	18	NA	NA	ND	**12**	19	9	NC	ND	ND	**262**	255	150
Sediment	29	NA	NA	ND	**15**	12	10	NC	ND	ND	**39**	46	63
Sediment	31	NA	NA	ND	**7**	6	11	NC	ND	ND	**124**	98	133
Sediment	32	NA	NA	ND	**21**	34	31	NC	ND	ND	**120**	96	75
Sediment	39	NA	NA	ND	**22**	18	14	NC	ND	ND	**76**	97	102
Sediment	65	NA	NA	11	**55**	287	250	NC	ND	44	**106**	340	303
Mean Rontec MDL		**NC**			**23**			**529**			**109**		

Matrix	Blend No.	Copper			Lead			Mercury			Nickel		
		Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]
Soil	2	**11**	46	47	NC	1342	1,200	NC	ND	ND	**48**	112	83
Soil	5	**21**	59	49	**502**	203	78	NC	ND	ND	**35**	47	60
Soil	6	**83**	149	160	NC	4289	3,986	NC	ND	0.83	**32**	42	70
Soil	8[5]	NC	1249	1,243	NC	47306	33,429	NC	ND	15	**51**	31	57
Soil	10[5]	**7**	33	31	**63**	86	72	NC	ND	0.14	**23**	51	60
Soil	12	NC	871	747	NC	5142	4,214	NC	ND	1.8	**98**	88	91
Soil	18	**40**	56	50	**60**	19	17	**153**	46	56	**134**	218	213
Sediment	29	NC	1638	1,986	**31**	29	33	NC	ND	0.24	**73**	59	72
Sediment	31	NC	683	1,514	**44**	36	51	NC	ND	ND	**200**	128	196
Sediment	32	**18**	37	36	**54**	41	26	NC	ND	ND	**66**	113	174
Sediment	39	**36**	95	94	**49**	47	27	NC	ND	ND	**83**	139	202
Sediment	65	**20**	73	69	**36**	32[6]	25	**15**	19[6]	32	**93**	138	214
Mean PicoTAX MDL		**29**			**105**			**84**			**78**		

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

Matrix	Blend No.	Selenium			Silver			Vanadium			Zinc		
		Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]	Rontec MDL[2]	Rontec Conc.[3]	Ref. Lab Conc.[4]
Soil	2	NC	ND	ND	NC	ND	ND	NC	ND	1.2	**6**	19	24
Soil	5	NC	ND	ND	NC	ND	0.93	**42**	63	55	**130**	274	229
Soil	6	NC	ND	ND	NC	ND	14	**26**	51	56	NC	754	886
Soil	8[5]	**18**	15	ND	**539**	128[6]	144	NC	ND	34	NC	5609	5,657
Soil	10[5]	NC	ND	ND	NC	ND	ND	**31**	51	51	**33**	113	92
Soil	12	**5**	4	15	NC	ND	38	**33**	42	45	NC	2681	2,114
Soil	18	NC	ND	ND	NC	ND	ND	**112**	108	67	**48**	125	90
Sediment	29	NC	ND	ND	NC	ND	ND	**53**	48	96	**151**	158	160
Sediment	31	NC	ND	ND	NC	ND	6.2	**37**	32	76	**87**	83	137
Sediment	32	NC	ND	4.6	NC	ND	ND	**57**	67	57	**27**	82	69
Sediment	39	NC	ND	ND	NC	ND	ND	**34**	32	38	**101**	138	137
Sediment	65	**2**	20	22	NC	ND	41	**15**	18	31	NC	1284	1,843
Mean PicoTAX MDL		**9**			**539**			**44**			**73**		

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 PicoTAX in this technology demonstration (in bold typeface for emphasis).

[3] This column lists the mean concentration reported for this MDL sample blend by the PicoTAX.

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

[5] Only six replicates are included for this blend because one outlier replicate with extreme concentrations for all elements was excluded.

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

Conc. Concentration.

MDL Method detection limit.

NA Results for this element were not provided by Rontec.

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 were not used for calculating the MDL for this element.

Ref. Lab. Reference laboratory.

in individual MDL sample blends for a number of other target elements. However, these extreme MDLs appeared to occur on a random and isolated basis, and no generalized trends in MDLs relative to sample medium (soil versus sediment) or blend could be discerned.

The mean MDLs calculated for the PicoTAX 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 PicoTAX were lower than the available mean MDLs calculated from EPA Method 6200 data for all elements except lead. However, when compared with the results for all eight XRF instruments that participated in the demonstration, the PicoTAX exhibited high relative mean MDLs for nine of the 11 target elements for which MDLs could be calculated. The only elements for which the PicoTAX had equivalent or lower MDLs were arsenic and selenium.

7.2 Primary Objective 2 — Accuracy and Comparability

The number of demonstration sample blends that met the criteria for evaluation of accuracy, as described in Section 4.2.2, was low for silver (9 samples) and cadmium (12 samples), but was greater than 20 for the remaining target elements. RPDs between the mean concentrations obtained from the PicoTAX and the reference laboratory were calculated for each blend that met the criteria. Table 7-3 presents the median RPDs for each target

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

Element	PicoTAX Mean MDLs[2]	All XRF Instrument Mean MDLs[3]	EPA Method 6200 Mean Detection Limits[4]
Antimony	NC	61	55 [5]
Arsenic	23	26	92
Cadmium	529	70	NR
Chromium	109	83	376
Copper	29	23	171
Lead	105	40	78
Mercury	84	23	NR
Nickel	78	50	100 [5]
Selenium	9	8	NR
Silver	539	42	NR
Vanadium	44	28	NR
Zinc	73	38	89

Notes:
[1] Detection limits are in units of milligrams per kilogram (mg/kg), or parts per million (ppm).
[2] The mean MDLs calculated for this technology demonstration, as presented in Table 7-1.
[3] The mean MDLs calculated for all eight XRF instruments that participated in this 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.
NC Not calculated; no XRF data reported for this metal.
NR Not reported; no MDLs reported for this element.

element, along with the number of RPD results used to calculate the median. These statistics are provided for all demonstration samples as well as for subpopulations grouped by medium (soil versus sediment) and concentration level (Levels 1 through 4, as documented in Table 3-1). Additional summary statistics for the RPDs (minimum, maximum, and mean) are provided in Appendix E (Table E-1).

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

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.) Further, the classification of the elements based on accuracy generally stayed the same when the mean rather than the median RPD was used for the evaluation, although the means were somewhat higher for many elements (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 nickel and silver. For silver, however, the limited number of sample blends available for evaluation and the high overall variability of the RPDs produce uncertainty in the accuracy evaluation.

- High median RPDs in the soil matrices were observed in the Level 1 samples for cadmium (with concentrations between 50 and 500 ppm). The median RPDs of 157 percent at this concentration level (classified in the "poor" range) was much higher than those for higher concentration levels, where the median RPDs were in the "fair" to "good" ranges. Although the accuracy data set was limited for cadmium, the Level 1 RPDs appeared to be skewed high by the results for sample Blends 7 through 9 from the Wickes Smelter site, which contained high concentrations of other elements (such as lead, zinc, and iron). A smaller effect was seen for selenium, where the Level 1 median RPDs were elevated for both soil and sediment, but remained in the "fair" range. Review of individual RPDs in the selenium data set indicated that this trend appeared to be generalized rather than caused by limited data or extreme results from specific blends.

- The best accuracy for mercury was observed in the Level 1 samples (with concentrations between 20 and 200 ppm) in both the soil and sediment matrices, where median RPDs were in the "good" range (the median RPDs for the other concentration levels were above 25 percent, falling in the "fair" range). These samples were generally characterized by very low concentrations of other elements, including elements adjacent to mercury in the periodic table such as cadmium and lead.

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 PicoTAX were equivalent to or below the all-instrument medians for 11 of the 12 elements compared. For arsenic, mercury, selenium, silver and zinc, the median RPDs for the PicoTAX were less than half the all-instrument medians. Only cadmium displayed a higher median RPD for the PicoTAX than for all eight instruments that participated in the demonstration.

In addition to calculating RPDs, the evaluation of accuracy included preparing linear correlation plots of PicoTAX concentration values against the reference laboratory values. These plots are presented for the individual target elements in Figures E-1 through E-12 of Appendix E. The plots include a 45-degree line showing the "ideal" relationship between the PicoTAX data and the reference laboratory data, as well as a "best fit" linear

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

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	0	--	15	4	28	16	5	16	7	22	4	0	12	20
		Median	NC	--	20.7%	157.0%	20.7%	10.4%	20.5%	24.9%	19.3%	16.7%	41.5%	NC	29.2%	21.0%
	Level 2	Number	0	0	4	6	4	8	13	4	7	5	5	0	4	6
		Median	NC	NC	4.6%	49.6%	10.9%	10.0%	23.2%	16.3%	43.3%	21.6%	6.9%	NC	17.5%	11.3%
	Level 3	Number	0	0	4	2	2	2	13	8	2	6	4	4	4	9
		Median	NC	NC	8.7%	10.0%	14.4%	24.9%	12.0%	20.4%	38.9%	5.6%	11.1%	10.4%	15.2%	10.9%
	Level 4	Number	--	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	--	2.4%	22.9%	--	--	--	--	--	--
	All Soil	Number	0	0	23	12	34	26	38	33	16	33	13	4	21	35
		Median	NC	NC	14.3%	83.2%	17.9%	11.5%	14.9%	21.6%	33.0%	12.2%	6.9%	10.4%	20.7%	16.0%
Sediment	Level 1	Number	0	0	17	0	21	8	3	16	2	18	5	1	6	19
		Median	NC	NC	12.9%	NC	20.3%	5.6%	37.6%	30.0%	27.5%	34.9%	17.4%	8.5%	45.0%	11.6%
	Level 2	Number	0	0	4	0	3	4	19	4	4	6	4	2	8	5
		Median	NC	NC	8.6%	NC	13.4%	17.2%	27.5%	10.9%	40.7%	29.4%	18.9%	86.9%	43.8%	19.4%
	Level 3	Number	0	0	2	0	3	10	4	3	2	4	3	2	3	4
		Median	NC	NC	19.2%	NC	25.0%	18.5%	31.1%	39.7%	31.7%	13.7%	8.8%	49.7%	12.9%	14.0%
	Level 4	Number	--	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	--	8.4%	--	--	--	--	--	--	--
	All Sediment	Number	0	0	23	0	27	22	32	23	8	28	12	5	17	28
`		Median	NC	NC	12.9%	NC	20.3%	15.4%	21.6%	25.5%	37.8%	29.4%	14.1%	59.1%	24.3%	16.2%
All Samples	Rontec	Number	0	0	46	12	61	48	70	56	24	61	25	9	38	63
	PicoTAX	Median	NC	NC	13.4%	83.2%	18.1%	12.3%	17.3%	22.9%	33.0%	21.2%	11.6%	19.3%	22.9%	16.0%
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 ranges.

ERA Environmental Resource Associates, Inc.

NC Not calculated; no XRF data provided for this element.

Number Number of samples appropriate for accuracy evaluation.

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

RPD Relative percent difference.

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 chromium, iron, nickel, and zinc met all three of these criteria. The correlation plot for zinc is displayed in Figure 7-1 as an example of the correlations obtained for these elements.

Figure 7-1. Linear correlation plot for PicoTAX showing high correlation for zinc.

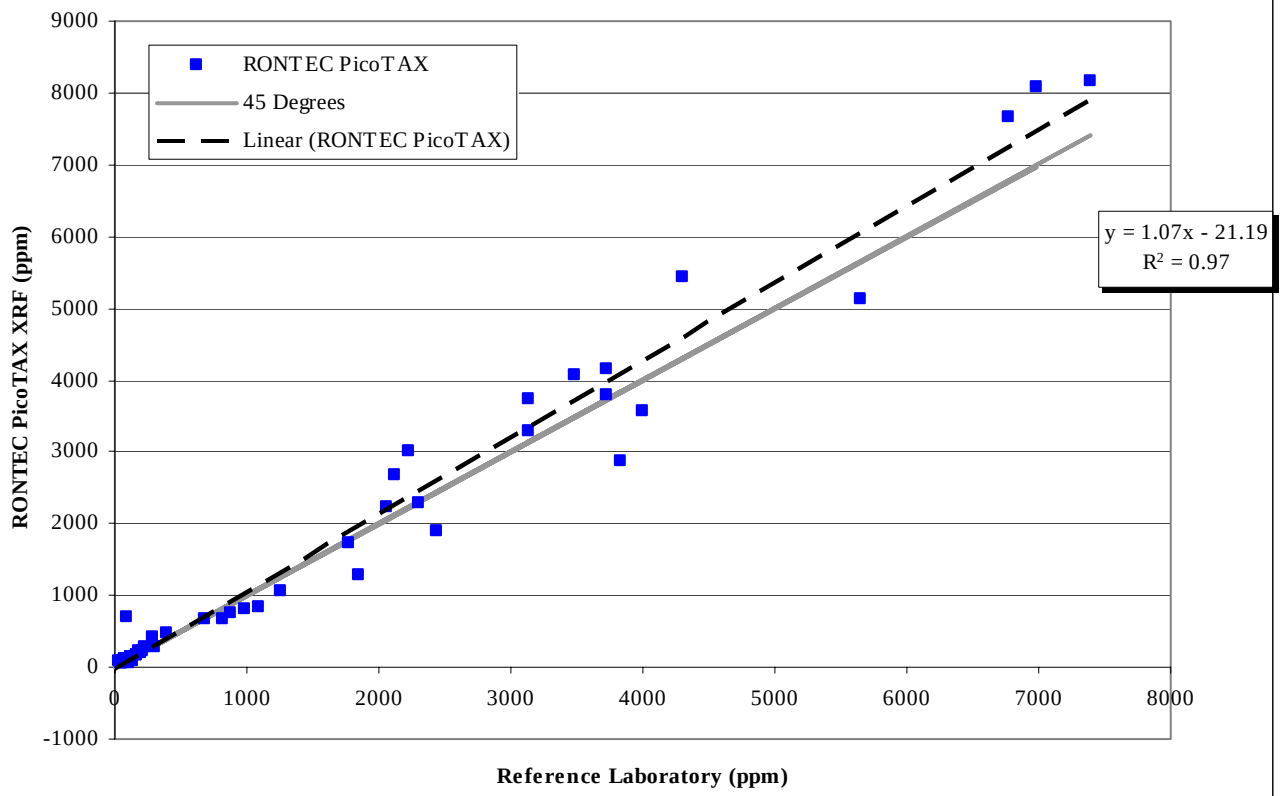


Table 7-4. Summary of Correlation Evaluation for the PicoTAX

Target Element	m	b	r^2	Correlation	Bias
Arsenic	0.90	56	0.95	High	--
Cadmium	0.78	272	0.62	Moderate	High[1]
Chromium	**1.04**	**27**	**0.95**	**High**	**--**
Copper	1.03	-33	0.86	Moderate	--
Iron	**1.02**	**4489 [2]**	**0.95**	**High**	**--**
Lead	1.42	-98	0.94	High	High
Mercury	0.73	-39	0.99	High	Low
Nickel	**0.89**	**0.07**	**0.96**	**High**	**--**
Selenium	1.03	14	0.70	Moderate	--
Silver	0.94	7.0	0.58	Moderate	--
Vanadium	1.04	-11	0.89	Moderate	--
Zinc	**1.07**	**-21**	**0.97**	**High**	**--**

Notes:

[1] A high bias was indicated for cadmium at low concentrations by the high relative y-intercept.

[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:

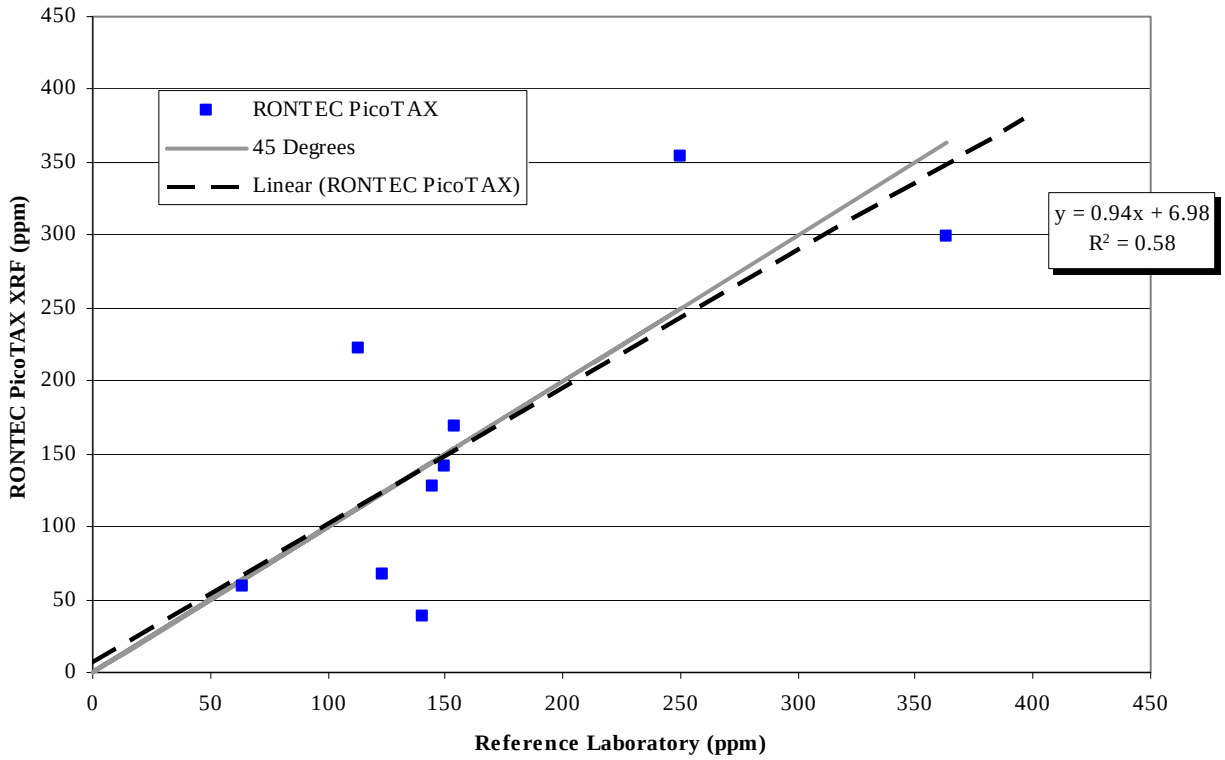
- Consistent with the RPD evaluation and developer predictions, the correlation analysis showed a low degree of accuracy for cadmium and silver. However, these findings may have been affected by the limited availability of data in the various concentration ranges. Removal of the high outliers associated with complex Blends 8 and 9 (Wickes Smelter slag) from the correlation analysis for cadmium improved the r^2 value to 0.90 and lowered the y-intercept to near 0, producing a high correlation and eliminating the apparent positive bias at low concentrations. For silver, however, the low degree of correlation appeared to be due to a broader variability in the data rather than a few outliers. Figure 7-2 shows the correlation plot for silver.

- Removal of a single outlier associated with the Alton Steel site (Blend 53) improved the correlation coefficient for selenium from 0.70 to 0.90.

- Mercury exhibited a high r^2 value (0.99) but a slightly low bias (m = 0.73). Removing two extreme Level 4 concentrations (Blends 21 and 22) from the plots produced a much poorer correlation coefficient (in the range of 0.81) and a lower bias (m = 0.58).

Overall, the evaluations of accuracy showed an accept-able overall level of performance by the PicoTAX for the target elements. Correlations with the reference laboratory were generally high, and median RPDs were better for most elements in comparison to the average of all eight XRF instruments that participated in the demonstration. Factors such as Rontec's rigorous sample preparation protocol and use of internal standards (Chapter 6) may have contributed to the high relative level of accuracy attained. However, the use of a molybdenum x-ray tube precluded the reporting of antimony results and produced low relative accuracy in the cadmium and silver results, consistent with Rontec's predictions.

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



7.3 Primary Objective 3 — Precision

As described in Section 4.2.3, the precision of the PicoTAX 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 50.2 percent (silver). The ranges of median RSDs are further summarized below:

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

The median RSDs for sediment were slightly larger for some target elements (such as arsenic, lead, and vanadium) than the median RSDs for soil.

The level of precision observed may have been facilitated by the level of pre-processing (homogenizing, sieving, crushing, and drying) on the sample blends before the demonstration (Chapter 3). This observation is consistent with the previous SITE MMT program demonstration of XRF technologies that occurred in 1995 (EPA 1996a, 1996b, 1998a, 1998b, 1998c, and 1998d). The high level of sample processing applied during both XRF technology demonstrations was necessary to minimize the effects of sample heterogeneity on the demonstration results

and on comparability with the reference laboratories. During project design, site investigation teams that intend to compare XRF and laboratory data should similarly assess the need for sample processing steps to manage sample heterogeneity and improve data comparability.

Over and above the pre-processing prior to the demonstration, Rontec performed an additional detailed preparation protocol during the demonstration to emulsify the samples, as described in Section 6.2.2. This level of additional sample preparation during the demonstration differentiated the PicoTAX from the other technology developers. (The other developers generally performed minimal additional sample preparation, consisting of only of transferring sample into analysis cups or pressing the samples into pellets.)

Further review of the median RSDs in Table 7-5 based on concentration range reveals slightly higher RSDs (in other words, lower precision) for the target elements in Level 1 samples when compared with the rest of the data set. This effect was observed for multiple target elements in both soil and sediment, with large relative effects for arsenic, chromium, lead, mercury, and nickel in the sediment samples. This observation indicates that, to a minor extent, analytical precision for the PicoTAX 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 PicoTAX were above the all-instrument medians for all 12 of the target elements compared. For cadmium, iron, mercury, and silver, the median RSDs for the PicoTAX were more than three times higher than the all-instrument medians.

Table 7-6 presents median RSD statistics for the reference laboratory and compares these to the summary data for the PicoTAX. (Complete summary statistics are provided in Table E-3 of Appendix E.) Table 7-6 indicates that the median RSDs for the PicoTAX were higher than the RSDs for the reference laboratory for all 12 target elements. For cadmium, mercury, and silver, the median RSDs for the PicoTAX were more than three times higher than the reference laboratory medians. Thus, the PicoTAX exhibited lower precision overall than either the reference laboratory or the other XRF instruments. The reduced precision of the PicoTAX relative to other XRF technologies may be a function of Rontec's sample preparation process, in which a very small subsample (10 µL of a soil emulsion prepared using 0.15 grams of soil) is used for analysis (see Section 6.2.2). Despite the grinding and homogenizing protocols applied prior to the demonstration, the use of such a small aliquot of sample may have diminished the representativeness of the samples, reducing precision.

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).
- 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 confirm significant interference effects of lead on arsenic. Specifically, as lead concentrations increased to greater than 10 times the arsenic concentration, the median RPD for arsenic increased from 12.9 percent (well within the "good" range defined in Section 7.2) to 43.0 percent (at the upper end of the "fair" range). Similar effects are observed for copper as an interferent for nickel; as copper

Table 7-5. Evaluation of Precision — Relative Standard Deviations for the Rontec PicoTAX

Matrix	Sample Group	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	0	15	4	28	16	5	16	7	22	4	0	13	20
		Median	NC	13.8%	27.6%	26.3%	11.9%	19.2%	20.9%	25.2%	19.5%	14.9%	NC	27.1%	15.5%
	Level 2	Number	0	4	6	4	8	13	4	7	5	5	0	4	6
		Median	NC	15.1%	31.5%	9.6%	16.7%	13.3%	12.6%	26.0%	11.6%	18.9%	NC	12.9%	13.8%
	Level 3	Number	0	4	2	2	2	13	8	2	6	4	4	4	9
		Median	NC	12.7%	26.2%	3.7%	15.9%	17.9%	12.0%	33.8%	19.5%	6.0%	51.4%	7.0%	12.0%
	Level 4	Number	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	15.7%	9.4%	--	--	--	--	--	--
	All Soil	Number	0	23	12	34	26	38	33	16	33	13	4	21	35
		Median	NC	14.3%	30.0%	22.1%	13.3%	16.6%	14.4%	25.6%	17.3%	7.0%	51.4%	18.2%	14.7%
Sediment	Level 1	Number	0	17	0	21	8	3	16	2	18	5	1	6	19
		Median	NC	28.3%	NC	32.5%	11.1%	20.3%	24.4%	55.8%	31.6%	5.6%	46.5%	31.0%	21.7%
	Level 2	Number	0	4	0	3	4	19	4	4	6	4	2	8	5
		Median	NC	8.1%	NC	9.9%	17.2%	14.0%	8.6%	29.8%	17.0%	15.8%	50.2%	24.4%	11.2%
	Level 3	Number	0	2	0	3	10	4	3	2	4	3	2	3	4
		Median	NC	10.1%	NC	7.2%	22.5%	16.0%	11.9%	12.9%	10.9%	8.2%	57.4%	9.9%	16.4%
	Level 4	Number	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	17.5%	--	--	--	--	--	--	--
	All Sediment	Number	0	23	0	27	22	32	23	8	28	12	5	17	28
		Median	NC	18.8%	NC	25.1%	16.2%	16.5%	21.3%	26.9%	18.8%	8.6%	50.2%	27.0%	15.5%
All Samples	PicoTAX	Number	0	46	12	61	48	70	56	24	61	25	9	38	63
		Median	NC	15.1%	30.0%	24.2%	15.2%	16.5%	14.5%	26.0%	18.5%	8.2%	50.2%	19.8%	14.7%
All Samples	All XRF Instruments	Number	206	320	209	338	363	558	392	192	403	195	177	218	471
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%	7.0%	4.5%	5.2%	8.5%	5.3%

Notes:

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

Table 7-6. Evaluation of Precision – Relative Standard Deviations for the Reference Laboratory versus the PicoTAX 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	PicoTAX	Number	0	46	13	61	48	70	56	24	61	25	9	38	63
		Median	NC	15.1%	31.0%	24.2%	15.2%	16.5%	14.5%	26.0%	18.5%	8.2%	50.2%	19.8%	14.7%
All Samples	All XRF Instruments	Number	206	320	209	338	363	558	392	192	403	195	177	218	471
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%	7.0%	4.5%	5.2%	8.5%	5.3%

Notes:
Number Number of samples appropriate for precision evaluation
Ref. Lab Reference Laboratory

Table 7-7. Effects of Interferent Elements on the RPDs (Accuracy) for Other Target Elements for the Rontec PicoTAX[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	42	5	14	39	1	8	35	2	11	50	3	10
Median RPD of Target Element [2]	12.9%	5.5%	43.0%	13.8%	42.2%	41.6%	11.1%	16.1%	14.4%	14.1%	6.6%	8.9%	16.8%	11.4%	17.2%
Median Interferent Concentration	89	9262	3434	123	871	1877	98	288	1906	177	4462	3015	169	938	2221
Median Target Element Concentration	135	1071	56	183	100	75	786	92	107	829	851	124	674	127	145

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.

concentrations increased to greater than 10 times the nickel concentration, the median RPD for nickel increased from 13.8 percent to 41.6 percent. Evaluation of the effects of nickel on copper, copper on zinc, and zinc on copper do not appear to show significant interferences.

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 lead appeared to produce an increasingly low bias in the arsenic data (as indicated by more positive raw RPDs). A similar trend was observed for 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 PicoTAX 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-12) and are further depicted for the various elements on box and whisker plots in Figure E-13.

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.) High relative median RPDs for cadmium and nickel were observed in blends from the Wickes Smelter site. The median RPDs in these blends were 165 percent for cadmium and 50 percent for nickel, which were significantly higher than blends from other sampling sites for these two elements. The soil matrix from this site was described during the demonstration sample collection program (Chapter 2) as roaster slag, consisting of a black, fairly coarse sand and gravel material. This slag is an intermediate product in processing ore, wherein volatile sulfide materials are thermally removed, leaving concentrated heavy elements. Effects of the Wickes Smelter sample blends on XRF data quality were noted earlier for cadmium in the accuracy evaluation (Section 7.2).

Review of the box and whiskers plot (Figure E-13) and the correlation plots from the accuracy evaluation revealed few other major trends in RPDs relative to sampling site. The outliers and extreme values apparent in Figure E-13 were broadly distributed between eight of the nine sampling sites. The Torch Lake site represented higher numbers of outliers relative to the other sampling sites. However, the evaluation found that sample matrix had a minor effect on the overall accuracy of the XRF data given that the ranges of RPDs observed for the target elements were very broad. The spread in the accuracy results is illustrated on the box and whiskers plot in Figure E-13. The plot shows that the broad overall distributions of RPDs precluded the identification of statistical outliers and extreme values for cadmium, mercury, and silver. Further data review indicated that the large spread in the RPD data for these metals was affected by high RPD values from the Wickes Smelter blends for cadmium, the Sulfur Bank mine blends for mercury, and a Ramsey Flats blend for silver.

7.6 Primary Objective 6 — Sample Throughput

The Rontec three-person field team was able to analyze all 326 demonstration samples in 4.5 days at the demonstration site and an equivalent of 1 additional day in Berlin, Germany. Once the PicoTax instrument had been set up and operations had been streamlined, the Rontec field team was able to analye 66 samples (that is, three batches of 22 samples) during an extended work day. This sample throughput was achieved by using different members of the field team to perform sample preparation and instrumental analysis and by loading one sample batch into the autosampler to run overnight. Without an extended work day, it was estimated that the

Table 7-8. Effect of Soil Type on the RPDs (Accuracy) for Target Elements, Rontec PicoTAX

Matrix	Site	Matrix Description	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead
Soil	AS	Fine to medium sand (steel processing)	Number	--	1	1	2	3	3	3
			Median	--	181.4%	0.8%	10.8%	34.8%	18.6%	22.9%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	--	7	5	7	6	7	7
			Median	--	5.5%	82.5%	17.9%	10.3%	15.8%	19.8%
Soil	CN	Sandy loam (burn pit residue)	Number	--	1	1	2	3	3	3
			Median	--	17.6%	83.8%	90.1%	20.7%	20.8%	27.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	--	4	2	6	6
			Median	--	--	--	16.3%	8.0%	30.3%	19.9%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	--	11	1	11	4	12	6
			Median	--	9.2%	6.1%	11.7%	6.4%	30.7%	42.8%
Sediment	RF	Silty fine sand (tailings)	Number	--	12	--	12	13	13	13
			Median	--	17.7%	--	22.2%	15.9%	20.5%	23.0%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	--	5	1	11	4	12	7
			Median	--	20.7%	6.3%	39.8%	13.6%	12.6%	19.5%
Sediment	TL	Silt and clay (slag-enriched)	Number	--	2	--	5	7	7	4
			Median	--	42.1%	--	20.3%	19.2%	8.9%	23.5%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	7	3	7	6	7	7
			Median	--	12.2%	165.3%	11.5%	4.2%	7.6%	25.7%
	All		Number	--	46	12	61	48	70	56
			Median	--	13.4%	83.2%	18.1%	12.3%	17.3%	22.9%

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

Matrix	Site	Matrix Description	Statistic	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	AS	Fine to medium sand (steel processing)	Number	--	1	1	1	1	3
			Median	--	74.6%	110.7%	19.3%	68.7%	15.4%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	1	6	4	1	4	7
			Median	110.4%	10.6%	40.6%	5.6%	13.5%	17.9%
Soil	CN	Sandy loam (burn pit residue)	Number	2	3	2	--	1	3
			Median	23.4%	21.6%	6.0%	--	14.5%	17.6%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	3	--	--	--	2
			Median	--	12.2%	--	--	--	7.0%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	4	11	5	1	9	10
			Median	41.9%	10.2%	14.7%	34.5%	15.0%	19.0%
Sediment	RF	Silty fine sand (tailings)	Number	4	13	5	2	3	13
			Median	11.2%	36.7%	11.6%	89.7%	17.2%	11.6%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	11	11	3	--	10	11
			Median	43.3%	5.7%	9.6%	--	33.7%	22.9%
Sediment	TL	Silt and clay (slag-enriched)	Number	2	6	4	2	7	7
			Median	81.7%	30.1%	34.4%	33.8%	52.4%	7.9%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	7	1	2	3	7
			Median	--	50.0%	4.8%	10.4%	8.2%	9.9%
	All		Number	24	61	25	9	38	63
			Median	33.0%	21.2%	11.6%	19.3%	22.9%	16.0%

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).

Rontec field team could have only processed 44 samples (that is, two batches of 22 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 (10 minutes per sample). Rontec selected this instrument run time to provide the maximum analytical precision and accuracy for the demonstration. Rontec indicated that a reduction in analysis time of up to 50 percent could still provide data of sufficient accuracy and precision. If this claim is valid, then a sample throughput of 66 samples (3 batches of 22 samples) per day could have been maintained with a normal 8-hour work day.

A detailed discussion of the time required to complete the various steps of sample analysis using the PicoTAX 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. Rontec offers on-site training, on-line support, and telephone support to instrument users on an informal, as needed basis. Although Rontec provided three Ph.D.-level scientists for the demonstration, plus an additional logistical support person, this level of expertise and staffing is not needed for analysis of soil and sediment samples.

Two operating manuals are provided with the instrument, including an instrument manual and a software manual. The instrument manual provides the user with instructions for installing and operating the instrument, including packaging, transporting, and setup. The manual provides a descriptive summary of connections, control, and display elements, as well as the structural elements of the spectrometer. A section is also provided that discusses instrument maintenance, including adjusting the optical path, gain correction, and change out of the x-ray tube. The observer assessed that proper instrument setup and operation requires a chemist or technician with a basic knowledge of spectroscopy.

The software manual provides a detailed description of the instrument operating system and Windows-based data management software. The software is operated from a notebook computer that is provided with the spectrometer. The manual includes instructions for installing software, starting the program, the user interface, spectral measurement, spectral evaluation, data export, and crystal orientation. This manual is detailed and thorough and is readily understandable for a technician-level analyst with basic computer and software skills along with an understanding of spectroscopy and RS-232 communications technology. On-line help is available to assist the analyst in operating the instrument and using the software.

Processing soil and sediment samples entailed relatively simple procedures that could be performed by a field technician. In addition to the instrument manual, an application note was provided that listed instructions for analysis of soil and sediment samples. This document, although intended as a marketing document on the performance of XRF against traditional laboratory methodology, provided an excellent description of the sample preparation and analysis procedures required by the PicoTAX. The application note is available from the developer.

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 PicoTAX spectrometer is enclosed within a cabinet; the x-ray tube is totally encased within the cabinet and emits no detectable radiation to the analyst or surrounding environment. Acetone (reagent grade)

was used to clean the quartz disks between uses. Acetone is extremely flammable, and the vapor may cause a flash fire. Inhalation of acetone fumes may irritate the respiratory tract. High concentrations of acetone fumes may cause coughing, dizziness, dullness, and headache. Higher concentrations can produce central nervous system depression, narcosis, and unconsciousness. However, the exposure to acetone during the disk cleaning process should be minimal as the quantities of acetone used are very small (a few drops). Further, exposure to acetone can be eliminated by using disposable acrylic disks that are available from Rontec.

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 PicoTAX is defined as transportable. The PicoTAX Spectrometer 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 consists of a spectrometer, autosampler, sampler holders, and notebook PC that runs the operating system of the instrument and provides data analysis and management. There are two handles on each side of the instrument for ease in transporting. The PicoTAX is transported in a wood-lined metal box provided by the developer to protect the instrument from damage during shipment. Peripheral supplies for sample preparation are transported in separate shipments weighing less than 10 pounds.

The PicoTAX spectrometer operates using a standard 110 V AC power source. The notebook computer for the spectrometer also uses 110 V AC. Additionally, a balance was used to weigh sample material, and a hot plate was used to dry a suspension of each sample into a residue on a quartz disk. In total, therefore, four separate devices were required to complete the PicoTAX system, each needing 110 V AC power.

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 PicoTAX system is constructed from impact-resistant coated metal and molded plastic. The instrument is operational up to a maximum temperature of 40°C and 80 percent relative humidity (limited by the air cooling requirements of the detector). Because the spectrometer is intended for indoor use, it requires a stable operating environment and must be protected from weather.

The metal ceramic x-ray tube is warranted for 2,500 hours of operation; typically, Rontec tubes have a minimum lifespan of 10,000 operating hours. The entire instrument is warranted for 1 year for full coverage, and software is upgradeable for up to 2 years at no additional cost to the owner. Rontec provides product support as requested throughout the life of the instrument.

7.12 Secondary Objective 5 — Availability

Rontec is headquartered in Berlin, Germany, but also maintains an office in Carlisle, Massachusetts. The PicoTAX is available from the manufacturer for purchase only; no rental or long-term leasing options are currently available. In addition, no third party distributors for Rontec instrumentation were identified at the time of the demonstration. Rontec operates telephone and on-line support in both the U.S. and Europe.

Chapter 8
Economic Analysis

This chapter provides cost information for the Rontec PicoTAX 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 PicoTAX.

8.1 Equipment Costs

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

The PicoTAX used at the demonstration costs approximately $99,990 for the complete equipment package. The package includes a required 2 day training program for first time users, which separately costs $4,600. The package includes a cassette for 25 sample discs and Messjobebitor PC-controller, which separately costs $15,440.

The standard equipment package includes the metal ceramic x-ray tube. Purchased models include a 1-year warranty on the x-ray tube. The x-ray tube is guaranteed for 2,500 hours. The lifespan of the x-ray tube is at least 5 years in normal usage.

Rontec indicated that the PicoTAX is not available for rental. For comparison to the rental cost of other XRF instruments and for general evaluation purposes, 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 PicoTAX exceed the average costs for all XRF instruments that participated in the demonstration, as shown in Table 8-1.

Table 8-1. Equipment Costs

Cost Element	PicoTAX	XRF Demonstration Average [1]
Shipping	$750	$410
Capital Cost (Purchase)[2]	$99,990	$54,300
Weekly Rental	$5,200[3]	$2,813
Autosampler (for Overnight Analysis)	Included	N/A

Notes:
[1] Average for all eight instruments in the demonstration
[2] Capital cost includes cost for required instrument training
[3] Estimated rental cost.
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 purchase of disposable acrylic discs rather than the quartz discs if the user wishes to eliminate disc cleaning efforts.

The PicoTAX was operated for five days at the demonstration site, and two days in Berlin, to complete the analysis of all 326 samples. 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 PicoTAX 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 PicoTAX is compared with the average of all XRF instruments in Table 8-2 and is compared with the range of all XRF instruments in Figure 8-1. In comparison to other XRF analyzers, the PicoTAX exhibited higher-than-average times except for daily shutdown and startup and end of project packing. Further, Rontec used a three-person team to operate the instrument during the field demonstration, whereas the field teams used by other developers included only one or two people.

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

Activity	PicoTAX	Average[2]
Initial Setup and Calibration	90	54
Sample Preparation	5.9	3.1
Sample Analysis	12.5	6.7
Daily Shutdown/Startup	0	10
End of Project Packing	20	43
Total Processing Time per Sample	18.7	10.0

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

The Rontec field team expended about 138 man-hours to complete all sample processing activities during the field demonstration using the PicoTAX. This was significantly higher than the overall average of 69 hours for all instruments that participated in the demonstration. The primary reasons that labor hours were higher for the PicoTAX include:

- The unique sample preparation protocol employed by Rontec, as described in Section 6.3, required substantial more time than the sample preparation procedures employed by other instruments.

- The instrument run time of 10 minutes was longer than most other instruments.

As noted by Rontec, however, the instrument run time could be reduced to 5 minutes without significantly affecting precision and accuracy. This would directly reduce the time required for sample analysis and significantly reduce the labor hours. Use of the autosampler saved significantly on the time required for sample analysis and the associated labor hours during the field demonstration.

8.4 Comparison of XRF Analysis and Reference Laboratory Costs

Two scenarios were evaluated to compare the cost for XRF analysis using the PicoTAX 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.

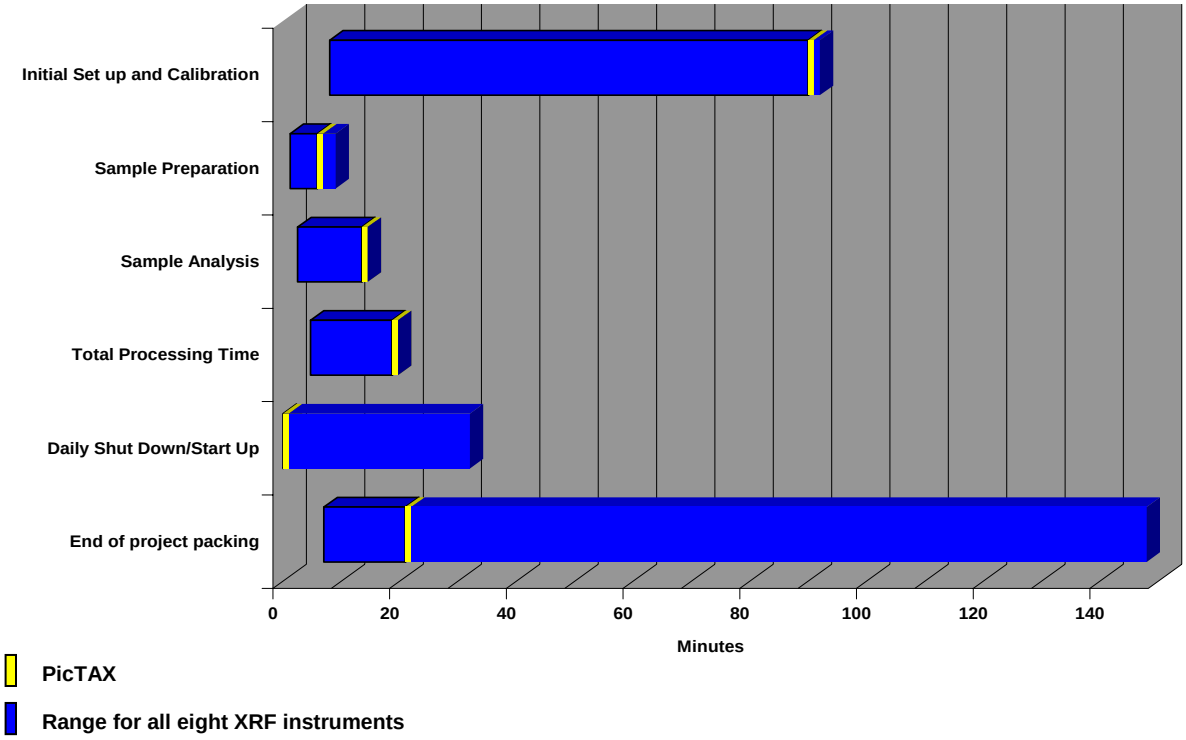


Figure 8-1. Comparison of activity times for the PicoTAX versus other XRF instruments.

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. However, Rontec did not report data for antimony during the field demonstration; thus, the second scenario includes only 12 elements for the PicoTAX.

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 PicoTAX was based on equipment rental for 1 week, along with labor and supplies estimates established during the field demonstration. Sample preparation and sample analysis labor were estimated based on the observed division of responsibilities during the field demonstration, wherein Rontec utilized two people to prepare samples and one person to monitor the spectrometer and manage data on the laptop computer,. Additional sample preparation labor was 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. 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 PicoTAX analysis cost was not adjusted for one element versus 12 elements.

Table 8-3 summarizes the costs for the PicoTAX versus the cost for analysis in a fixed laboratory. This comparison shows that the PicoTAX compares favorably to a fixed laboratory in terms of overall cost when a large number of elements are to be determined. The PicoTAX compares unfavorably to a fixed laboratory when one element are to be determined. Use of the PicoTAX will likely produce additional cost savings, however, 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 PicoTAX in the example scenario (326 samples) was estimated at $14,678, whether one or a number of elements was analyzed. This estimate compares with the average of $8,932 for all XRF instruments that participated in the demonstration. However, it should be noted that bench-top instruments, such as the PicoTax, are known to cost more than hand-held instruments that were included in the calculation of the average cost for all XRF instruments. In comparison to other bench-top XRF instruments, the PicoTAX cost for the example scenario only slightly exceeded other instruments.

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

Analytical Approach	Quantity	Item	Unit Rate	Total
PicoTAX (1 to 12 elements)				
Shipping	1	Roundtrip	$750	$750
Weekly Rental[1]	1	Week	$5,200[1]	$5,200
Supplies	326	Sample	$0.75	$245
Labor	192	Hours	$43.75	$8,393
IDW	N/A	Each	N/A	$90
Total PicoTAX Analysis Cost (1 to 12 elements)				**$14,678**
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 (12 elements)				**$63,896**

Notes:
[1] Estimated value as Rontec currently does not have a rental rate for the PicoTAX.

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

The findings of the evaluation of the PicoTAX for each primary and secondary objective of the technology demonstration are summarized in Tables 9-1 and 9-2. The PicoTAX 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 PicoTAX showed:

- Equivalent or better MDLs for only two elements including arsenic and selenium (iron was not included in the MDL evaluation).
- Equivalent or better accuracy (lower RPDs) for 11 target elements (cadmium was the lone exception).
- Equivalent or better precision (lower RSDs) for no target elements.

As a transportable bench-top instrument that requires AC power, the PicoTAX 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 metal-ceramic x-ray tube used in this instrument produced poor results for cadmium and silver and precluded the reporting of any results for antimony (reducing the number of target elements from 13 to 12 for the PicoTAX). Moreover, a rigorous sample preparation protocol was applied by the developer during the demonstration to convert small aliquots of sample into emulsified residues on quartz disks for analysis. This on-site preparation protocol required additional equipment and space, reduced sample throughput, and may have also reduced analytical precision.

Table 9-1. Summary of Rontec PicoTAX Performance – Primary Objectives

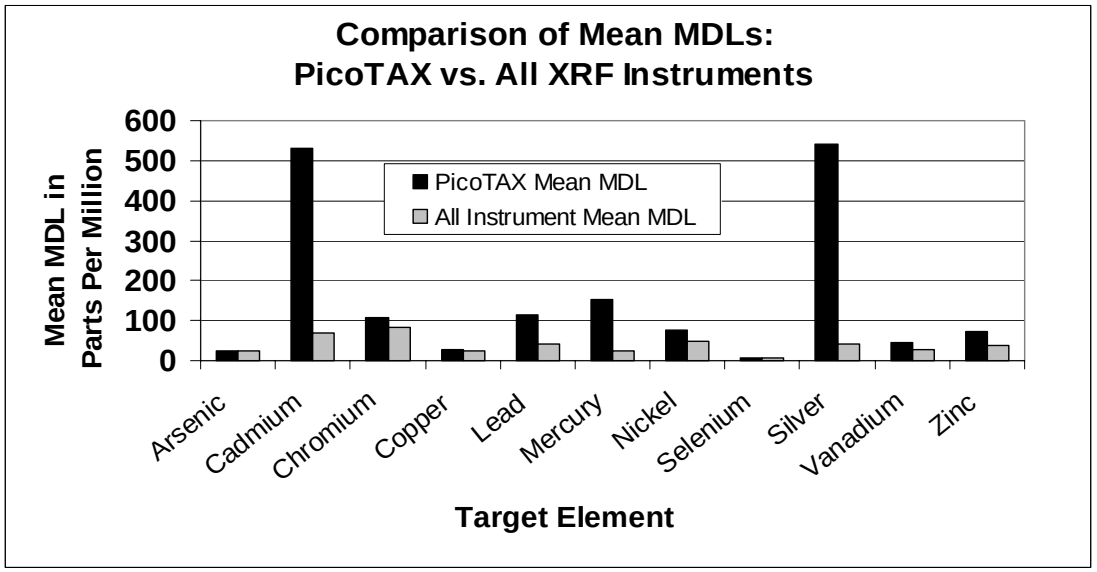
Objective	Performance Summary
P1: Method Detection Limits	• Low numbers of detections in the MDL blends produced limited data and therefore, uncertainty in the MDL calculations for cadmium, mercury, selenium, and silver. • Mean MDLs for the target elements ranged as follows: o MDLs of 1 to 20 ppm: selenium. o MDLs of 20 to 50 ppm: arsenic, copper, and vanadium. o MDLs of 50 to 100 ppm: mercury, nickel, and zinc. o MDLs of greater than 100 ppm: cadmium, chromium, lead, and silver. (MDLs were greater than 500 ppm for cadmium and silver. Iron was not included in the MDL evaluation.) • The MDLs calculated for the PicoTAX 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: arsenic, chromium, copper, iron, lead, nickel, selenium, silver, vanadium, and zinc. o RPDs of 25 to 50 percent: mercury. o RPDs of greater than 50 percent: cadmium. • Correlation plots relative to reference laboratory data indicated: o High correlation coefficients (greater than 0.9) for seven of the 12 target elements evaluated. However, the high correlation observed for one of these elements, mercury, was artificially improved by a few extreme concentrations. o Moderate correlation coefficients for cadmium, copper, selenium, silver, and vanadium. o High biases in the XRF data versus the lab data for cadmium and lead. A low bias was observed for mercury. • Significant uncertainty was introduced into the accuracy assessment for cadmium and silver because the low sensitivity of the instrument limited the sample blends available for evaluation.
P3: Precision	• Median RSDs for sample replicates were as follows, with lower RSDs indicating greater precision: o RSDs below 5 percent: none. o RSDs between 5 and 10 percent: selenium, o RSDs between 10 and 20 percent: arsenic, copper, iron, lead, nickel, vanadium, and zinc. o RSDs greater than 20 percent: cadmium, chromium, mercury, and silver. • 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 all 12 of the target elements evaluated, median RSDs for the PicoTAX were higher than the RSDs for the reference laboratory data, indicating better precision for the reference laboratory.

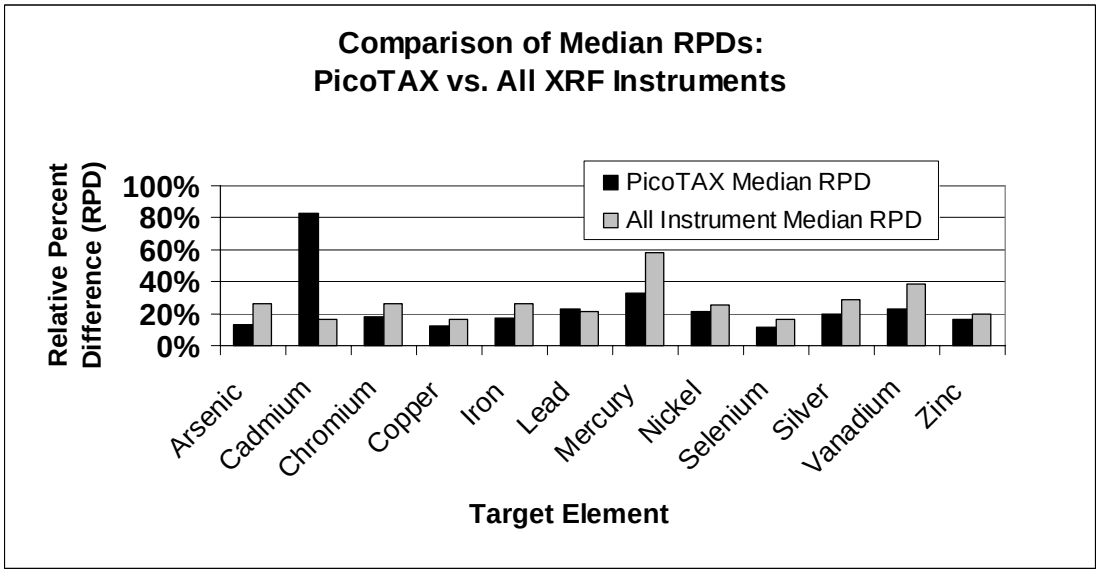
Table 9-1. Summary of Rontec PicoTAX Performance – Primary Objectives (continued)

Objective	Performance Summary
P4: Effects of Sample Interferences	• High relative concentrations (greater than 10X) of lead as an interfering element reduced accuracy for arsenic from "good" (median RPDs between 10 percent and 25 percent) to "fair" (median RPDs between 25 and 50 percent). Further, the high concentrations of lead produced an increasingly low bias in arsenic results. • Similar effects (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 nickel on copper, copper on zinc, and zinc on copper did not appear to show significant interference effects.
P5: Effects of Soil Type	• Low relative accuracy was observed for cadmium and nickel in blends of roaster slag from the Wickes Smelter site, which contained high overall element concentrations. • Slightly higher numbers of extreme RPDs were observed in blends from the Torch Lake site (copper, selenium, vanadium, zinc), the Sulfur Bank mine (mercury), and the Ramsey Flats site (silver). However, the evaluation found that sample matrix had a minor overall effect on accuracy for the PicoTAX.
P6: Sample Throughput	• Rontec's rigorous sample preparation of protocol that included grinding the soil, creating a suspension, spiking internal standard, applying droplets of the suspension to a quartz disc, and then drying the disc to produce a thin residue for analysis took an average of 5.9 minutes per sample. • With an average instrument analysis time of 12.5 minutes per sample, the total sample processing time was 18.7 minutes per sample. • A maximum sample throughput of 66 samples per day (three batches of 22 samples) was achieved during the demonstration by loading one sample batch into the autosampler to run overnight after sample preparation during the day. A more typical sample throughput was estimated to be 44 samples per day (two batches of 22 samples) for an 8-hour work day.
P7: Costs	• Purchase cost is about $99,990 for the instrument as equipped in the demonstration (with autosampler, sample preparation equipment, and laptop PC). The purchase cost includes training. • The Rontec field team expended approximately 138 man-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 shipping/supplies costs, a total project cost of $14,678 was estimated for a project the size of the demonstration. In comparison, the average project cost for all participating XRF instruments was $8,932 and for fixed-laboratory analysis of all 13 elements was $63,896.

Table 9-2. Summary of Rontec PicoTAX Performance – Secondary Objectives

Objective	Performance Summary
S1: Training Requirements	• Field or laboratory technicians that have some familiarity with analytical chemistry and spectroscopy are qualified to operate the PicoTAX. • Rontec offers unlimited product support throughout the lifetime of the instrument, including on-line support and training as needed. Instrument purchase costs include a required 2-day training program ($4,600 separately). • Detailed instrument and software manuals, as well as application notes, assist operators with soil analysis.
S2: Health and Safety	• The PicoTAX's x-ray tube is totally encased and emits no detectable radiation outside of the instrument cabinet. • Acetone is used in the sample preparation process. This solvent is flammable and toxic, but exposure can be eliminated by using disposable acrylic disks.
S3: Portability	• Based on dimensions, weight, and power requirements, the PicoTAX is a transportable instrument and is designed to be used on a table top or possibly a truck bed. Required accessories for efficient sample processing include the autosampler, sample holders, a laptop, and sample preparation equipment. • The instrument and its laptop computer, along with an analytical balance and hotplate, require 110 volt AC power.
S4: Durability	• The PicoTAX's x-ray tube is warranted for 2,500 hours, with an anticipated lifetime of 10,000 hours. • The instrument is fully warranted for 1 year, and software is upgradeable for 2 years at no cost. • The instrument is operational up to 40°C and 80 percent humidity. It requires a stable operating environment and protection from weather.
S5: Availability	• In November, 2005, Rontec was acquired by Bruker AXS Inc. with several world-wide offices, including Berlin, Germany and Madison, Wisconsin. • The PicoTAX is available for purchase only; no rental or long-term leasing options are currently available.





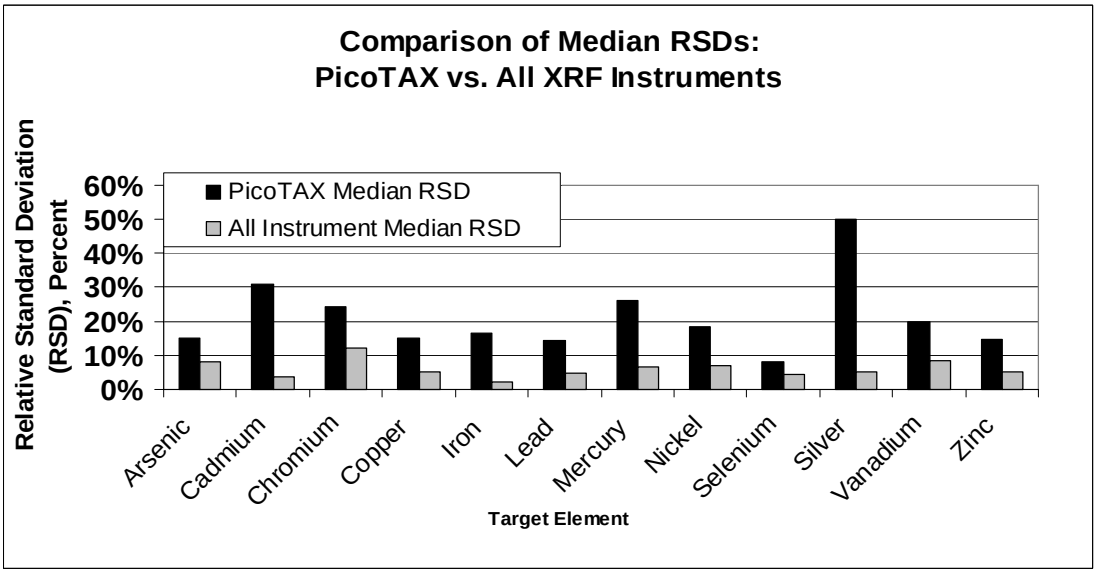


Figure 9-1. Method detection limits (sensitivity), accuracy, and precision of the PicoTAX in comparison to the average of all eight XRF instruments.

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Chapter 10
References

Gilbert, R.O. 1987. *Statistical Methods for Environmental Pollution Monitoring*. Van Nostrand Reinhold, New York.

Tetra Tech EM Inc. 2005. *Demonstration and Quality Assurance Plan*. Prepared for U.S. Environmental Protection Agency, Superfund Innovative Technology Evaluation Program. March.

U.S. Environmental Protection Agency (EPA). 1996a. *TN Spectrace TN 9000 and TN Pb Field Portable X-ray Fluorescence Analyzers*. EPA/600/R-97/145. March.

EPA. 1996b. *Field Portable X-ray Fluorescence Analyzer HNU Systems SEFA-P*. EPA/600/R-97/144. March.

EPA. 1996c. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)*. December.

EPA. 1998a. *Environmental Technology Verification Report; Field Portable X-ray Fluorescence Analyzer, Metorex X-Met 920-MP*. EPA/600/R-97/151. March.

EPA. 1998b. *Environmental Technology Verification Report; Field Portable X-ray Fluorescence Analyzer, Niton XL Spectrum Analyzer*. EPA/600/R-97/150. March.

EPA. 1998c. *Scitect MAP Spectrum Analyzer Field Portable X-Ray Fluorescence Analyzers*. EPA/600/R-97/147. March.

EPA. 1998d. *Metorex X-MET 920-P and 940 Field Portable X-ray Fluorescence Analyzers*. EPA/600/R-97/146. March.

EPA. 1998e. *EPA Method 6200, from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Update IVA*. December.

EPA. 2000. *Guidance for Data Quality Assessment: Practical Methods for Data Analysis*. EPA QA/G-9 QA00 Update. EPA/600/R-96/084. July.

EPA. 2004a. *Innovative Technology Verification Report: Field Measurement Technology for Mercury in Soil and Sediment - Metorex's X-MET® 2000 X-Ray Fluorescence Technology*. EPA/600/R-03/149. May.

EPA. 2004b. *Innovative Technology Verification Report: Field Measurement Technology for Mercury in Soil and Sediment - Niton's XLi/XLt 700 Series X-Ray Fluorescence Analyzers*. EPA/600/R-03/148. May.

EPA. 2004c. *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. *Final*. OSWER 9240.1-45. EPA 540-R-04-004. October.

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:	PicoTAX XRF Analyzer
COMPANY:	Rontec
ADDRESS:	90 Martin Street Carlisle, MA 01741
Telephone:	(800) 875-1578
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Email:	psmith@RONTECusa.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 Rontec PicoTAX hand-held x-ray fluorescence (XRF) analyzer for the analysis of 12 target elements in soil and sediment, including arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, vanadium, and zinc. (One other target element for the demonstration, antimony, could not be analyzed by the PicoTAX.)

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 PicoTAX, 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 Rontec PicoTAX XRF analyzer. More detailed discussion can be found in the *Innovative Technology Verification Report – XRF Technologies for Measuring Trace Elements in Soil and Sediment: Rontec PicoTAX XRF Analyzer* (EPA/540/R-06/005).

TECHNOLOGY DESCRIPTION

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

The PicoTAX is a portable bench-top device that provides quantitative and semi-quantitative multi-element microanalysis of soils and sediments using total reflection XRF spectroscopy. The spectrometer includes a 40-watt metal-ceramic tube excitation source and a thermoelectrically cooled, silicon drift detector. The XRF analyzer is capable of detecting up to 75 elements from aluminum (atomic number [Z] = 13) to yttrium (Z = 39) and from palladium (Z = 46) to uranium (Z = 92). According to Rontec, the molybdenum tube source used for the demonstration displays poor performance for antimony, cadmium, and silver. (Although Rontec proceeded to report data for cadmium and silver, the demonstration confirmed poor overall performance for these metals.)

The PicoTAX uses an internal standard for instrument calibration, thus an initial calibration is not required. A solution of an internal standard element (gallium was selected for the demonstration) is added to each sample to establish response factors (determined by the software). Element quantitation is determined by comparing the response of the unknown elements to the response of the internal standard that has a known concentration. The PicoTAX analysis method requires a rigorous sample preparation protocol that involves grinding a small soil aliquot (150 mg), emulsifying it, spiking the internal standard, applying drops of the emulsion to quartz disks, and drying the disks to create a uniform film. The dried disks are loaded on the instrument's autosampler in batches of 25 samples.

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 further calculated for each element. The ranges into which the mean MDLs fell for the PicoTAX are listed below.

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

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

Accuracy: Accuracy was evaluated based on the agreement of the PicoTAX 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 PicoTAX was classified from high to very low for the various target elements, as indicated in the table below, based on the overall median RPDs for the demonstration.

Relative Accuracy	Median RPD	Target Elements
High	0% - 10%	None.
Moderate	10% - 25%	Arsenic, Chromium, Copper, Iron, Lead, Nickel, Selenium, Silver, Vanadium, and Zinc.
Low	25% - 50%	Mercury.
Very Low	> 50%	Cadmium.

Accuracy was also assessed through correlation plots between the mean PicoTAX 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.

	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Correlation	0.95	0.62	0.95	0.86	0.95	0.94	0.99	0.96	0.70	0.58	0.89	0.97
Bias	--	High	--	--	--	High	Low	--	--	--	--	--

Notes: -- = No significant bias

Precision: Replicates were analyzed for all sample blends. Precision was evaluated by calculating the standard deviation of the replicates, dividing by the average concentration of the replicates, and multiplying by 100 percent to yield the relative standard deviation (RSD) for each blend. Precision of the PicoTAX was classified from high to very low for each target element, as indicated in the table below, based on the overall median RSDs. These results indicated a lower level of precision in the PicoTAX data than in the reference laboratory data for all 12 of the target elements.

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

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 arsenic was reduced from “moderate” (median RPDs of 10 percent to 25 percent) to “low” (median RPDs between 25 and 50 percent) by high relative concentrations of lead (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 arsenic 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. This evaluation found high outlier RPD values, indicating low relative accuracy, for cadmium and nickel in blends of roaster slag from the Wickes Smelter site. These blends contained high overall element concentrations. Extreme RPDs were also observed in other blends of mining wastes from the Sulfur Bank Mercury Mine (mercury), the Ramsey Flats site (silver), and the Torch Lake site (multiple elements). However, the evaluation found that sample matrix had a minor overall effect on accuracy for the PicoTAX.

Sample Throughput: The total processing time per sample was estimated at 18.7 minutes, which included 5.9 minutes of sample preparation and 12.5 minutes of instrument analysis time. On this basis, a sample throughput of 44 samples per 8-hour work day was estimated with the use of the instrument's autosampler. As noted above, however, the sample blends had undergone rigorous pre-processing before the demonstration. Sample throughput would have decreased if these sample preparation steps (grinding, drying, sieving) had been performed during the demonstration; these steps can add from 10 minutes to 2 hours to the sample processing time.

Costs: A cost assessment identified a purchase cost of $99,990 for the PicoTAX as equipped for the demonstration. Using a hypothetical rental cost approximated from similar types of instruments, a total cost of $14,678 (with a labor cost of $8,393 at $43.75/hr) was estimated for a project similar to the demonstration (326 samples of soil and sediment). In comparison, the project cost averaged $8,932 for all eight XRF instruments participating in the demonstration and $63,896 for fixed-laboratory analysis of all 13 target elements.

Skills and Training Required: Field or laboratory technicians that have some familiarity with analytical chemistry and spectroscopy are qualified to operate the PicoTAX. Rontec offers product support as required throughout the lifetime of the instrument, including on-line support and training. A mandatory 2-day introductory training course is included in the instrument purchase cost. Detailed instrument and software manuals, as well as application notes, assist operators with soil analysis.

Health and Safety Aspects: The PicoTAX's x-ray tube is totally encased and emits no detectable radiation outside of the instrument cabinet. Acetone is used to clean the quartz disks in the sample preparation process, but use of acetone can be eliminated by using disposable acrylic disks.

Portability: Based on dimensions (42 X 59 X 30 centimeters) and weight (28 kilograms), the PicoTAX is a transportable instrument, designed to be used on a table top or possibly a truck bed. Required accessories for efficient sample processing include the autosampler, sample holders, a laptop, and sample preparation equipment. The instrument and its laptop computer, along with an analytical balance and hotplate for sample preparation, require 110 volt AC power.

Durability: The PicoTAX's x-ray tube is warranted for 2,500 hours, with an anticipated lifetime of 10,000 hours. The instrument is fully warranted for 1 year, and software is upgradeable for 2 years at no cost. The instrument is operational up to 40°C and 80 percent humidity. It requires a stable operating environment and protection from weather.

Availability: Rontec maintains offices in Berlin, Germany, and Carlisle, Massachusetts. There are currently no third-party distributors in the U.S. The PicoTAX is available for purchase only; no rental or long-term leasing options are currently available.

RELATIVE PERFORMANCE

The performance of the PicoTAX relative to the average of all eight XRF instruments that participated in the demonstration is shown below:

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

1. Recent Technological Improvements

1.1 Introduction

The results of the EPA SITE demonstration measurements have shown several benefits of the PicoTAX TXRF spectrometer but also some analytical restrictions which desire technical improvements. The following sections discuss the objectives of the performance evaluation and how the performance could be improved.

1.2 Method Detection Limits

In addition to counting statistics and instrument sensitivity, the major limiting factor for MDLs is the reproducibility of measurement results when analysing elements close to the expected MDL.
In TXRF analysis the small analysed sample amount restricts the reproducibility because small inhomogeneities of element distribution will have a large influence. In addition, the detector of the PicoTAX TXRF spectrometer is equipped with an active area of 10 mm^2, while the average sample area is about 30 – 40 mm^2. Thus, the complete sample is not taken into account for data acquisition.

Two recent developments have improved the performance of the PicoTAX TXRF spectrometer and increased the quality of MDLs. First, the line focus X-ray tube was replaced by a micro focus tube. The improvement of excitation intensity can be estimated to be approximately 60 %. In addition, a 30 mm^2 detector was introduced recently. This led to an average increase of signal intensity of 200 %. As a second positive effect, the new detector influences the overall counting statistics by increasing the actually analyzed sample area by a factor of three.

To evaluate the benefits of these technical improvements for the MDLs, the reproducibility measurements of 12 soil and sediment samples were repeated. The sample preparation and measurement conditions were exactly the same as during the first measurement campaign. A summary of the results is given in Table 1; the complete data set can be found at the end of this chapter (Table 4). As the quality of measurement results for the elements antimony, cadmium and silver is poor in general, the MDLs for these elements are displayed in parentheses and are approximate.

Table 1. Comparison of Old PicoTAX-, new PicoTAX- and All XRF Instrument Mean-MDLs. All Values are Given in mg/kg.

Element	PicoTAX Mean MDLs Old Values	PicoTAX Mean MDLs New Values	All XRF Instrument Mean MDLs
Antimony	NC	(167)	61
Arsenic	23	15	26
Cadmium	529	(329)	70
Chromium	109	30	83
Copper	29	9	23
Lead	105	37	40
Mercury	84	16	23
Nickel	78	28	50
Selenium	9	7	8
Silver	539	(58)	42
Vanadium	44	37	28
Zinc	73	39	38

It is obvious that the application of the recent technology enhancements lead to a distinct improvement of the MDLs for some elements up to a factor of 3 to 4.

1.3 Accuracy and comparability

The larger detector area will certainly improve the accuracy of the PicoTAX. For a detailed evaluation of the improvements, the analysis of the complete set of 326 samples would be necessary. Therefore, only a qualitative assessment of the improvements can be provided at this point. Since for a detailed evaluation of accuracy and comparability the complete set of 326 samples would have to be analyzed, only an assessment of possible improvements by the recent technology enhancements is possible. Due to the larger detected sample area, samples with inhomogeneous element distribution will deliver more accurate results. The enhanced MDL will allow the analysis of elements in concentrations which were not detectable with the original equipment.

The quality of measurement results for the elements antimony, cadmium and silver can not be improved significantly by the introduction of a micro focus tube and a larger detector. As mentioned in the previous sections, the detection of these elements is limited to the L-lines when applying a Mo tube. In soil and sediment samples, these lines are completely overlapped by the K-lines of the matrix elements calcium and potassium. A technical solution can be an alternative excitation source. Recently, initial measurements with a W-anode tube have been performed successfully. Figure 1 shows a TXRF spectra of a soil sample, containing 64 mg/kg of Cd. A commercially available system with W-excitation is planned to be released mid-2006.

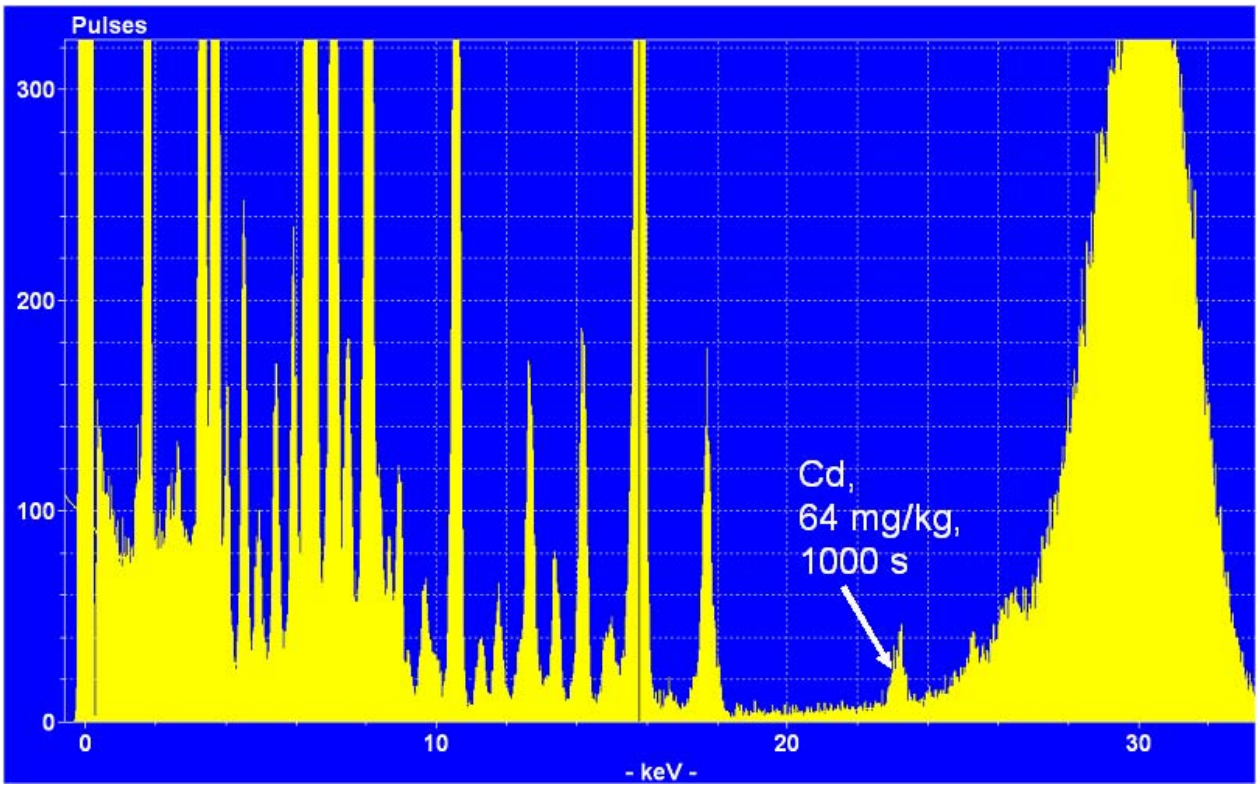

Figure 1. TXRF spectra of a soil sample analysed with W-excitation.

1.4 Precision

The precision defined by the Relative Standard Deviation (RSD) can be assessed on the RSD values of the repetitive MDL measurements. A comparison of the ranges of median RSDs according to the classification described in chapter 7.3 is summarized in Table 2. The corresponding data set is given in Table 5 at the end of this chapter.

Table 2. Comparison of Old and New RSD Values for the PicoTAX TXRF Spectrometer

	Old values	**New values**
Arsenic	Moderate	Moderate
Cadmium	High	High
Chromium	High	High
Copper	Moderate	Low
Iron	Moderate	Low
Lead	Moderate	Moderate
Mercury	High	High
Nickel	Moderate	Moderate
Selenium	Low	Low
Silver	High	High
Vanadium	Moderate	Moderate
Zinc	Moderate	Moderate

Although numerical enhancements are visible, a step into better classification ranges could just be achieved for the elements copper and iron.

2. Analysis of Digested Soils and Sediments

In contrast to common XRF systems, the application of TXRF spectroscopy is capable for trace element analysis in liquids. For an assessment of this laboratory based analysis, two samples from the EPA SITE program were analysed after microwave digestion. Microwave digestion was performed according to the EPA Method 3051; 10 µl of Ga solution (Merck, 1 g/L) were added to 1 ml of the digested solution for internal standardisation. After the resulting solution was thoroughly homogenized, an aliquot of 10 µL was transferred onto a quartz glass sample carrier and dried on a heating plate. TXRF analysis was performed with the same instrument as described in chapter 6.0 by applying measurement times of 600 seconds. The results of the measurements are summarized in Table 3.

Table 3. Comparison of Reference Laboratory Values and PicoTAX Results of Microwave Digested Samples. All values in mg/kg.

Sample	CN-SO-03		KP-SO-02	
Element	EPA values	PicoTAX values	EPA values	PicoTAX values
Arsenic	120	106	1.2	1.0
Cadmium	88	Not detected	Not detected	Not detected
Chromium	18	17	350	329
Copper	100	76	31	26
Iron	20,000	20,611	1,400	1,592
Lead	180	179	580	588
Mercury	42	Not detected	0.91	Not detected
Nickel	110	74	150	167
Selenium	52	41	Not detected	Not detected
Silver	130	Not detected	0.059	Not detected
Vanadium	36	27	1.7	Not detected
Zinc	78	69	15	10
Barium	Not analysed	95	Not analysed	Not detected
Bromine	Not analysed	1.0	Not analysed	3.0
Calcium	Not analysed	2 854	Not analysed	217
Manganese	Not analysed	227	Not analysed	33
Potassium	Not analysed	1 961	Not analysed	23
Rubidium	Not analysed	39	Not analysed	3
Strontium	Not analysed	65	Not analysed	Not detected
Thorium	Not analysed	65	Not analysed	Not detected
Titanium	Not analysed	391	Not analysed	20
Yttrium	Not analysed	22	Not analysed	Not detected

Obviously, all values obtained after analysis of microwave digested samples show accuracies which can be classified either as “very good” or “good”. As digestion of the samples has no influence on the matrix composition, antimony, cadmium and silver could still not be analysed. The analysis of mercury after microwave digestion is not possible due to the volatility of this element.

No MDL or RPD evaluation was performed for digested samples. But because one of the major limiting influences on these factors can be found in the sample inhomogeneity, values with increased quality can be expected.

Table 4. Evaluation of Sensitivity – Method Detection Limits for the Röntec PicoTAX (30 mm^2 detector)

Matrix	Sample No.	Antimony			Arsenic			Cadmium			Chromium		
		Rontec MDL	**Rontec Conc**	**Ref. Lab Conc**	**Rontec MDL**	**Rontec Conc**	**Ref. Lab Conc**	**Rontec MDL**	**Rontec Conc**	**Ref. Lab Conc**	**Rontec MDL**	**Rontec Conc**	**Ref. Lab Conc**
Soil	AS-SO-01	NC	NA	ND	**4**	3	24	NC	NA	62	NC	196	220
Soil	BN-SO-01	NC	NA	140	NC	1632	1900	**607**	1399	1000	**29**	74	91
Soil	KP-SO-01	**27**	207	270	NC	NA	8	NC	NA	0,1	**5**	3	5
Soil	KP-SO-02	NC	NA	6	**6**	4	1	NC	NA	ND	**85**	306	350
Soil	SB-SO-02	NC	NA	ND	**9**	22	10	NC	NA	ND	NC	205	170
Soil	SB-SO-03	NC	NA	17	**24**	62	30	NC	NA	ND	**11**	45	18
Soil	WS-SO-02	NC	NA	110	NC	10808	6300	NC	NA	170	**33**	27	70
Soil	CN-SO-01	**205**	127	16	**11**	145	100	**51**	47	72	**7**	3	15
Sediment	TL-SE-02	NC	NA	2	**5**	18	10	NC	NA	ND	**25**	40	74
Sediment	RF-SE-02	NC	NA	2	**57**	277	220	NC	NA	11	**12**	12	38
Sediment	LV-SE-01	NC	NA	ND	NC	767	800	NC	NA	ND	**50**	71	56
Sediment	LV-SE-02	NC	NA	ND	**3**	59	31	NC	NA	ND	**40**	37	67
Mean Rontec MDL		**167**			**15**			**329**			**30**		

Table 4. Evaluation of Sensitivity – Method Detection Limits for the Röntec PicoTAX (30 mm^2 detector) – continued.

		Copper			Lead			Mercury			Nickel		
		Rontec	**Rontec**	**Ref. Lab**	**Rontec**	**Rontec**	**Ref. Lab**	**Rontec**	**Rontec**	**Ref. Lab**	**Rontec**	**Rontec**	**Ref. Lab**
Matrix	**Sample No.**	**MDL**	**Conc**	**Conc**	**MDL**	**Conc**	**Conc**	**MDL**	**Conc**	**Conc**	**MDL**	**Conc**	**Conc**
Soil	AS-SO-01	**12**	87	180	NC	1404	1900	NC	NA	3	84	48	100
Soil	BN-SO-01	NC	2384	3000	NC	12797	12000	NC	NA	6	46	99	180
Soil	KP-SO-01	NC	516	780	NC	18253	22000	NC	NA	7	NC	NA	3
Soil	KP-SO-02	**7**	20	31	**32**	615	580	NC	NA	1	40	141	150
Soil	SB-SO-02	**4**	41	52	**24**	5	22	**45**	25	66	39	189	230
Soil	SB-SO-03	**4**	8	7	**42**	12	35	NC	4843	1900	3	15	23
Soil	WS-SO-02	NC	3101	1900	NC	105085	50000	**1**	0,3	13	31	40	88
Soil	CN-SO-01	**11**	78	86	**87**	126	150	**10**	15	41	10	82	88
Sediment	TL-SE-02	NC	1601	2000	**44**	13	15	NC	NA	1	13	89	120
Sediment	RF-SE-02	NC	1396	1700	NC	707	700	**10**	15	6	16	98	120
Sediment	LV-SE-01	**14**	36	46	**23**	9	30	NC	NA	18	9	20	64
Sediment	LV-SE-02	**13**	20	28	**10**	33	70	NC	NA	22	15	92	130
Mean Rontec MDL		**9**			**37**			**16**			**28**		

Table 4. Evaluation of Sensitivity – Method Detection Limits for the Röntec PicoTAX (30 mm^2 detector) – continued.

Matrix	Sample No.	Selenium			Silver			Vanadium			Zinc		
		Rontec MDL	Rontec Conc	Ref. Lab Conc	Rontec MDL	Rontec Conc	Ref. Lab Conc	Rontec MDL	Rontec Conc	Ref. Lab Conc	Rontec MDL	Rontec Conc	Ref. Lab Conc
Soil	AS-SO-01	NC	NA	3	NC	NA	4	**17**	59	53	NC	3025	4100
Soil	BN-SO-01	**6**	35	52	NC	NA	150	**30**	71	44	NC	7330	7700
Soil	KP-SO-01	**9**	14	0,2	NC	NA	1	NC	NA	0,4	**13**	98	94
Soil	KP-SO-02	NC	NA	ND	NC	NA	0,1	**6**	5	2	**16**	8	15
Soil	SB-SO-02	NC	NA	2	NC	NA	ND	**13**	140	66	**80**	118	97
Soil	SB-SO-03	NC	NA	ND	NC	NA	0,2	**44**	136	9	NC	NA	14
Soil	WS-SO-02	**17**	99	3,6	NC	NA	230	NC	NA	24	NC	16576	11000
Soil	CN-SO-01	**7**	40	41	**58**	71	100	**42**	68	30	**63**	84	66
Sediment	TL-SE-02	**1**	1	ND	NC	NA	2	**97**	30	140	**19**	226	220
Sediment	RF-SE-02	NC	1	ND	NC	NA	11	**20**	53	43	NC	2070	2200
Sediment	LV-SE-01	NC	2	14	NC	NA	ND	**67**	141	150	**72**	33	16
Sediment	LV-SE-02	**4**	2	4	NC	NA	ND	**34**	171	46	**13**	75	62
Mean Rontec MDL		**7**			**58**			**37**			**39**		

Table 5. Evaluation of Precision – Relative Standard Deviations for the Röntec PicoTAX (30 mm^2 detector)

	AS-SO-01	BN-SO-01	CN-SO-01	KP-SO-02	SB-SO-02	SB-SO-03	WS-SO-02	LV-SE-01	LV-SE-02	RF-SE-02	TL-SE-02	Mean
Antimony	NC	NC	NC	NC	NC	NC	8.3 [1)]	NC	NC	NC	NC	NC
Arsenic	45.6	3.5	2.4	50.0	13.5	12.4	12.5	9.4	1.8	6.6	8.5	15
Cadmium	NC	13.8	100.9	NC	NC	NC	42.7	NC	NC	NC	NC	52
Chromium	48.6	12.5	80.1	8.8	42.2	7.7	38.5	22.5	35.0	32.3	20.1	32
Copper	4.4	3.5	4.5	10.3	3.2	14.9	8.4	12.0	20.3	7.3	4.8	9
Iron	3.4	4.1	11.3	13.3	2.0	8.1	15.6	2.7	6.0	5.9	2.9	7
Lead	3.2	2.8	21.9	1.7	(137.9) [2)]	(111.6) [2)]	8.4	78.9	9.1	9.7	(104.3) [2)]	17
Mercury	NC	NC	21.3	NC	58.0	14.3	36.9	NC	NC	20.6	NC	30
Nickel	55.1	14.7	3.8	9.0	6.5	6.3	23.7	13.8	5.3	5.3	4.6	13
Selenium	NC	5.2	5.4	NC	NC	NC	20.3	NC	(63.8) [2)]	NC	(33.3) [2)]	10
Silver	NC	NC	89.8	NC	NC	NC	NC	NC	NC	NC	NC	90
Vanadium	9.4	13.4	20.0	(34.3) [2)]	2.9	10.2	NC	15.2	6.3	12.2	(103.6) [2)]	11
Zinc	13.0	2.0	24.0	65.6	21.6	NC	10.1	16.7	5.5	24.9	2.6	19

[1)] Sample with extraordinary element distribution (Sb ~ 2000 µg/kg, K and Ca ~ 3000 resp. 4000 mg/kg)
[2)] Element concentration close to or below the MDL.

APPENDIX C

DATA VALIDATION SUMMARY REPORT

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APPENDIX

DATA VALIDATION REPORTS

ABBREVIATIONS AND ACRONYMS

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

1.0 INTRODUCTION

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

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

2.0 VALIDATION METHODOLOGY

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

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

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

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

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

- Holding times

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

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

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

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

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

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

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

Prior to the XRF demonstration, soil and sediment samples were collected from nine locations across the U.S. and then blended, dried, sieved, and homogenized in the characterization laboratory to produce a set of 326 reference samples. Each of these samples were subsequently analyzed by both the reference

laboratory and all participating technology vendors. As such, 326 prepared soil/sediment samples were delivered to Shealy for the measurement of total metals. The analytical program included the following analyses and methods:

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

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

3.0 DATA VALIDATION RESULTS

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

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

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

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

3.1 Holding Time

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

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

3.2 Calibration

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

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

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

3.3 Laboratory Blanks

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

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

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

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

Of all target analyte data, 2.6 percent of the data was qualified as undetected because of laboratory blank contamination (U, b), and less than 1 percent of the data was qualified as estimated (either J+, b or J-, b). The low occurrence of results affected by blank contamination indicates that the general quality of the

analytical data was not significantly compromised by blank contamination. Table 3 provides all results that were qualified based on laboratory blanks.

3.4 Laboratory Control Samples

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

3.5 Matrix Spike Samples

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

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

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

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

3.6 Serial Dilution Results

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

3.7 ICP Interference Check Samples

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

3.8 Target Analyte Identification and Quantitation

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

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

3.9 Quantitation Limit Verification

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

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

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

4.1 Precision

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

4.2 Accuracy

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

4.3 Representativeness

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

4.4 Completeness

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

4.5 Comparability

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

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

5.0 CONCLUSIONS FOR DATA QUALITY AND DATA USABILITY

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

6.0 REFERENCES

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

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

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

TABLES

TABLE 1: DATA VALIDATION QUALIFIERS AND COMMENT CODES

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

TABLE 2: QC CRITERIA

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

TABLE 3: DATA QUALIFICATION: LABORATORY METHOD BLANK CONTAMINATION

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

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

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

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

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

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

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

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

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Notes:

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

TABLE 5: DATA QUALIFICATION: SERIAL DILUTION EXCEEDANCES

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

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

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
LV-SO-34-XX	Antimony	870	mg/kg	J-	j
LV-SO-34-XX	Arsenic	110	mg/kg	J-	j
LV-SO-34-XX	Cadmium	2300	mg/kg	J-	j
LV-SO-34-XX	Chromium	2200	mg/kg	J-	j
LV-SO-34-XX	Iron	20000	mg/kg	J-	j
LV-SO-34-XX	Lead	3700	mg/kg	J-	j
LV-SO-34-XX	Nickel	1900	mg/kg	J-	j
LV-SO-34-XX	Selenium	220	mg/kg	J-	j
LV-SO-34-XX	Vanadium	230	mg/kg	J-	j
LV-SO-34-XX	Zinc	48	mg/kg	J-	j
RF-SE-16-XX	Antimony	85	mg/kg	J-	j
RF-SE-16-XX	Arsenic	72	mg/kg	J-	j
RF-SE-16-XX	Cadmium	310	mg/kg	J-	j
RF-SE-16-XX	Chromium	820	mg/kg	J-	j
RF-SE-16-XX	Copper	73	mg/kg	J-	j
RF-SE-16-XX	Iron	16000	mg/kg	J-	j
RF-SE-16-XX	Lead	24	mg/kg	J-	j
RF-SE-16-XX	Nickel	1700	mg/kg	J-	j
RF-SE-16-XX	Silver	130	mg/kg	J-	j
RF-SE-16-XX	Vanadium	32	mg/kg	J-	j
RF-SE-16-XX	Zinc	760	mg/kg	J-	j
RF-SE-24-XX	Arsenic	130	mg/kg	J+	j
RF-SE-24-XX	Cadmium	6.5	mg/kg	J+	j
RF-SE-24-XX	Chromium	74	mg/kg	J+	j
RF-SE-24-XX	Copper	860	mg/kg	J+	j
RF-SE-24-XX	Iron	24000	mg/kg	J+	j
RF-SE-24-XX	Lead	410	mg/kg	J+	j
RF-SE-24-XX	Nickel	170	mg/kg	J+	j
RF-SE-24-XX	Silver	3.8	mg/kg	J+	j
RF-SE-24-XX	Vanadium	46	mg/kg	J+	j
RF-SE-24-XX	Zinc	1400	mg/kg	J-	j
SB-SO-02-XX	Antimony	44	mg/kg	J-	e, j
SB-SO-02-XX	Arsenic	23	mg/kg	J-	j
SB-SO-02-XX	Lead	22	mg/kg	J-	j
SB-SO-02-XX	Mercury	130	mg/kg	J+	j
SB-SO-15-XX	Antimony	600	mg/kg	J-	j, e
SB-SO-15-XX	Arsenic	170	mg/kg	J-	j
SB-SO-15-XX	Chromium	91	mg/kg	J-	j
SB-SO-15-XX	Copper	30	mg/kg	J-	j

TABLE 5: DATA QUALIFICATIONS: 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, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		314	32	1,764	780	<LLD	
1	KP-SO-10-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		352	31	1,912	679	<LLD	
1	KP-SO-15-RU	RONTEC USA Inc.	n.d.		0		<LLD		368	29	2,037	600	<LLD	
1	KP-SO-18-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		282	20	1,567	506	<LLD	
1	KP-SO-22-RU	RONTEC USA Inc.	n.d.		0		<LLD		287	24	1,661	650	<LLD	
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-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		209	40	1,318	1,115	<LLD	
2	KP-SO-13-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		178	44	1,109	1,501	<LLD	
2	KP-SO-20-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		267	49	1,620	1,329	<LLD	
2	KP-SO-24-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		204	48	1,973	1,098	<LLD	
2	KP-SO-27-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		269	50	1,632	1,450	<LLD	
2	KP-SO-29-RU	RONTEC USA Inc.	n.d.		0		<LLD		220	45	1,373	1,531	<LLD	
2	KP-SO-32-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		261	43	1,790	1,368	<LLD	
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-RU	RONTEC USA Inc.	n.d.		8		48		158	220	1,484	9,684	<LLD	
3	KP-SO-16-RU	RONTEC USA Inc.	n.d.		3		60		190	199	1,781	6,648	<LLD	
3	KP-SO-23-RU	RONTEC USA Inc.	n.d.		4		218		159	213	1,581	6,909	<LLD	
3	KP-SO-26-RU	RONTEC USA Inc.	n.d.		2		102		136	177	1,150	5,934	<LLD	
3	KP-SO-31-RU	RONTEC USA Inc.	n.d.		5		177		205	190	1,598	5,661	<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	160	<LLD		<LLD		<LLD		11
1	KP-SO-10-RU	RONTEC USA Inc.	180	<LLD		<LLD		<LLD		9
1	KP-SO-15-RU	RONTEC USA Inc.	174	<LLD		<LLD		<LLD		15
1	KP-SO-18-RU	RONTEC USA Inc.	133	<LLD		<LLD		<LLD		11
1	KP-SO-22-RU	RONTEC USA Inc.	139	<LLD		29		2		9
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-RU	RONTEC USA Inc.	104	<LLD		<LLD		<LLD		19
2	KP-SO-13-RU	RONTEC USA Inc.	91	<LLD		<LLD		<LLD		17
2	KP-SO-20-RU	RONTEC USA Inc.	126	<LLD		<LLD		<LLD		21
2	KP-SO-24-RU	RONTEC USA Inc.	98	<LLD		<LLD		<LLD		18
2	KP-SO-27-RU	RONTEC USA Inc.	129	<LLD		43		<LLD		18
2	KP-SO-29-RU	RONTEC USA Inc.	105	<LLD		25		<LLD		22
2	KP-SO-32-RU	RONTEC USA Inc.	127	<LLD		<LLD		<LLD		18
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-RU	RONTEC USA Inc.	77	<LLD		28		<LLD		43
3	KP-SO-16-RU	RONTEC USA Inc.	89	<LLD		130		<LLD		49
3	KP-SO-23-RU	RONTEC USA Inc.	77	<LLD		55		<LLD		57
3	KP-SO-26-RU	RONTEC USA Inc.	71	<LLD		77		<LLD		35
3	KP-SO-31-RU	RONTEC USA Inc.	97	<LLD		133		<LLD		39

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		28		149		<LLD		834		2,343		27,985		<LLD	
4	KP-SO-03-RU	RONTEC USA Inc.	n.d.		32		91		<LLD		1,232		2,184		32,531		<LLD	
4	KP-SO-05-RU	RONTEC USA Inc.	n.d.		35		205		<LLD		686		1,081		20,657		<LLD	
4	KP-SO-09-RU	RONTEC USA Inc.	n.d.		20		901		<LLD		685		2,628		25,315		<LLD	
4	KP-SO-21-RU	RONTEC USA Inc.	n.d.		29		885		2		708		1,851		21,934		<LLD	
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-RU	RONTEC USA Inc.	n.d.		50		<LLD		130		52		32,525		108		<LLD	
5	WS-SO-08-RU	RONTEC USA Inc.	n.d.		49		<LLD		132		60		32,178		89		<LLD	
5	WS-SO-12-RU	RONTEC USA Inc.	n.d.		48		<LLD		204		58		30,859		85		<LLD	
5	WS-SO-17-RU	RONTEC USA Inc.	n.d.		62		<LLD		111		56		27,017		514		<LLD	
5	WS-SO-27-RU	RONTEC USA Inc.	n.d.		52		<LLD		125		55		30,405		121		<LLD	
5	WS-SO-30-RU	RONTEC USA Inc.	n.d.		73		<LLD		148		56		29,626		319		<LLD	
5	WS-SO-35-RU	RONTEC USA Inc.	n.d.		78		<LLD		166		73		44,622		185		<LLD	
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-RU	RONTEC USA Inc.	n.d.		402		<LLD		94		203		32,172		4,422		<LLD	
6	WS-SO-05-RU	RONTEC USA Inc.	n.d.		418		<LLD		109		139		29,831		4,231		<LLD	
6	WS-SO-09-RU	RONTEC USA Inc.	n.d.		281		<LLD		119		137		30,539		3,933		<LLD	
6	WS-SO-14-RU	RONTEC USA Inc.	n.d.		282		<LLD		123		152		31,519		5,216		<LLD	
6	WS-SO-26-RU	RONTEC USA Inc.	n.d.		334		<LLD		100		118		26,067		3,546		<LLD	
6	WS-SO-31-RU	RONTEC USA Inc.	n.d.		336		<LLD		131		148		33,898		4,960		<LLD	
6	WS-SO-33-RU	RONTEC USA Inc.	n.d.		351		<LLD		101		148		33,397		3,718		<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	2		9		113		<LLD		128	
4	KP-SO-03-RU	RONTEC USA Inc.	3		14		83		<LLD		146	
4	KP-SO-05-RU	RONTEC USA Inc.	5		4		181		<LLD		112	
4	KP-SO-09-RU	RONTEC USA Inc.	<LLD		11		154		<LLD		101	
4	KP-SO-21-RU	RONTEC USA Inc.	<LLD		11		240		6		103	
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-RU	RONTEC USA Inc.	41		<LLD		<LLD		65		266	
5	WS-SO-08-RU	RONTEC USA Inc.	38		<LLD		<LLD		66		254	
5	WS-SO-12-RU	RONTEC USA Inc.	51		<LLD		<LLD		59		248	
5	WS-SO-17-RU	RONTEC USA Inc.	36		<LLD		<LLD		45		249	
5	WS-SO-27-RU	RONTEC USA Inc.	39		<LLD		<LLD		57		245	
5	WS-SO-30-RU	RONTEC USA Inc.	58		<LLD		<LLD		61		296	
5	WS-SO-35-RU	RONTEC USA Inc.	64		<LLD		<LLD		89		358	
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-RU	RONTEC USA Inc.	32		<LLD		<LLD		58		784	
6	WS-SO-05-RU	RONTEC USA Inc.	40		<LLD		<LLD		54		809	
6	WS-SO-09-RU	RONTEC USA Inc.	45		<LLD		<LLD		50		669	
6	WS-SO-14-RU	RONTEC USA Inc.	42		<LLD		<LLD		44		817	
6	WS-SO-26-RU	RONTEC USA Inc.	36		<LLD		<LLD		38		623	
6	WS-SO-31-RU	RONTEC USA Inc.	63		<LLD		<LLD		53		848	
6	WS-SO-33-RU	RONTEC USA Inc.	35		<LLD		<LLD		63		729	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		1,907	346	107	793	38,275	25,213	<LLD	
7	WS-SO-04-RU	RONTEC USA Inc.	n.d.		1,890	432	127	755	36,058	25,936	<LLD	
7	WS-SO-15-RU	RONTEC USA Inc.	n.d.		1,496	161	34	477	30,305	19,872	<LLD	
7	WS-SO-22-RU	RONTEC USA Inc.	n.d.		1,553	399	59	559	32,206	21,032	<LLD	
7	WS-SO-34-RU	RONTEC USA Inc.	n.d.		2,095	365	75	693	40,750	25,972	<LLD	
8	WS-SO-02-XX	Reference Laboratory	130	J-	4200	98	49	1300	44,000	35,000	17	
8	WS-SO-16-XX	Reference Laboratory	110	J-	3900	91	59	1300	42,000	24,000	15	
8	WS-SO-18-XX	Reference Laboratory	130	J-	4100	95	63	1300	44,000	37,000	17	
8	WS-SO-21-XX	Reference Laboratory	120	J-	3900	90	43	1200	40,000	43,000	14	
8	WS-SO-24-XX	Reference Laboratory	97	J-	3600	81	54	1100	38,000	27,000	16	
8	WS-SO-29-XX	Reference Laboratory	120	J-	3800	90	51	1200	40,000	42,000	15	
8	WS-SO-37-XX	Reference Laboratory	120	J-	4100	95	63	1300	42,000	26,000	14	
8	WS-SO-02-RU	RONTEC USA Inc.	n.d.		3,327	803	26	1,424	43,729	48,941	<LLD	
8	WS-SO-16-RU	RONTEC USA Inc.	n.d.		3,670	827	54	1,217	40,476	47,661	<LLD	
8	WS-SO-18-RU	RONTEC USA Inc.	n.d.		3,343	998	24	1,151	35,927	45,505	<LLD	
8	WS-SO-21-RU	RONTEC USA Inc.	n.d.		76	<LLD	63	76	20,751	2,454	<LLD	
8	WS-SO-24-RU	RONTEC USA Inc.	n.d.		3,722	740	52	1,246	40,749	47,457	<LLD	
8	WS-SO-29-RU	RONTEC USA Inc.	n.d.		3,884	1,502	145	1,329	45,579	49,382	<LLD	
8	WS-SO-37-RU	RONTEC USA Inc.	n.d.		3,633	905	34	1,128	38,835	44,889	<LLD	
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-RU	RONTEC USA Inc.	n.d.		5,274	1,552	22	1,488	38,933	60,992	<LLD	
9	WS-SO-19-RU	RONTEC USA Inc.	n.d.		4,034	1,100	51	1,424	33,213	57,265	<LLD	
9	WS-SO-28-RU	RONTEC USA Inc.	n.d.		6,204	1,201	24	1,713	47,171	69,055	<LLD	
9	WS-SO-32-RU	RONTEC USA Inc.	n.d.		4,168	1,571	39	1,406	35,036	54,828	<LLD	
9	WS-SO-36-RU	RONTEC USA Inc.	n.d.		5,987	1,645	28	1,734	53,791	65,262	<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	33	<LLD		<LLD		26	3,696
7	WS-SO-04-RU	RONTEC USA Inc.	40	<LLD		<LLD		27	3,189
7	WS-SO-15-RU	RONTEC USA Inc.	22	7		<LLD		14	2,629
7	WS-SO-22-RU	RONTEC USA Inc.	35	4		107		12	3,620
7	WS-SO-34-RU	RONTEC USA Inc.	28	<LLD		<LLD		43	3,296
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-RU	RONTEC USA Inc.	15	14		37		8	6,365
8	WS-SO-16-RU	RONTEC USA Inc.	32	13		88		<LLD	5,526
8	WS-SO-18-RU	RONTEC USA Inc.	20	18		98		5	5,486
8	WS-SO-21-RU	RONTEC USA Inc.	298	256		<LLD		222	2,325
8	WS-SO-24-RU	RONTEC USA Inc.	35	24		64		3	5,855
8	WS-SO-29-RU	RONTEC USA Inc.	58	16		451		9	5,455
8	WS-SO-37-RU	RONTEC USA Inc.	25	8		30		10	4,967
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-RU	RONTEC USA Inc.	40	14		149		5	8,204
9	WS-SO-19-RU	RONTEC USA Inc.	29	33		199		<LLD	7,055
9	WS-SO-28-RU	RONTEC USA Inc.	35	29		212		<LLD	9,125
9	WS-SO-32-RU	RONTEC USA Inc.	30	20		217		<LLD	7,130
9	WS-SO-36-RU	RONTEC USA Inc.	65	10		65		7	9,334

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		45	<LLD	166	36	27,190	70	<LLD
10	BN-SO-10-RU	RONTEC USA Inc.	n.d.		3,042	494	741	113	26,768	85	1
10	BN-SO-15-RU	RONTEC USA Inc.	n.d.		53	<LLD	127	35	30,747	84	<LLD
10	BN-SO-18-RU	RONTEC USA Inc.	n.d.		41	<LLD	157	32	25,640	121	<LLD
10	BN-SO-28-RU	RONTEC USA Inc.	n.d.		42	<LLD	97	29	24,685	71	<LLD
10	BN-SO-31-RU	RONTEC USA Inc.	n.d.		40	<LLD	126	32	25,015	83	<LLD
10	BN-SO-35-RU	RONTEC USA Inc.	n.d.		38	<LLD	102	32	24,168	86	<LLD
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-RU	RONTEC USA Inc.	n.d.		112	<LLD	71	160	27,648	860	<LLD
11	BN-SO-04-RU	RONTEC USA Inc.	n.d.		132	<LLD	88	163	34,057	1,003	<LLD
11	BN-SO-17-RU	RONTEC USA Inc.	n.d.		126	<LLD	60	179	31,054	938	<LLD
11	BN-SO-22-RU	RONTEC USA Inc.	n.d.		99	<LLD	65	115	20,222	678	<LLD
11	BN-SO-27-RU	RONTEC USA Inc.	n.d.		126	<LLD	203	161	26,812	845	<LLD
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-RU	RONTEC USA Inc.	n.d.		642	46	126	989	32,118	5,894	<LLD
12	BN-SO-06-RU	RONTEC USA Inc.	n.d.		490	65	107	764	28,845	4,927	<LLD
12	BN-SO-08-RU	RONTEC USA Inc.	n.d.		751	43	121	1,103	36,069	6,416	<LLD
12	BN-SO-13-RU	RONTEC USA Inc.	n.d.		506	42	156	755	30,263	4,946	<LLD
12	BN-SO-20-RU	RONTEC USA Inc.	n.d.		583	46	128	767	23,249	4,276	<LLD
12	BN-SO-30-RU	RONTEC USA Inc.	n.d.		572	30	74	886	23,876	4,819	<LLD
12	BN-SO-34-RU	RONTEC USA Inc.	n.d.		586	63	130	833	28,817	4,714	<LLD

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	51	<LLD		<LLD		51	115
10	BN-SO-10-RU	RONTEC USA Inc.	2,982	135		20		139	4,299
10	BN-SO-15-RU	RONTEC USA Inc.	60	<LLD		<LLD		67	120
10	BN-SO-18-RU	RONTEC USA Inc.	59	<LLD		<LLD		42	100
10	BN-SO-28-RU	RONTEC USA Inc.	45	<LLD		53		55	103
10	BN-SO-31-RU	RONTEC USA Inc.	45	<LLD		<LLD		51	124
10	BN-SO-35-RU	RONTEC USA Inc.	46	<LLD		<LLD		41	118
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-RU	RONTEC USA Inc.	43	<LLD		<LLD		38	469
11	BN-SO-04-RU	RONTEC USA Inc.	54	<LLD		<LLD		74	525
11	BN-SO-17-RU	RONTEC USA Inc.	31	<LLD		<LLD		63	498
11	BN-SO-22-RU	RONTEC USA Inc.	26	<LLD		<LLD		38	384
11	BN-SO-27-RU	RONTEC USA Inc.	62	<LLD		<LLD		59	481
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-RU	RONTEC USA Inc.	134	6		<LLD		36	2,842
12	BN-SO-06-RU	RONTEC USA Inc.	67	1		<LLD		50	2,358
12	BN-SO-08-RU	RONTEC USA Inc.	86	5		<LLD		51	3,458
12	BN-SO-13-RU	RONTEC USA Inc.	127	6		<LLD		46	2,296
12	BN-SO-20-RU	RONTEC USA Inc.	69	4		<LLD		26	2,476
12	BN-SO-30-RU	RONTEC USA Inc.	52	5		<LLD		33	2,602
12	BN-SO-34-RU	RONTEC USA Inc.	78	4		<LLD		54	2,735

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		978		181		74		1,699		30,616		8,915		<LLD
13	BN-SO-16-RU	RONTEC USA Inc.	n.d.		1,106		254		84		1,641		35,714		9,449		<LLD
13	BN-SO-21-RU	RONTEC USA Inc.	n.d.		1,380		292		137		2,236		35,315		11,655		<LLD
13	BN-SO-25-RU	RONTEC USA Inc.	n.d.		938		263		99		1,509		26,293		8,292		<LLD
13	BN-SO-33-RU	RONTEC USA Inc.	n.d.		952		158		135		1,461		28,501		8,002		<LLD
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-RU	RONTEC USA Inc.	n.d.		1,700		615		105		2,998		34,577		16,038		<LLD
14	BN-SO-19-RU	RONTEC USA Inc.	n.d.		1,472		640		38		2,301		27,347		13,086		<LLD
14	BN-SO-26-RU	RONTEC USA Inc.	n.d.		1,451		764		88		2,252		28,547		13,665		<LLD
14	BN-SO-29-RU	RONTEC USA Inc.	n.d.		1,593		1,125		44		2,246		27,404		12,899		<LLD
14	BN-SO-32-RU	RONTEC USA Inc.	n.d.		1,584		539		38		2,660		35,560		14,268		<LLD
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-RU	RONTEC USA Inc.	n.d.		6		<LLD		397		391		27,736		904		<LLD
15	CN-SO-04-RU	RONTEC USA Inc.	n.d.		9		<LLD		284		647		38,159		980		<LLD
15	CN-SO-08-RU	RONTEC USA Inc.	n.d.		3		<LLD		352		508		47,045		1,682		<LLD
15	CN-SO-10-RU	RONTEC USA Inc.	n.d.		5		<LLD		343		694		50,623		1,415		<LLD
15	CN-SO-11-RU	RONTEC USA Inc.	n.d.		6		<LLD		359		615		45,485		1,379		<LLD

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	87		12		<LLD		29		4,783	
13	BN-SO-16-RU	RONTEC USA Inc.	91		13		<LLD		65		5,688	
13	BN-SO-21-RU	RONTEC USA Inc.	112		15		<LLD		33		7,118	
13	BN-SO-25-RU	RONTEC USA Inc.	98		9		<LLD		30		5,010	
13	BN-SO-33-RU	RONTEC USA Inc.	87		13		<LLD		25		4,611	
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-RU	RONTEC USA Inc.	93		24		<LLD		23		8,850	
14	BN-SO-19-RU	RONTEC USA Inc.	87		21		<LLD		24		7,024	
14	BN-SO-26-RU	RONTEC USA Inc.	66		18		108		34		6,757	
14	BN-SO-29-RU	RONTEC USA Inc.	125		22		75		14		7,871	
14	BN-SO-32-RU	RONTEC USA Inc.	115		21		<LLD		39		7,875	
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-RU	RONTEC USA Inc.	169		<LLD		<LLD		15		2,793	
15	CN-SO-04-RU	RONTEC USA Inc.	192		<LLD		<LLD		26		3,673	
15	CN-SO-08-RU	RONTEC USA Inc.	237		<LLD		<LLD		19		3,949	
15	CN-SO-10-RU	RONTEC USA Inc.	226		<LLD		<LLD		10		4,458	
15	CN-SO-11-RU	RONTEC USA Inc.	230		<LLD		<LLD		15		3,866	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As		Cd		Cr		Cu		Fe		Pb		Hg
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-RU	RONTEC USA Inc.	n.d.		6		<LLD		171		95		41,649		1,830		<LLD
16	AS-SO-06-RU	RONTEC USA Inc.	n.d.		3		14		223		121		31,662		1,922		<LLD
16	AS-SO-10-RU	RONTEC USA Inc.	n.d.		3		<LLD		234		149		61,654		2,459		<LLD
16	AS-SO-11-RU	RONTEC USA Inc.	n.d.		4		<LLD		273		151		69,371		2,153		5
16	AS-SO-13-RU	RONTEC USA Inc.	n.d.		4		<LLD		178		105		44,658		2,050		<LLD
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-RU	RONTEC USA Inc.	n.d.		1		152		355		163		55,703		4,085		<LLD
17	AS-SO-04-RU	RONTEC USA Inc.	n.d.		3		188		339		177		59,143		4,057		<LLD
17	AS-SO-07-RU	RONTEC USA Inc.	n.d.		1		<LLD		626		183		104,786		3,847		<LLD
17	AS-SO-09-RU	RONTEC USA Inc.	n.d.		1		<LLD		338		171		91,767		3,591		<LLD
17	AS-SO-12-RU	RONTEC USA Inc.	n.d.		1		35		723		199		91,093		4,439		<LLD
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-RU	RONTEC USA Inc.	n.d.		17		<LLD		206		49		43,686		11		30
18	SB-SO-06-RU	RONTEC USA Inc.	n.d.		18		21		194		47		45,687		8		144
18	SB-SO-14-RU	RONTEC USA Inc.	n.d.		18		<LLD		223		51		45,684		14		41
18	SB-SO-38-RU	RONTEC USA Inc.	n.d.		13		<LLD		228		45		37,030		15		41
18	SB-SO-41-RU	RONTEC USA Inc.	n.d.		18		<LLD		233		59		52,708		19		65
18	SB-SO-47-RU	RONTEC USA Inc.	n.d.		25		<LLD		438		82		55,521		61		2
18	SB-SO-51-RU	RONTEC USA Inc.	n.d.		22		<LLD		260		62		57,083		5		1

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
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-RU	RONTEC USA Inc.	<LLD		81		<LLD		50		3,367	
16	AS-SO-06-RU	RONTEC USA Inc.	<LLD		46		<LLD		40		3,334	
16	AS-SO-10-RU	RONTEC USA Inc.	<LLD		95		<LLD		39		5,103	
16	AS-SO-11-RU	RONTEC USA Inc.	<LLD		105		<LLD		33		4,747	
16	AS-SO-13-RU	RONTEC USA Inc.	<LLD		87		<LLD		39		3,751	
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-RU	RONTEC USA Inc.	<LLD		81		199		55		7,605	
17	AS-SO-04-RU	RONTEC USA Inc.	<LLD		86		239		45		7,598	
17	AS-SO-07-RU	RONTEC USA Inc.	<LLD		129		<LLD		12		9,169	
17	AS-SO-09-RU	RONTEC USA Inc.	<LLD		101		<LLD		25		7,149	
17	AS-SO-12-RU	RONTEC USA Inc.	<LLD		108		<LLD		30		8,832	
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-RU	RONTEC USA Inc.	209		<LLD		<LLD		85		114	
18	SB-SO-06-RU	RONTEC USA Inc.	192		<LLD		<LLD		111		114	
18	SB-SO-14-RU	RONTEC USA Inc.	177		<LLD		<LLD		78		116	
18	SB-SO-38-RU	RONTEC USA Inc.	165		<LLD		<LLD		56		107	
18	SB-SO-41-RU	RONTEC USA Inc.	271		<LLD		<LLD		132		140	
18	SB-SO-47-RU	RONTEC USA Inc.	254		<LLD		<LLD		146		142	
18	SB-SO-51-RU	RONTEC USA Inc.	257		<LLD		<LLD		148		141	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As	Cd		Cr	Cu	Fe	Pb	Hg
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-RU	RONTEC USA Inc.	n.d.		22	<LLD		176	42	40,162	11	137
19	SB-SO-18-RU	RONTEC USA Inc.	n.d.		19	<LLD		204	49	43,287	12	358
19	SB-SO-30-RU	RONTEC USA Inc.	n.d.		13	<LLD		120	31	28,889	11	219
19	SB-SO-40-RU	RONTEC USA Inc.	n.d.		21	<LLD		227	51	44,833	7	192
19	SB-SO-53-RU	RONTEC USA Inc.	n.d.		14	<LLD		142	37	31,242	8	163
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-RU	RONTEC USA Inc.	n.d.		27	<LLD		195	50	47,131	13	760
20	SB-SO-11-RU	RONTEC USA Inc.	n.d.		24	<LLD		246	45	41,552	12	528
20	SB-SO-21-RU	RONTEC USA Inc.	n.d.		19	<LLD		156	34	30,321	8	820
20	SB-SO-39-RU	RONTEC USA Inc.	n.d.		25	<LLD		174	45	40,830	5	776
20	SB-SO-42-RU	RONTEC USA Inc.	n.d.		29	<LLD		232	63	49,786	34	1,109
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-RU	RONTEC USA Inc.	n.d.		29	<LLD		173	30	29,418	14	1,570
21	SB-SO-25-RU	RONTEC USA Inc.	n.d.		24	<LLD		163	29	26,599	12	1,867
21	SB-SO-27-RU	RONTEC USA Inc.	n.d.		21	<LLD		107	28	25,354	14	1,446
21	SB-SO-35-RU	RONTEC USA Inc.	n.d.		27	<LLD		365	40	33,860	30	1,954
21	SB-SO-44-RU	RONTEC USA Inc.	n.d.		31	<LLD		206	46	41,728	19	2,550

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se		Ag		V	Zn	
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-RU	RONTEC USA Inc.	180	<LLD		<LLD		76	93	
19	SB-SO-18-RU	RONTEC USA Inc.	178	<LLD		<LLD		76	102	
19	SB-SO-30-RU	RONTEC USA Inc.	141	<LLD		<LLD		82	77	
19	SB-SO-40-RU	RONTEC USA Inc.	225	<LLD		<LLD		98	117	
19	SB-SO-53-RU	RONTEC USA Inc.	136	<LLD		<LLD		65	76	
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-RU	RONTEC USA Inc.	214	<LLD		<LLD		125	107	
20	SB-SO-11-RU	RONTEC USA Inc.	208	<LLD		<LLD		107	117	
20	SB-SO-21-RU	RONTEC USA Inc.	232	<LLD		<LLD		65	72	
20	SB-SO-39-RU	RONTEC USA Inc.	177	<LLD		<LLD		53	99	
20	SB-SO-42-RU	RONTEC USA Inc.	226	<LLD		<LLD		128	134	
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-RU	RONTEC USA Inc.	146	<LLD		<LLD		72	67	
21	SB-SO-25-RU	RONTEC USA Inc.	122	<LLD		<LLD		84	62	
21	SB-SO-27-RU	RONTEC USA Inc.	136	<LLD		<LLD		78	56	
21	SB-SO-35-RU	RONTEC USA Inc.	216	<LLD		<LLD		50	79	
21	SB-SO-44-RU	RONTEC USA Inc.	235	<LLD		<LLD		109	97	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As		Cd		Cr	Cu	Fe	Pb		Hg	
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-RU	RONTEC USA Inc.	n.d.		72		<LLD		93	11	7,298	60		10,967	
22	SB-SO-28-RU	RONTEC USA Inc.	n.d.		37		<LLD		28	7	3,460	29		3,335	
22	SB-SO-32-RU	RONTEC USA Inc.	n.d.		60		<LLD		97	14	6,026	40		5,855	
22	SB-SO-43-RU	RONTEC USA Inc.	n.d.		52		<LLD		84	11	5,353	39		5,577	
22	SB-SO-48-RU	RONTEC USA Inc.	n.d.		61		<LLD		70	10	5,512	41		5,737	
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-RU	RONTEC USA Inc.	n.d.		34		<LLD		186	47	43,933	23		100	
23	SB-SO-07-RU	RONTEC USA Inc.	n.d.		45		<LLD		186	53	48,139	33		103	
23	SB-SO-10-RU	RONTEC USA Inc.	n.d.		33		17		153	40	38,473	16		89	
23	SB-SO-26-RU	RONTEC USA Inc.	n.d.		31		<LLD		296	36	34,334	22		489	
23	SB-SO-50-RU	RONTEC USA Inc.	n.d.		50		<LLD		865	60	59,622	17		182	
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-RU	RONTEC USA Inc.	n.d.		86		<LLD		205	51	59,634	20		236	
24	SB-SO-16-RU	RONTEC USA Inc.	n.d.		71		<LLD		194	42	43,135	14		186	
24	SB-SO-24-RU	RONTEC USA Inc.	n.d.		75		<LLD		136	40	40,606	18		182	
24	SB-SO-45-RU	RONTEC USA Inc.	n.d.		85		<LLD		170	53	52,600	20		224	
24	SB-SO-52-RU	RONTEC USA Inc.	n.d.		77		<LLD		159	43	47,037	19		362	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se		Ag		V	Zn
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-RU	RONTEC USA Inc.	14	<LLD		<LLD		130	<LLD
22	SB-SO-28-RU	RONTEC USA Inc.	14	<LLD		<LLD		70	25
22	SB-SO-32-RU	RONTEC USA Inc.	37	<LLD		<LLD		67	46
22	SB-SO-43-RU	RONTEC USA Inc.	17	<LLD		<LLD		126	11
22	SB-SO-48-RU	RONTEC USA Inc.	16	<LLD		<LLD		167	<LLD
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-RU	RONTEC USA Inc.	182	<LLD		<LLD		105	112
23	SB-SO-07-RU	RONTEC USA Inc.	181	<LLD		<LLD		81	132
23	SB-SO-10-RU	RONTEC USA Inc.	157	<LLD		<LLD		86	98
23	SB-SO-26-RU	RONTEC USA Inc.	153	<LLD		<LLD		94	101
23	SB-SO-50-RU	RONTEC USA Inc.	232	<LLD		<LLD		137	152
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-RU	RONTEC USA Inc.	157	<LLD		<LLD		90	120
24	SB-SO-16-RU	RONTEC USA Inc.	166	<LLD		<LLD		104	94
24	SB-SO-24-RU	RONTEC USA Inc.	143	<LLD		<LLD		73	92
24	SB-SO-45-RU	RONTEC USA Inc.	181	<LLD		<LLD		105	125
24	SB-SO-52-RU	RONTEC USA Inc.	167	<LLD		<LLD		105	109

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As		Cd		Cr		Cu		Fe		Pb		Hg	
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-RU	RONTEC USA Inc.	n.d.		152		<LLD		146		39		53,366		17		377	
25	SB-SO-19-RU	RONTEC USA Inc.	n.d.		104		<LLD		96		30		38,031		29		253	
25	SB-SO-33-RU	RONTEC USA Inc.	n.d.		140		<LLD		381		50		54,289		14		498	
25	SB-SO-37-RU	RONTEC USA Inc.	n.d.		145		<LLD		250		49		58,186		11		446	
25	SB-SO-55-RU	RONTEC USA Inc.	n.d.		133		<LLD		174		41		49,892		14		358	
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-RU	RONTEC USA Inc.	n.d.		217		<LLD		153		42		61,880		36		610	
26	SB-SO-15-RU	RONTEC USA Inc.	n.d.		174		<LLD		136		40		49,739		38		426	
26	SB-SO-17-RU	RONTEC USA Inc.	n.d.		161		<LLD		93		35		43,888		37		412	
26	SB-SO-46-RU	RONTEC USA Inc.	n.d.		190		<LLD		92		35		53,422		25		469	
26	SB-SO-54-RU	RONTEC USA Inc.	n.d.		210		<LLD		119		38		58,068		32		670	
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-RU	RONTEC USA Inc.	n.d.		1		<LLD		133		5		1,371		414		<LLD	
27	KP-SE-11-RU	RONTEC USA Inc.	n.d.		3		<LLD		110		5		1,235		387		<LLD	
27	KP-SE-17-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		96		6		1,304		438		<LLD	
27	KP-SE-25-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		110		5		1,180		437		<LLD	
27	KP-SE-30-RU	RONTEC USA Inc.	n.d.		<LLD		<LLD		130		5		1,509		420		<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
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-RU	RONTEC USA Inc.	138		<LLD		<LLD		107		98	
25	SB-SO-19-RU	RONTEC USA Inc.	114		<LLD		<LLD		73		57	
25	SB-SO-33-RU	RONTEC USA Inc.	429		35		<LLD		57		86	
25	SB-SO-37-RU	RONTEC USA Inc.	152		<LLD		<LLD		52		99	
25	SB-SO-55-RU	RONTEC USA Inc.	122		<LLD		<LLD		66		80	
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-RU	RONTEC USA Inc.	125		<LLD		<LLD		99		131	
26	SB-SO-15-RU	RONTEC USA Inc.	87		<LLD		<LLD		53		100	
26	SB-SO-17-RU	RONTEC USA Inc.	81		<LLD		<LLD		57		47	
26	SB-SO-46-RU	RONTEC USA Inc.	89		<LLD		<LLD		80		67	
26	SB-SO-54-RU	RONTEC USA Inc.	84		<LLD		<LLD		65		61	
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-RU	RONTEC USA Inc.	61		<LLD		<LLD		<LLD		7	
27	KP-SE-11-RU	RONTEC USA Inc.	47		<LLD		<LLD		2		7	
27	KP-SE-17-RU	RONTEC USA Inc.	45		<LLD		<LLD		<LLD		7	
27	KP-SE-25-RU	RONTEC USA Inc.	50		<LLD		<LLD		<LLD		7	
27	KP-SE-30-RU	RONTEC USA Inc.	63		<LLD		<LLD		3		7	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		2	<LLD		46		3		1,038		568		<LLD	
28	KP-SE-12-RU	RONTEC USA Inc.	n.d.		<LLD	<LLD		41		2		1,245		507		<LLD	
28	KP-SE-14-RU	RONTEC USA Inc.	n.d.		<LLD	<LLD		48		2		954		559		<LLD	
28	KP-SE-19-RU	RONTEC USA Inc.	n.d.		1	<LLD		39		3		881		564		<LLD	
28	KP-SE-28-RU	RONTEC USA Inc.	n.d.		<LLD	<LLD		50		2		713		400		<LLD	
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-RU	RONTEC USA Inc.	n.d.		12	<LLD		54		1,888		47,319		37		<LLD	
29	TL-SE-10-RU	RONTEC USA Inc.	n.d.		8	<LLD		45		1,002		31,807		23		<LLD	
29	TL-SE-12-RU	RONTEC USA Inc.	n.d.		10	<LLD		30		1,235		27,621		16		<LLD	
29	TL-SE-15-RU	RONTEC USA Inc.	n.d.		8	10		29		829		25,442		19		<LLD	
29	TL-SE-20-RU	RONTEC USA Inc.	n.d.		15	<LLD		57		2,549		50,279		33		<LLD	
29	TL-SE-24-RU	RONTEC USA Inc.	n.d.		21	<LLD		56		2,120		53,443		33		<LLD	
29	TL-SE-26-RU	RONTEC USA Inc.	n.d.		13	<LLD		55		1,842		49,680		42		<LLD	
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-RU	RONTEC USA Inc.	n.d.		7	<LLD		62		1,061		48,412		8		<LLD	
30	TL-SE-19-RU	RONTEC USA Inc.	n.d.		18	<LLD		76		1,746		75,114		39		<LLD	
30	TL-SE-23-RU	RONTEC USA Inc.	n.d.		12	555		85		1,775		75,899		11		<LLD	
30	TL-SE-25-RU	RONTEC USA Inc.	n.d.		13	<LLD		84		1,891		74,691		2		<LLD	
30	TL-SE-31-RU	RONTEC USA Inc.	n.d.		13	<LLD		85		1,857		80,869		10		<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	20		<LLD		<LLD		4		23
28	KP-SE-12-RU	RONTEC USA Inc.	22		<LLD		<LLD		23		9
28	KP-SE-14-RU	RONTEC USA Inc.	21		<LLD		<LLD		<LLD		8
28	KP-SE-19-RU	RONTEC USA Inc.	17		<LLD		<LLD		<LLD		6
28	KP-SE-28-RU	RONTEC USA Inc.	23		<LLD		<LLD		<LLD		7
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-RU	RONTEC USA Inc.	54		<LLD		<LLD		60		186
29	TL-SE-10-RU	RONTEC USA Inc.	33		<LLD		<LLD		36		111
29	TL-SE-12-RU	RONTEC USA Inc.	94		<LLD		<LLD		37		119
29	TL-SE-15-RU	RONTEC USA Inc.	30		<LLD		<LLD		27		97
29	TL-SE-20-RU	RONTEC USA Inc.	82		<LLD		<LLD		77		187
29	TL-SE-24-RU	RONTEC USA Inc.	61		<LLD		<LLD		54		221
29	TL-SE-26-RU	RONTEC USA Inc.	56		<LLD		<LLD		46		187
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-RU	RONTEC USA Inc.	63		<LLD		<LLD		49		133
30	TL-SE-19-RU	RONTEC USA Inc.	93		<LLD		<LLD		109		307
30	TL-SE-23-RU	RONTEC USA Inc.	99		<LLD		93		93		222
30	TL-SE-25-RU	RONTEC USA Inc.	92		<LLD		<LLD		77		219
30	TL-SE-31-RU	RONTEC USA Inc.	101		<LLD		<LLD		118		236

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		7		<LLD		104		1,007	35,595		59		<LLD	
31	TL-SE-11-RU	RONTEC USA Inc.	n.d.		5		<LLD		80		674	23,745		30		<LLD	
31	TL-SE-14-RU	RONTEC USA Inc.	n.d.		4		<LLD		55		344	11,035		18		<LLD	
31	TL-SE-18-RU	RONTEC USA Inc.	n.d.		4		<LLD		61		560	14,856		22		<LLD	
31	TL-SE-22-RU	RONTEC USA Inc.	n.d.		11		<LLD		167		783	31,767		43		<LLD	
31	TL-SE-27-RU	RONTEC USA Inc.	n.d.		6		<LLD		89		713	22,219		39		<LLD	
31	TL-SE-29-RU	RONTEC USA Inc.	n.d.		8		<LLD		129		700	22,100		43		<LLD	
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-RU	RONTEC USA Inc.	n.d.		25		<LLD		83		36	34,858		50		<LLD	
32	LV-SE-10-RU	RONTEC USA Inc.	n.d.		44		<LLD		168		47	36,427		37		2	
32	LV-SE-22-RU	RONTEC USA Inc.	n.d.		30		<LLD		128		42	32,042		32		<LLD	
32	LV-SE-25-RU	RONTEC USA Inc.	n.d.		39		<LLD		59		31	31,844		26		<LLD	
32	LV-SE-31-RU	RONTEC USA Inc.	n.d.		29		<LLD		79		35	28,942		50		<LLD	
32	LV-SE-35-RU	RONTEC USA Inc.	n.d.		38		<LLD		81		34	26,193		21		<LLD	
32	LV-SE-50-RU	RONTEC USA Inc.	n.d.		31		<LLD		73		34	36,500		71		<LLD	
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-RU	RONTEC USA Inc.	n.d.		247		<LLD		89		44	103,600		14		2	
33	LV-SE-26-RU	RONTEC USA Inc.	n.d.		201		<LLD		95		43	84,371		12		21	
33	LV-SE-33-RU	RONTEC USA Inc.	n.d.		185		<LLD		59		35	71,708		20		7	
33	LV-SE-39-RU	RONTEC USA Inc.	n.d.		191		<LLD		75		30	72,280		46		<LLD	
33	LV-SE-42-RU	RONTEC USA Inc.	n.d.		145		<LLD		54		31	62,867		137		<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	113		<LLD		<LLD		49		126	
31	TL-SE-11-RU	RONTEC USA Inc.	80		<LLD		<LLD		34		76	
31	TL-SE-14-RU	RONTEC USA Inc.	57		<LLD		<LLD		18		48	
31	TL-SE-18-RU	RONTEC USA Inc.	92		<LLD		<LLD		22		52	
31	TL-SE-22-RU	RONTEC USA Inc.	216		<LLD		<LLD		47		102	
31	TL-SE-27-RU	RONTEC USA Inc.	215		<LLD		<LLD		27		87	
31	TL-SE-29-RU	RONTEC USA Inc.	121		<LLD		<LLD		29		93	
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-RU	RONTEC USA Inc.	110		<LLD		<LLD		84		88	
32	LV-SE-10-RU	RONTEC USA Inc.	129		1		<LLD		57		86	
32	LV-SE-22-RU	RONTEC USA Inc.	137		1		<LLD		80		88	
32	LV-SE-25-RU	RONTEC USA Inc.	119		0		<LLD		42		76	
32	LV-SE-31-RU	RONTEC USA Inc.	123		1		<LLD		64		74	
32	LV-SE-35-RU	RONTEC USA Inc.	86		2		<LLD		53		70	
32	LV-SE-50-RU	RONTEC USA Inc.	83		2		<LLD		91		92	
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-RU	RONTEC USA Inc.	92		<LLD		<LLD		64		104	
33	LV-SE-26-RU	RONTEC USA Inc.	67		<LLD		<LLD		66		88	
33	LV-SE-33-RU	RONTEC USA Inc.	37		1		<LLD		38		77	
33	LV-SE-39-RU	RONTEC USA Inc.	53		2		<LLD		59		71	
33	LV-SE-42-RU	RONTEC USA Inc.	74		3		<LLD		85		71	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	n.d.		595	<LLD		50	37	200,522	1		4	
34	LV-SE-19-RU	RONTEC USA Inc.	n.d.		541	<LLD		26	33	181,384	<LLD		<LLD	
34	LV-SE-27-RU	RONTEC USA Inc.	n.d.		424	<LLD		35	29	150,539	1		3	
34	LV-SE-36-RU	RONTEC USA Inc.	n.d.		455	<LLD		54	34	150,866	12		<LLD	
34	LV-SE-38-RU	RONTEC USA Inc.	n.d.		505	<LLD		94	36	164,068	17		3	
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-RU	RONTEC USA Inc.	n.d.		825	<LLD		50	44	253,402	<LLD		<LLD	
35	LV-SE-18-RU	RONTEC USA Inc.	n.d.		775	<LLD		44	56	202,701	<LLD		<LLD	
35	LV-SE-23-RU	RONTEC USA Inc.	n.d.		772	<LLD		37	40	228,099	<LLD		<LLD	
35	LV-SE-45-RU	RONTEC USA Inc.	n.d.		667	<LLD		134	33	179,984	30		<LLD	
35	LV-SE-48-RU	RONTEC USA Inc.	n.d.		653	<LLD		142	29	168,659	4		<LLD	
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-RU	RONTEC USA Inc.	n.d.		<LLD	1,404		<LLD	7	730	14		<LLD	
36	LV-SE-14-RU	RONTEC USA Inc.	n.d.		1	372		4	10	673	14		<LLD	
36	LV-SE-21-RU	RONTEC USA Inc.	n.d.		<LLD	1,197		<LLD	12	672	13		<LLD	
36	LV-SE-24-RU	RONTEC USA Inc.	n.d.		3	927		<LLD	14	797	10		<LLD	
36	LV-SE-32-RU	RONTEC USA Inc.	n.d.		2	<LLD		<LLD	8	436	8		<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax 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-RU	RONTEC USA Inc.	160		<LLD		<LLD		79		88	
34	LV-SE-19-RU	RONTEC USA Inc.	61		1		<LLD		89		78	
34	LV-SE-27-RU	RONTEC USA Inc.	54		<LLD		<LLD		40		86	
34	LV-SE-36-RU	RONTEC USA Inc.	45		2		<LLD		40		68	
34	LV-SE-38-RU	RONTEC USA Inc.	23		4		<LLD		30		76	
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-RU	RONTEC USA Inc.	70		2		<LLD		116		48	
35	LV-SE-18-RU	RONTEC USA Inc.	46		5		<LLD		72		41	
35	LV-SE-23-RU	RONTEC USA Inc.	54		2		<LLD		64		45	
35	LV-SE-45-RU	RONTEC USA Inc.	25		3		<LLD		62		35	
35	LV-SE-48-RU	RONTEC USA Inc.	19		2		450		64		29	
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-RU	RONTEC USA Inc.	44		<LLD		514		<LLD		7	
36	LV-SE-14-RU	RONTEC USA Inc.	39		<LLD		193		3		8	
36	LV-SE-21-RU	RONTEC USA Inc.	40		<LLD		428		<LLD		13	
36	LV-SE-24-RU	RONTEC USA Inc.	38		<LLD		400		<LLD		8	
36	LV-SE-32-RU	RONTEC USA Inc.	32		<LLD		<LLD		3		6	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As		Cd		Cr		Cu		Fe		Pb		Hg	
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-RU	RONTEC USA Inc.	n.d.		42		<LLD		51		27		39,625		109		5	
37	LV-SE-16-RU	RONTEC USA Inc.	n.d.		33		<LLD		85		35		32,967		102		5	
37	LV-SE-28-RU	RONTEC USA Inc.	n.d.		27		<LLD		62		25		278		94		<LLD	
37	LV-SE-30-RU	RONTEC USA Inc.	n.d.		39		<LLD		49		22		22,219		54		<LLD	
37	LV-SE-47-RU	RONTEC USA Inc.	n.d.		28		<LLD		54		26		26,058		103		<LLD	
38	LV-SE-11-XX	Reference Laboratory	1.4	UJ	150		6.6		120		270		42,000		7		2.8	
38	LV-SE-29-XX	Reference Laboratory	1.4	UJ	150		6.3		120		260		42,000		7	J+	1.5	J-
38	LV-SE-44-XX	Reference Laboratory	1.4	U	140		6.1		120		250		40,000		8		1.5	
38	LV-SE-46-XX	Reference Laboratory	0.88	U	110		5		92		200		32,000		6		1.4	
38	LV-SE-52-XX	Reference Laboratory	1.4	U	160		6.8		130		280		44,000		8		21	
38	LV-SE-11-RU	RONTEC USA Inc.	n.d.		147		<LLD		74		254		47,660		11		1	
38	LV-SE-29-RU	RONTEC USA Inc.	n.d.		138		<LLD		125		237		39,654		4		<LLD	
38	LV-SE-44-RU	RONTEC USA Inc.	n.d.		151		<LLD		110		276		46,297		25		<LLD	
38	LV-SE-46-RU	RONTEC USA Inc.	n.d.		117		<LLD		98		220		38,544		34		<LLD	
38	LV-SE-52-RU	RONTEC USA Inc.	n.d.		149		<LLD		112		282		49,693		29		<LLD	
39	RF-SE-07-XX	Reference Laboratory	1.3	U	12		0.5	U	92		81		17,000		24		0.091	U
39	RF-SE-12-XX	Reference Laboratory	1.2	U	14		0.5	U	100		110		20,000		25		0.099	U
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	
39	RF-SE-36-XX	Reference Laboratory	1.2	U	12		0.5	U	91		82		17,000		22		0.081	U
39	RF-SE-42-XX	Reference Laboratory	1.3	UJ	14		0.56		110		95		19,000		28		0.084	U
39	RF-SE-45-XX	Reference Laboratory	1.3	UJ	15		0.52	U	110		100		21,000		33		0.084	U
39	RF-SE-53-XX	Reference Laboratory	1.3	UJ	14		0.57	U	110		95		19,000		28		0.084	U
39	RF-SE-07-RU	RONTEC USA Inc.	n.d.		21		<LLD		90		104		22,218		37		<LLD	
39	RF-SE-12-RU	RONTEC USA Inc.	n.d.		27		<LLD		145		103		24,091		28		<LLD	
39	RF-SE-23-RU	RONTEC USA Inc.	n.d.		5		<LLD		83		82		20,014		76		<LLD	
39	RF-SE-36-RU	RONTEC USA Inc.	n.d.		21		<LLD		111		112		27,871		55		<LLD	
39	RF-SE-42-RU	RONTEC USA Inc.	n.d.		16		<LLD		85		84		21,471		38		2	
39	RF-SE-45-RU	RONTEC USA Inc.	n.d.		20		<LLD		91		92		22,497		51		<LLD	
39	RF-SE-53-RU	RONTEC USA Inc.	n.d.		15		<LLD		72		88		20,362		47		<LLD	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
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-RU	RONTEC USA Inc.	100		1		<LLD		57		100	
37	LV-SE-16-RU	RONTEC USA Inc.	120		2		<LLD		56		90	
37	LV-SE-28-RU	RONTEC USA Inc.	104		1		<LLD		45		63	
37	LV-SE-30-RU	RONTEC USA Inc.	90		2		<LLD		39		64	
37	LV-SE-47-RU	RONTEC USA Inc.	84		2		134		48		70	
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-RU	RONTEC USA Inc.	883		<LLD		<LLD		18		206	
38	LV-SE-29-RU	RONTEC USA Inc.	821		<LLD		<LLD		17		184	
38	LV-SE-44-RU	RONTEC USA Inc.	946		1		<LLD		29		227	
38	LV-SE-46-RU	RONTEC USA Inc.	772		<LLD		<LLD		24		177	
38	LV-SE-52-RU	RONTEC USA Inc.	952		<LLD		423		31		228	
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-RU	RONTEC USA Inc.	137		2		<LLD		15		141	
39	RF-SE-12-RU	RONTEC USA Inc.	175		<LLD		<LLD		33		123	
39	RF-SE-23-RU	RONTEC USA Inc.	120		<LLD		28		27		114	
39	RF-SE-36-RU	RONTEC USA Inc.	170		<LLD		38		41		207	
39	RF-SE-42-RU	RONTEC USA Inc.	132		<LLD		<LLD		27		122	
39	RF-SE-45-RU	RONTEC USA Inc.	100		<LLD		<LLD		49		140	
39	RF-SE-53-RU	RONTEC USA Inc.	140		<LLD		59		30		119	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As	Cd	Cr	Cu	Fe	Pb	Hg
40	RF-SE-03-XX	Reference Laboratory	1.2	UJ	27	1.3	93	200	17,000	88	0.48
40	RF-SE-28-XX	Reference Laboratory	1.2	UJ	31	1.5	100	220	18,000	99	0.57
40	RF-SE-38-XX	Reference Laboratory	1.2	UJ	27	1.2	90	190	16,000	83	0.41
40	RF-SE-49-XX	Reference Laboratory	1.2	UJ	31	1.5	100	220	18,000	97	0.43
40	RF-SE-55-XX	Reference Laboratory	1.2	UJ	24	1.1	91	180	15,000	75	0.42
40	RF-SE-03-RU	RONTEC USA Inc.	n.d.		21	<LLD	86	185	22,413	140	<LLD
40	RF-SE-28-RU	RONTEC USA Inc.	n.d.		19	<LLD	82	174	16,154	112	<LLD
40	RF-SE-38-RU	RONTEC USA Inc.	n.d.		44	<LLD	171	219	26,782	94	<LLD
40	RF-SE-49-RU	RONTEC USA Inc.	n.d.		28	<LLD	79	185	19,425	116	<LLD
40	RF-SE-55-RU	RONTEC USA Inc.	n.d.		26	<LLD	83	202	20,676	176	4
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-RU	RONTEC USA Inc.	n.d.		52	<LLD	76	397	18,911	276	1
41	RF-SE-13-RU	RONTEC USA Inc.	n.d.		93	<LLD	150	373	20,401	239	<LLD
41	RF-SE-27-RU	RONTEC USA Inc.	n.d.		90	<LLD	78	531	24,575	250	<LLD
41	RF-SE-31-RU	RONTEC USA Inc.	n.d.		79	<LLD	97	389	23,439	240	<LLD
41	RF-SE-58-RU	RONTEC USA Inc.	n.d.		50	<LLD	60	404	21,685	287	<LLD
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-RU	RONTEC USA Inc.	n.d.		75	<LLD	56	543	19,787	342	2
42	RF-SE-22-RU	RONTEC USA Inc.	n.d.		83	<LLD	66	510	23,425	344	<LLD
42	RF-SE-25-RU	RONTEC USA Inc.	n.d.		83	<LLD	53	539	24,307	255	<LLD
42	RF-SE-30-RU	RONTEC USA Inc.	n.d.		68	<LLD	92	546	22,093	360	2
42	RF-SE-57-RU	RONTEC USA Inc.	n.d.		74	<LLD	54	530	21,034	365	2

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se		Ag		V	Zn
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-RU	RONTEC USA Inc.	108	<LLD		<LLD		37	415
40	RF-SE-28-RU	RONTEC USA Inc.	138	<LLD		221		42	190
40	RF-SE-38-RU	RONTEC USA Inc.	108	<LLD		36		50	304
40	RF-SE-49-RU	RONTEC USA Inc.	115	<LLD		<LLD		30	259
40	RF-SE-55-RU	RONTEC USA Inc.	95	<LLD		<LLD		50	241
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-RU	RONTEC USA Inc.	78	<LLD		<LLD		33	616
41	RF-SE-13-RU	RONTEC USA Inc.	82	<LLD		<LLD		35	579
41	RF-SE-27-RU	RONTEC USA Inc.	150	<LLD		<LLD		29	790
41	RF-SE-31-RU	RONTEC USA Inc.	85	<LLD		<LLD		24	731
41	RF-SE-58-RU	RONTEC USA Inc.	77	<LLD		120		55	631
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-RU	RONTEC USA Inc.	67	<LLD		<LLD		35	770
42	RF-SE-22-RU	RONTEC USA Inc.	70	<LLD		<LLD		37	831
42	RF-SE-25-RU	RONTEC USA Inc.	80	<LLD		<LLD		31	717
42	RF-SE-30-RU	RONTEC USA Inc.	90	<LLD		<LLD		52	937
42	RF-SE-57-RU	RONTEC USA Inc.	71	<LLD		23		46	800

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As		Cd		Cr		Cu		Fe		Pb		Hg	
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-RU	RONTEC USA Inc.	n.d.		174		<LLD		89		889		26,090		465		<LLD	
43	RF-SE-24-RU	RONTEC USA Inc.	n.d.		122		<LLD		69		686		25,855		488		3	
43	RF-SE-32-RU	RONTEC USA Inc.	n.d.		167		<LLD		80		862		28,931		435		<LLD	
43	RF-SE-43-RU	RONTEC USA Inc.	n.d.		123		<LLD		51		744		26,941		494		<LLD	
43	RF-SE-59-RU	RONTEC USA Inc.	n.d.		140		<LLD		37		747		29,784		521		<LLD	
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-RU	RONTEC USA Inc.	n.d.		163		<LLD		56		945		23,076		458		1	
44	RF-SE-26-RU	RONTEC USA Inc.	n.d.		218		<LLD		101		1,847		25,154		783		<LLD	
44	RF-SE-39-RU	RONTEC USA Inc.	n.d.		304		<LLD		56		1,520		34,504		583		<LLD	
44	RF-SE-44-RU	RONTEC USA Inc.	n.d.		180		<LLD		46		1,060		27,517		505		<LLD	
44	RF-SE-56-RU	RONTEC USA Inc.	n.d.		135		<LLD		409		976		40,100		629		3	
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-RU	RONTEC USA Inc.	n.d.		227		<LLD		25		1,163		23,502		680		2	
45	RF-SE-14-RU	RONTEC USA Inc.	n.d.		216		<LLD		32		1,183		23,637		739		2	
45	RF-SE-19-RU	RONTEC USA Inc.	n.d.		265		<LLD		47		1,205		27,027		704		<LLD	
45	RF-SE-34-RU	RONTEC USA Inc.	n.d.		246		<LLD		29		1,398		28,603		757		<LLD	
45	RF-SE-52-RU	RONTEC USA Inc.	n.d.		197		<LLD		34		1,296		26,306		832		2	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
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-RU	RONTEC USA Inc.	110		<LLD		<LLD		41		1,034	
43	RF-SE-24-RU	RONTEC USA Inc.	68		<LLD		<LLD		41		1,170	
43	RF-SE-32-RU	RONTEC USA Inc.	171		<LLD		18		35		1,123	
43	RF-SE-43-RU	RONTEC USA Inc.	82		<LLD		<LLD		54		981	
43	RF-SE-59-RU	RONTEC USA Inc.	67		<LLD		39		51		1,067	
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-RU	RONTEC USA Inc.	49		<LLD		<LLD		21		1,179	
44	RF-SE-26-RU	RONTEC USA Inc.	158		<LLD		128		67		1,997	
44	RF-SE-39-RU	RONTEC USA Inc.	116		<LLD		<LLD		43		1,810	
44	RF-SE-44-RU	RONTEC USA Inc.	72		<LLD		<LLD		51		1,945	
44	RF-SE-56-RU	RONTEC USA Inc.	100		<LLD		<LLD		95		1,738	
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-RU	RONTEC USA Inc.	61		<LLD		<LLD		35		1,518	
45	RF-SE-14-RU	RONTEC USA Inc.	49		<LLD		<LLD		35		1,823	
45	RF-SE-19-RU	RONTEC USA Inc.	62		<LLD		40		40		1,917	
45	RF-SE-34-RU	RONTEC USA Inc.	77		<LLD		<LLD		44		2,250	
45	RF-SE-52-RU	RONTEC USA Inc.	71		<LLD		<LLD		55		1,953	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As		Cd		Cr	Cu	Fe	Pb	Hg	
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-RU	RONTEC USA Inc.	n.d.		47		<LLD		137	32	28,672	74	<LLD	
46	BN-SO-14-RU	RONTEC USA Inc.	n.d.		3,318		188		806	124	28,671	69	10	
46	BN-SO-23-RU	RONTEC USA Inc.	n.d.		2,830		385		651	106	21,762	64	6	
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-RU	RONTEC USA Inc.	n.d.		54		2,656		2,479	98	26,685	5,275	<LLD	
47	BN-SO-12-RU	RONTEC USA Inc.	n.d.		64		3,791		2,485	96	24,218	5,480	<LLD	
47	BN-SO-24-RU	RONTEC USA Inc.	n.d.		54		2,962		2,302	93	21,118	4,805	<LLD	
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-RU	RONTEC USA Inc.	n.d.		19		<LLD		165	141	47,351	4	20	
48	SB-SO-20-RU	RONTEC USA Inc.	n.d.		12		21		189	123	34,051	8	29	
48	SB-SO-31-RU	RONTEC USA Inc.	n.d.		11		<LLD		118	83	26,680	6	19	
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-RU	RONTEC USA Inc.	n.d.		14		<LLD		532	124	45,006	11	40	
49	SB-SO-36-RU	RONTEC USA Inc.	n.d.		14		<LLD		187	142	44,131	17	45	
49	SB-SO-56-RU	RONTEC USA Inc.	n.d.		10		<LLD		115	108	31,211	1	35	
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-RU	RONTEC USA Inc.	n.d.		15		1,666		2,868	102	45,051	21	11	
50	SB-SO-34-RU	RONTEC USA Inc.	n.d.		24		2,357		2,945	122	43,034	15	85	
50	SB-SO-49-RU	RONTEC USA Inc.	n.d.		15		3,301		2,767	100	37,444	15	215	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V	Zn	
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-RU	RONTEC USA Inc.	63		<LLD		<LLD		61	112	
46	BN-SO-14-RU	RONTEC USA Inc.	2,924		153		<LLD		127	4,272	
46	BN-SO-23-RU	RONTEC USA Inc.	2,986		117		109		117	4,229	
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-RU	RONTEC USA Inc.	1,399		285		106		275	96	
47	BN-SO-12-RU	RONTEC USA Inc.	1,594		297		203		291	86	
47	BN-SO-24-RU	RONTEC USA Inc.	1,464		267		116		234	83	
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-RU	RONTEC USA Inc.	2,958		34		<LLD		154	3,871	
48	SB-SO-20-RU	RONTEC USA Inc.	3,188		25		<LLD		115	4,241	
48	SB-SO-31-RU	RONTEC USA Inc.	1,984		20		<LLD		73	2,629	
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-RU	RONTEC USA Inc.	195		189		<LLD		406	3,793	
49	SB-SO-36-RU	RONTEC USA Inc.	202		183		<LLD		373	4,730	
49	SB-SO-56-RU	RONTEC USA Inc.	156		124		<LLD		304	3,965	
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-RU	RONTEC USA Inc.	2,524		457		<LLD		86	103	
50	SB-SO-34-RU	RONTEC USA Inc.	3,632		432		<LLD		43	109	
50	SB-SO-49-RU	RONTEC USA Inc.	3,447		397		<LLD		88	108	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As		Cd		Cr		Cu		Fe		Pb		Hg	
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-RU	RONTEC USA Inc.	n.d.		48		<LLD		631		4,546		29,751		2,028		<LLD	
51	WS-SO-11-RU	RONTEC USA Inc.	n.d.		47		<LLD		632		5,153		24,951		2,067		<LLD	
51	WS-SO-25-RU	RONTEC USA Inc.	n.d.		68		<LLD		668		5,506		28,037		2,511		<LLD	
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-RU	RONTEC USA Inc.	n.d.		66		<LLD		103		89		29,399		2,789		<LLD	
52	WS-SO-20-RU	RONTEC USA Inc.	n.d.		2,876		927		35		966		33,229		39,286		<LLD	
52	WS-SO-23-RU	RONTEC USA Inc.	n.d.		69		<LLD		66		89		27,609		2,725		<LLD	
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-RU	RONTEC USA Inc.	n.d.		10		1,282		34		8,590		22,713		260		<LLD	
53	AS-SO-05-RU	RONTEC USA Inc.	n.d.		10		1,202		47		7,844		22,911		288		<LLD	
53	AS-SO-08-RU	RONTEC USA Inc.	n.d.		6		672		23		5,491		20,294		192		<LLD	
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-RU	RONTEC USA Inc.	n.d.		41		<LLD		581		124		34,151		139		52	
54	LV-SO-40-RU	RONTEC USA Inc.	n.d.		54		<LLD		680		121		31,441		131		47	
54	LV-SO-49-RU	RONTEC USA Inc.	n.d.		52		<LLD		763		119		37,325		159		31	
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-RU	RONTEC USA Inc.	n.d.		174		1,990		2,673		121		41,565		5,898		91	
55	LV-SO-34-RU	RONTEC USA Inc.	n.d.		133		2,795		2,927		103		31,759		5,331		92	
55	LV-SO-37-RU	RONTEC USA Inc.	n.d.		107		2,019		2,633		94		30,341		4,879		90	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
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-RU	RONTEC USA Inc.	211		<LLD		<LLD		46		188	
51	WS-SO-11-RU	RONTEC USA Inc.	260		<LLD		188		37		206	
51	WS-SO-25-RU	RONTEC USA Inc.	281		<LLD		77		34		252	
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-RU	RONTEC USA Inc.	327		320		<LLD		279		2,392	
52	WS-SO-20-RU	RONTEC USA Inc.	21		11		30		12		4,425	
52	WS-SO-23-RU	RONTEC USA Inc.	296		303		<LLD		280		2,229	
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-RU	RONTEC USA Inc.	206		655		415		59		454	
53	AS-SO-05-RU	RONTEC USA Inc.	207		597		409		42		447	
53	AS-SO-08-RU	RONTEC USA Inc.	168		417		75		45		334	
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-RU	RONTEC USA Inc.	2,124		120		53		105		4,380	
54	LV-SO-40-RU	RONTEC USA Inc.	2,344		129		<LLD		126		4,819	
54	LV-SO-49-RU	RONTEC USA Inc.	1,100		137		<LLD		130		2,171	
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-RU	RONTEC USA Inc.	2,235		293		<LLD		277		85	
55	LV-SO-34-RU	RONTEC USA Inc.	2,136		270		<LLD		294		67	
55	LV-SO-37-RU	RONTEC USA Inc.	1,496		257		<LLD		287		64	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As	Cd		Cr		Cu		Fe		Pb		Hg	
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-RU	RONTEC USA Inc.	n.d.		109	<LLD		24		92		23,426		167		33	
56	CN-SO-06-RU	RONTEC USA Inc.	n.d.		106	17		11		92		16,552		173		31	
56	CN-SO-07-RU	RONTEC USA Inc.	n.d.		105	<LLD		10		85		19,147		173		30	
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-RU	RONTEC USA Inc.	n.d.		10	238		27		145		24,617		55		159	
57	CN-SO-05-RU	RONTEC USA Inc.	n.d.		11	415		31		204		25,341		38		226	
57	CN-SO-09-RU	RONTEC USA Inc.	n.d.		11	179		13		158		35,028		36		210	
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-RU	RONTEC USA Inc.	n.d.		96	<LLD		527		34		33,642		2,064		340	
58	LV-SE-13-RU	RONTEC USA Inc.	n.d.		52	<LLD		542		34		33,065		2,358		395	
58	LV-SE-41-RU	RONTEC USA Inc.	n.d.		40	<LLD		715		37		27,720		2,620		401	
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-RU	RONTEC USA Inc.	n.d.		33	197		1,193		53		37,411		48		<LLD	
59	LV-SE-20-RU	RONTEC USA Inc.	n.d.		27	111		1,059		52		35,911		28		<LLD	
59	LV-SE-43-RU	RONTEC USA Inc.	n.d.		14	<LLD		1,210		45		26,671		58		<LLD	
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-RU	RONTEC USA Inc.	n.d.		47	779		48		2,108		28,157		18		395	
60	LV-SE-17-RU	RONTEC USA Inc.	n.d.		51	855		118		2,373		34,264		26		411	
60	LV-SE-51-RU	RONTEC USA Inc.	n.d.		27	<LLD		61		1,656		27,276		19		37	
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-RU	RONTEC USA Inc.	n.d.		74	66		52		2,515		31,161		2,079		<LLD	
61	TL-SE-09-RU	RONTEC USA Inc.	n.d.		25	<LLD		16		2,351		13,275		594		309	
61	TL-SE-13-RU	RONTEC USA Inc.	n.d.		30	<LLD		22		2,322		18,679		652		359	

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag		V		Zn
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-RU	RONTEC USA Inc.	92	40	<LLD		51		79
56	CN-SO-06-RU	RONTEC USA Inc.	100	38	47		35		76
56	CN-SO-07-RU	RONTEC USA Inc.	86	39	<LLD		33		75
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-RU	RONTEC USA Inc.	311	160	<LLD		113		2,121
57	CN-SO-05-RU	RONTEC USA Inc.	344	180	<LLD		133		2,312
57	CN-SO-09-RU	RONTEC USA Inc.	328	174	<LLD		151		2,237
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-RU	RONTEC USA Inc.	389	180	<LLD		479		83
58	LV-SE-13-RU	RONTEC USA Inc.	369	189	<LLD		483		79
58	LV-SE-41-RU	RONTEC USA Inc.	338	176	<LLD		597		70
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-RU	RONTEC USA Inc.	471	496	90		484		2,112
59	LV-SE-20-RU	RONTEC USA Inc.	537	466	<LLD		441		2,481
59	LV-SE-43-RU	RONTEC USA Inc.	463	414	<LLD		489		2,256
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-RU	RONTEC USA Inc.	158	72	204		129		61
60	LV-SE-17-RU	RONTEC USA Inc.	190	76	365		166		77
60	LV-SE-51-RU	RONTEC USA Inc.	136	68	494		161		67
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-RU	RONTEC USA Inc.	37	43	<LLD		44		81
61	TL-SE-09-RU	RONTEC USA Inc.	13	59	<LLD		6		40
61	TL-SE-13-RU	RONTEC USA Inc.	20	60	<LLD		18		50

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb		As	Cd	Cr	Cu	Fe	Pb
62	TL-SE-06-XX	Reference Laboratory	1.2	U	86	350	34	2000	22,000	1,700
62	TL-SE-17-XX	Reference Laboratory	1.2	U	85	340	33	2100	21,000	1,700
62	TL-SE-28-XX	Reference Laboratory	1.2	U	89	360	34	2100	22,000	1,700
62	TL-SE-06-RU	RONTEC USA Inc.	n.d.		19	<LLD	60	2,613	66,310	4
62	TL-SE-17-RU	RONTEC USA Inc.	n.d.		46	<LLD	42	939	36,870	1,303
62	TL-SE-28-RU	RONTEC USA Inc.	n.d.		80	<LLD	46	2,140	37,399	2,104
63	TL-SE-07-XX	Reference Laboratory	30		11	48	66	2200	37,000	13
63	TL-SE-21-XX	Reference Laboratory	33		13	51	73	2300	44,000	15
63	TL-SE-30-XX	Reference Laboratory	31		11	47	64	2200	36,000	14
63	TL-SE-07-RU	RONTEC USA Inc.	n.d.		23	23	62	3,699	72,976	<LLD
63	TL-SE-21-RU	RONTEC USA Inc.	n.d.		17	<LLD	50	2,435	62,156	9
63	TL-SE-30-RU	RONTEC USA Inc.	n.d.		17	<LLD	63	2,534	69,874	4
64	TL-SE-02-XX	Reference Laboratory	77		15	160	64	3,100	32,000	12
64	TL-SE-08-XX	Reference Laboratory	66		10	180	74	3,200	45,000	11
64	TL-SE-16-XX	Reference Laboratory	73		15	170	69	3,100	38,000	13
64	TL-SE-02-RU	RONTEC USA Inc.	n.d.		21	<LLD	44	3,085	65,681	7
64	TL-SE-08-RU	RONTEC USA Inc.	n.d.		24	<LLD	90	4,183	75,135	1
64	TL-SE-16-RU	RONTEC USA Inc.	n.d.		12	16	39	1,689	40,259	1
65	RF-SE-01-XX	Reference Laboratory	12		230	40	280	63	14,000	22
65	RF-SE-09-XX	Reference Laboratory	10		260	45	310	71	16,000	26
65	RF-SE-11-XX	Reference Laboratory	11		240	43	300	72	15,000	25
65	RF-SE-17-XX	Reference Laboratory	11		250	43	300	67	15,000	26
65	RF-SE-29-XX	Reference Laboratory	13		280	49	330	75	17,000	26
65	RF-SE-37-XX	Reference Laboratory	11		260	45	320	72	16,000	27
65	RF-SE-50-XX	Reference Laboratory	8.9		230	40	280	65	14,000	23
65	RF-SE-01-RU	RONTEC USA Inc.	n.d.		304	<LLD	335	77	1,573	11
65	RF-SE-09-RU	RONTEC USA Inc.	n.d.		276	<LLD	400	78	19,246	36
65	RF-SE-11-RU	RONTEC USA Inc.	n.d.		305	<LLD	370	72	16,797	<LLD
65	RF-SE-17-RU	RONTEC USA Inc.	n.d.		274	<LLD	323	70	18,068	46
65	RF-SE-29-RU	RONTEC USA Inc.	n.d.		297	<LLD	319	63	18,387	30
65	RF-SE-37-RU	RONTEC USA Inc.	n.d.		260	<LLD	301	82	17,706	30
65	RF-SE-50-RU	RONTEC USA Inc.	n.d.		296	<LLD	333	72	23,497	36

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-RU	RONTEC USA Inc.	77	160	<LLD	73	175
62	TL-SE-17-RU	RONTEC USA Inc.	33	24	<LLD	55	99
62	TL-SE-28-RU	RONTEC USA Inc.	36	41	<LLD	59	91
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-RU	RONTEC USA Inc.	82	89	30	59	190
63	TL-SE-21-RU	RONTEC USA Inc.	75	152	85	85	159
63	TL-SE-30-RU	RONTEC USA Inc.	81	156	62	69	182
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-RU	RONTEC USA Inc.	77	47	33	97	165
64	TL-SE-08-RU	RONTEC USA Inc.	88	77	67	69	213
64	TL-SE-16-RU	RONTEC USA Inc.	46	28	101	51	102
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-RU	RONTEC USA Inc.	175	20	55	14	1,573
65	RF-SE-09-RU	RONTEC USA Inc.	113	20	<LLD	17	996
65	RF-SE-11-RU	RONTEC USA Inc.	140	20	<LLD	16	1,309
65	RF-SE-17-RU	RONTEC USA Inc.	99	18	<LLD	27	935
65	RF-SE-29-RU	RONTEC USA Inc.	161	20	<LLD	15	1,473
65	RF-SE-37-RU	RONTEC USA Inc.	115	20	<LLD	14	1,000
65	RF-SE-50-RU	RONTEC USA Inc.	164	20	231	20	1,704

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-RU	RONTEC USA Inc.	n.d.	601	<LLD	615	1,204	22,179	640	24
66	RF-SE-10-RU	RONTEC USA Inc.	n.d.	660	<LLD	736	1,347	26,903	690	31
66	RF-SE-33-RU	RONTEC USA Inc.	n.d.	656	<LLD	633	1,628	29,358	633	23
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-RU	RONTEC USA Inc.	n.d.	112	<LLD	1,246	84	23,391	32	298
67	RF-SE-41-RU	RONTEC USA Inc.	n.d.	99	<LLD	1,606	213	24,704	54	157
67	RF-SE-48-RU	RONTEC USA Inc.	n.d.	109	<LLD	1,082	85	31,425	48	288
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-RU	RONTEC USA Inc.	n.d.	1,162	<LLD	1,244	87	24,728	996	647
68	RF-SE-35-RU	RONTEC USA Inc.	n.d.	1,239	<LLD	1,261	109	34,720	955	541
68	RF-SE-54-RU	RONTEC USA Inc.	n.d.	906	<LLD	1,109	79	19,404	1,140	463
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-RU	RONTEC USA Inc.	n.d.	876	<LLD	104	87	20,301	23	<LLD
69	RF-SE-46-RU	RONTEC USA Inc.	n.d.	956	<LLD	88	78	18,922	25	<LLD
69	RF-SE-51-RU	RONTEC USA Inc.	n.d.	932	1,001	79	79	21,808	25	<LLD
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-RU	RONTEC USA Inc.	n.d.	45	<LLD	76	743	23,337	2,518	220
70	RF-SE-40-RU	RONTEC USA Inc.	n.d.	37	1,756	89	896	23,550	2,899	174
70	RF-SE-47-RU	RONTEC USA Inc.	n.d.	18	2,056	146	1,176	27,209	3,175	301

Appendix D: Analytical Data Summary, RONTEC Pico Tax and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni		Se		Ag		V		Zn	
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-RU	RONTEC USA Inc.	113		40		35		132		124	
66	RF-SE-10-RU	RONTEC USA Inc.	136		44		44		142		130	
66	RF-SE-33-RU	RONTEC USA Inc.	160		44		<LLD		128		140	
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-RU	RONTEC USA Inc.	1,037		<LLD		60		21		588	
67	RF-SE-41-RU	RONTEC USA Inc.	1,768		<LLD		26		17		731	
67	RF-SE-48-RU	RONTEC USA Inc.	1,569		<LLD		28		47		715	
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-RU	RONTEC USA Inc.	263		186		<LLD		486		130	
68	RF-SE-35-RU	RONTEC USA Inc.	277		210		63		533		159	
68	RF-SE-54-RU	RONTEC USA Inc.	324		159		254		437		104	
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-RU	RONTEC USA Inc.	770		324		<LLD		23		731	
69	RF-SE-46-RU	RONTEC USA Inc.	999		342		20		26		907	
69	RF-SE-51-RU	RONTEC USA Inc.	901		329		130		17		876	
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-RU	RONTEC USA Inc.	167		434		51		152		94	
70	RF-SE-40-RU	RONTEC USA Inc.	179		490		378		156		126	
70	RF-SE-47-RU	RONTEC USA Inc.	338		509		237		203		161	

Notes:

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

<LLC Reported by RONTEC as less than the instrument's statistical lower limit of detection (LLD).

J Estimated concentration.

J+ Concentration is considered estimated and biased high.

J- Concentration is considered estimated and biased low.

n.d. Not detectable by instrument.

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 Arsenic

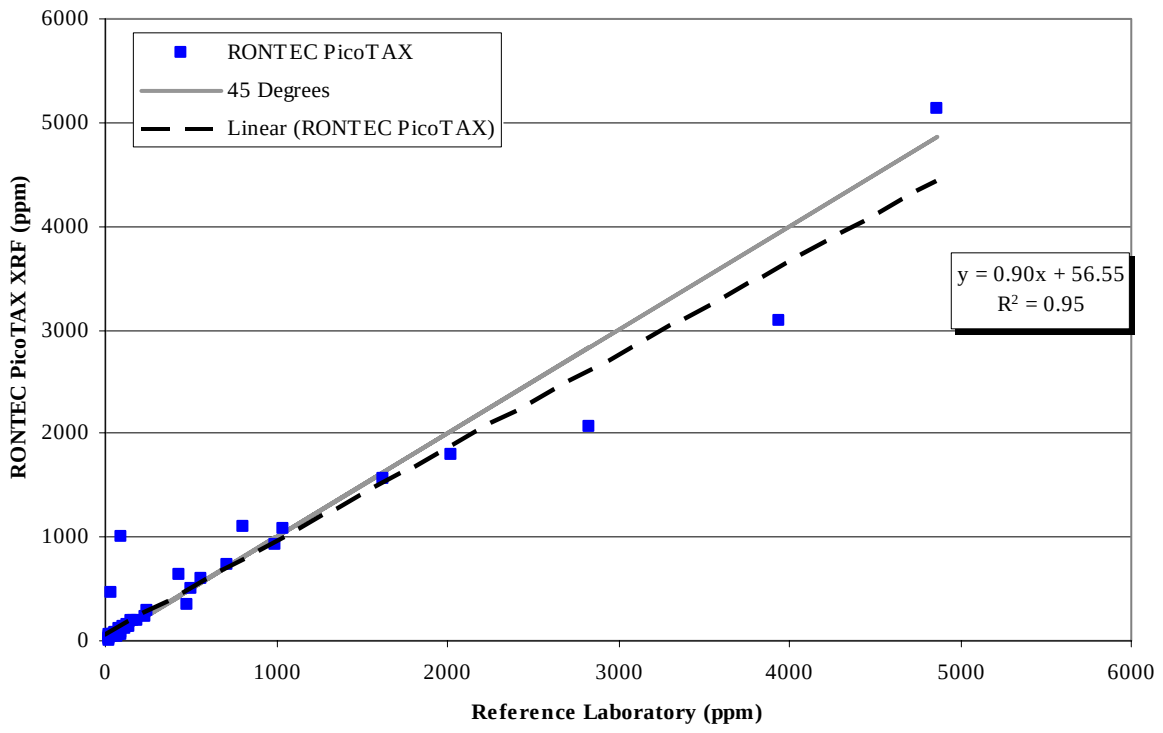


Figure E-2: Linear Correlation Plot for Cadmium

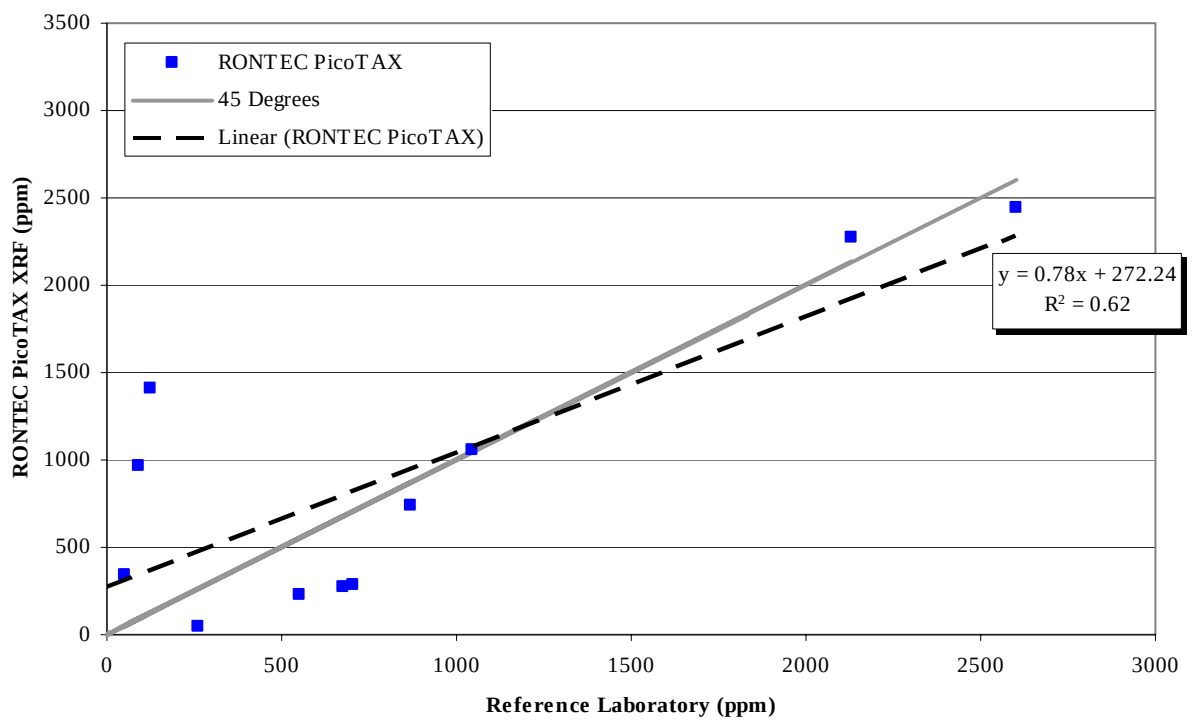


Figure E-3: Linear Correlation Plot for Chromium

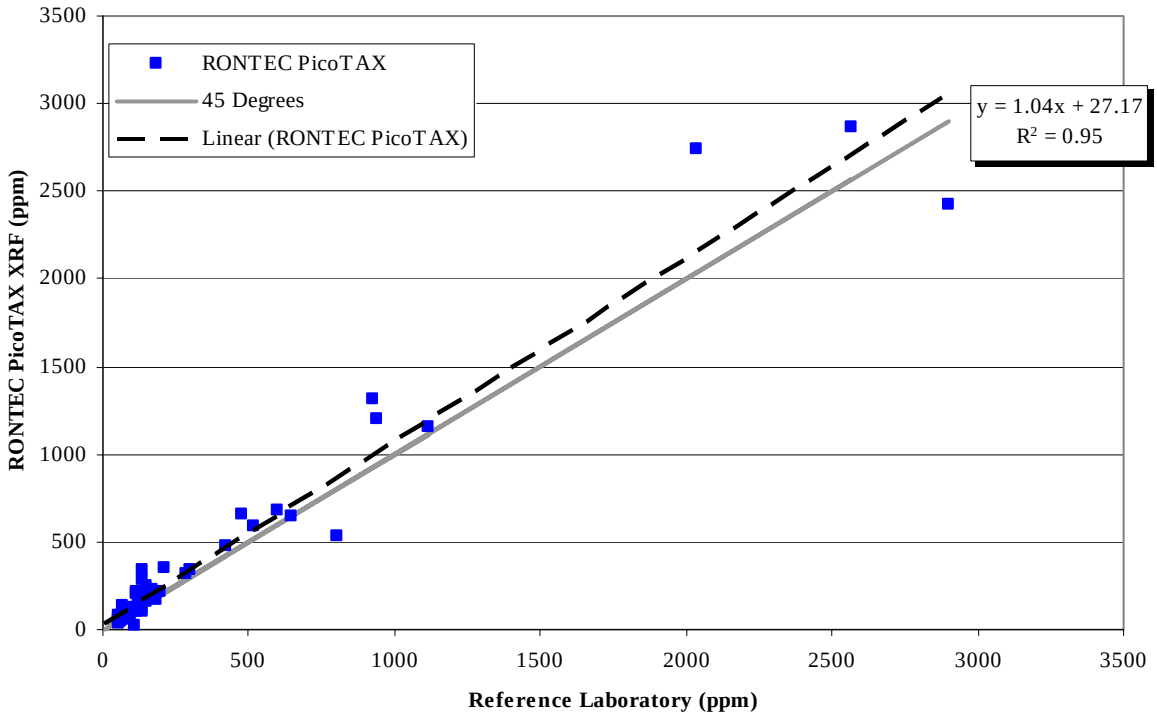


Figure E-4: Linear Correlation Plot for Copper

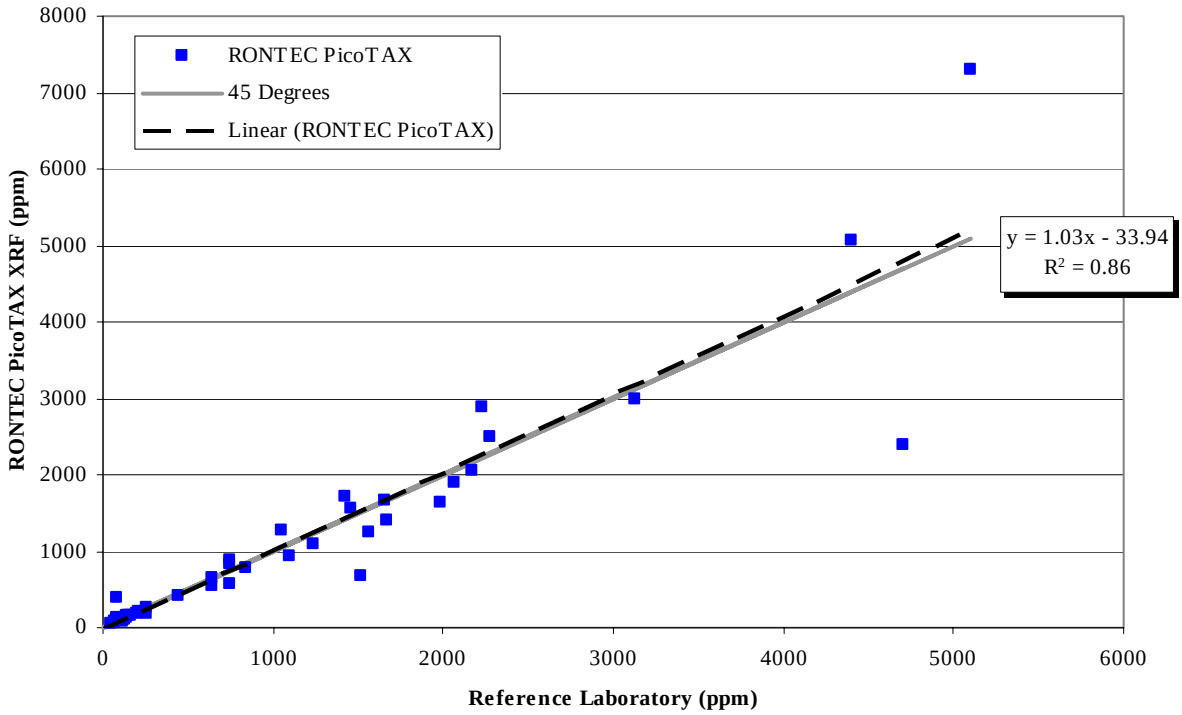


Figure E-5: Linear Correlation Plot for Iron

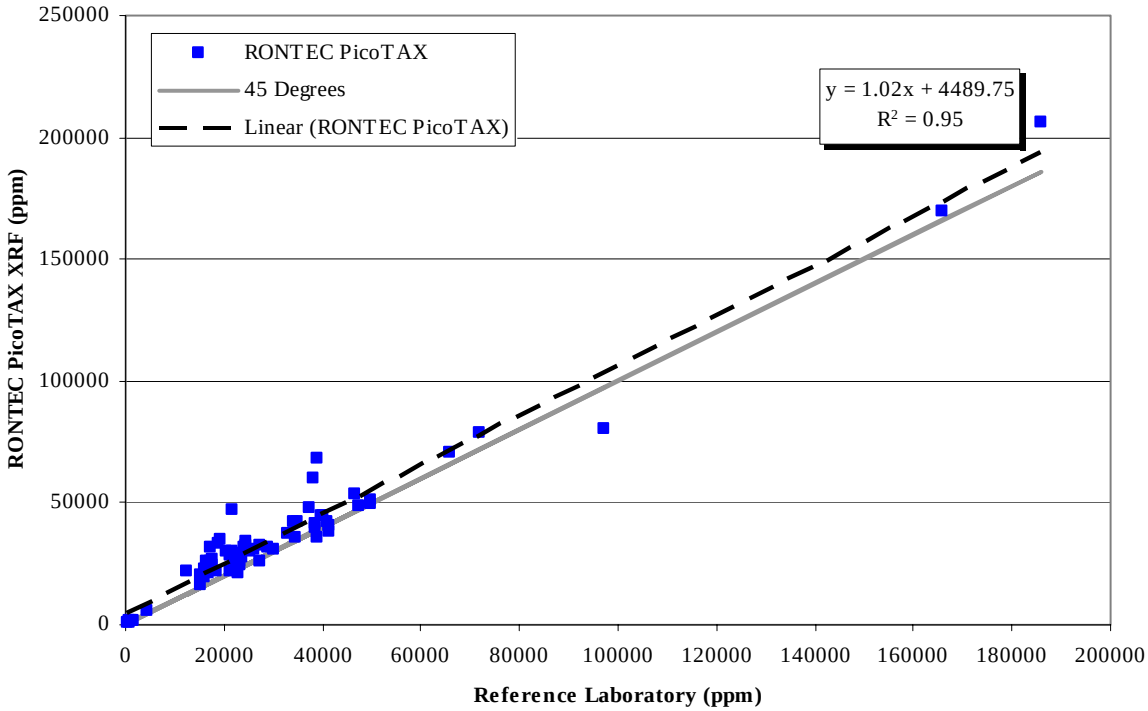


Figure E-6: Linear Correlation Plot for Lead

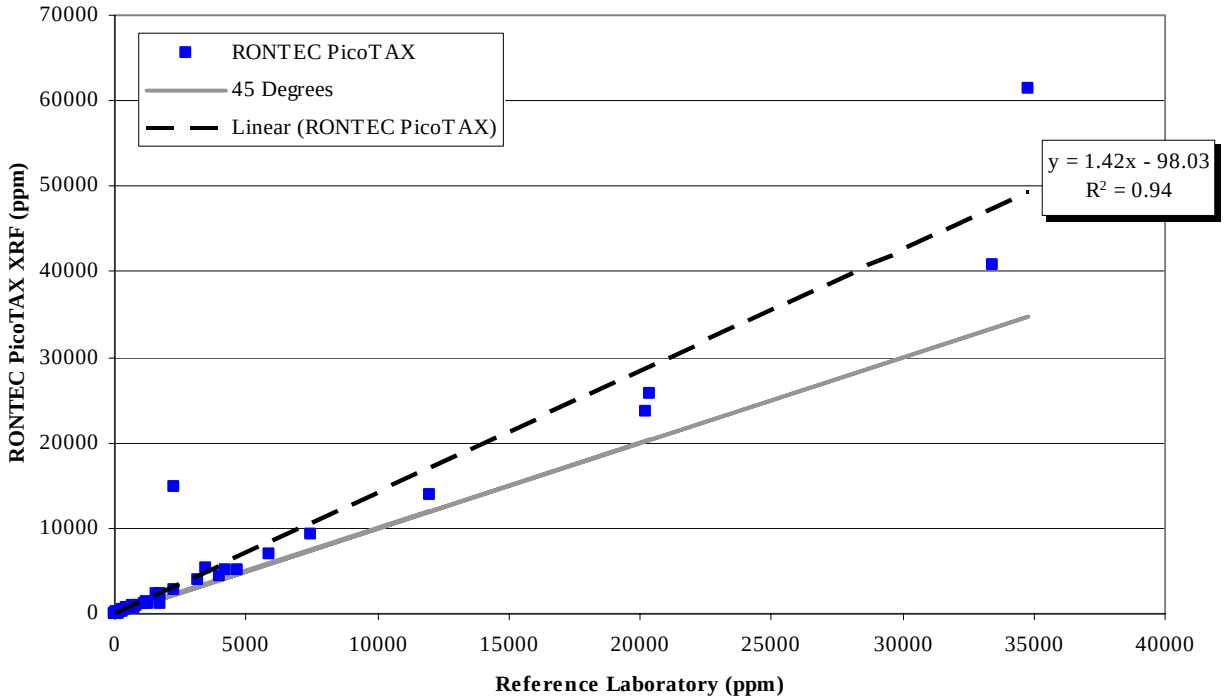


Figure E-7: Linear Correlation Plot for Mercury

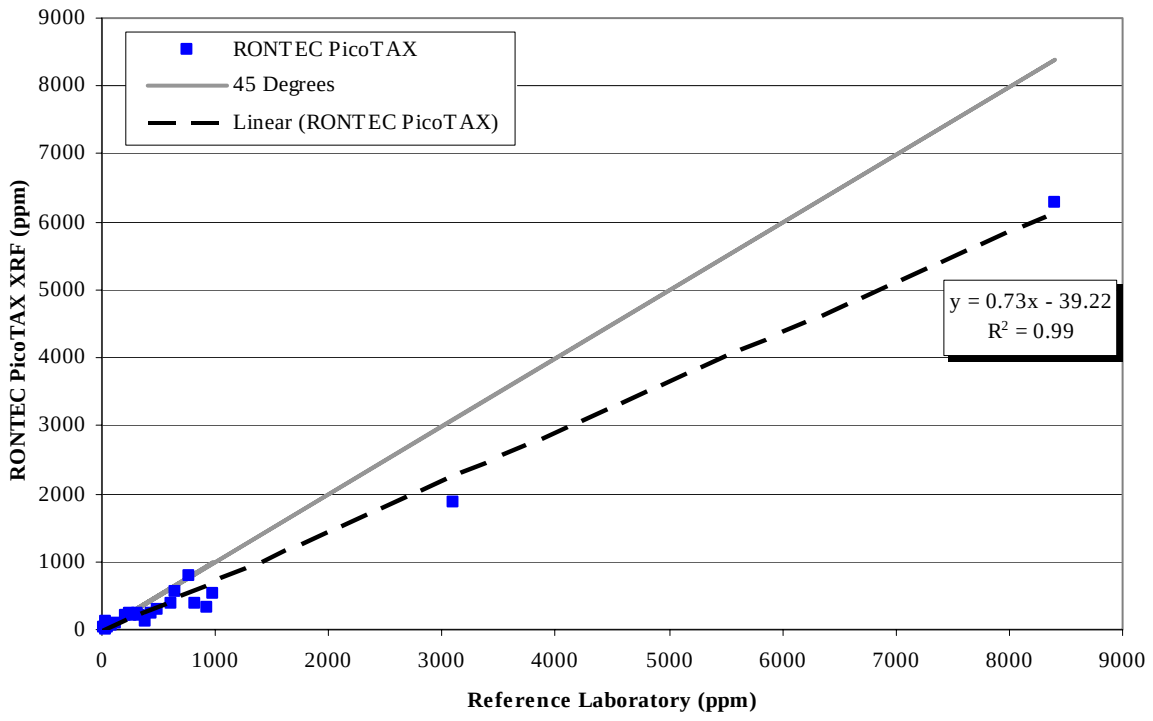


Figure E-8: Linear Correlation Plot for Nickel

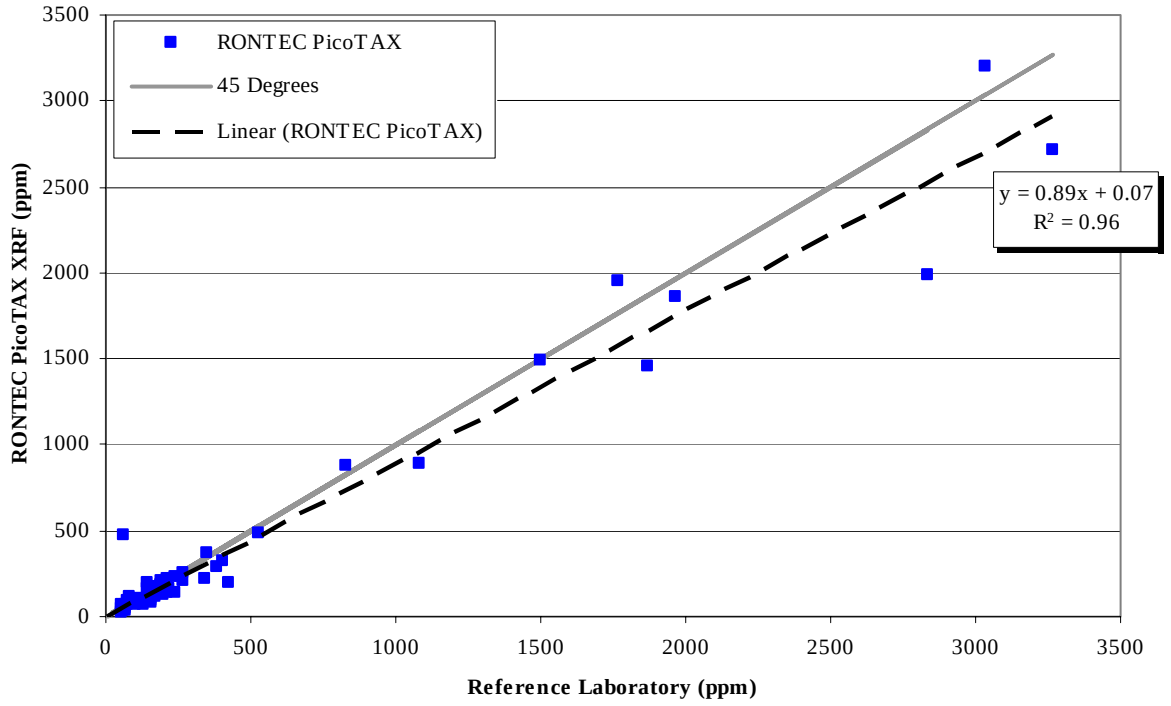


Figure E-9: Linear Correlation Plot for Selenium

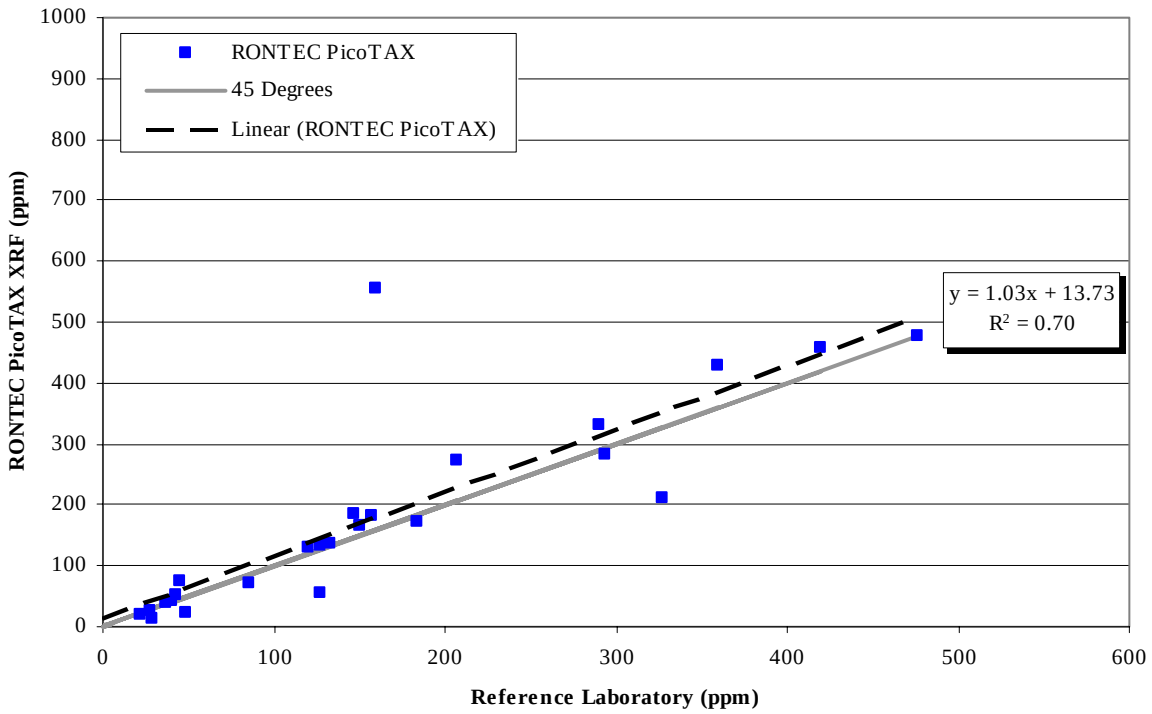


Figure E-10: Linear Correlation Plot for Silver

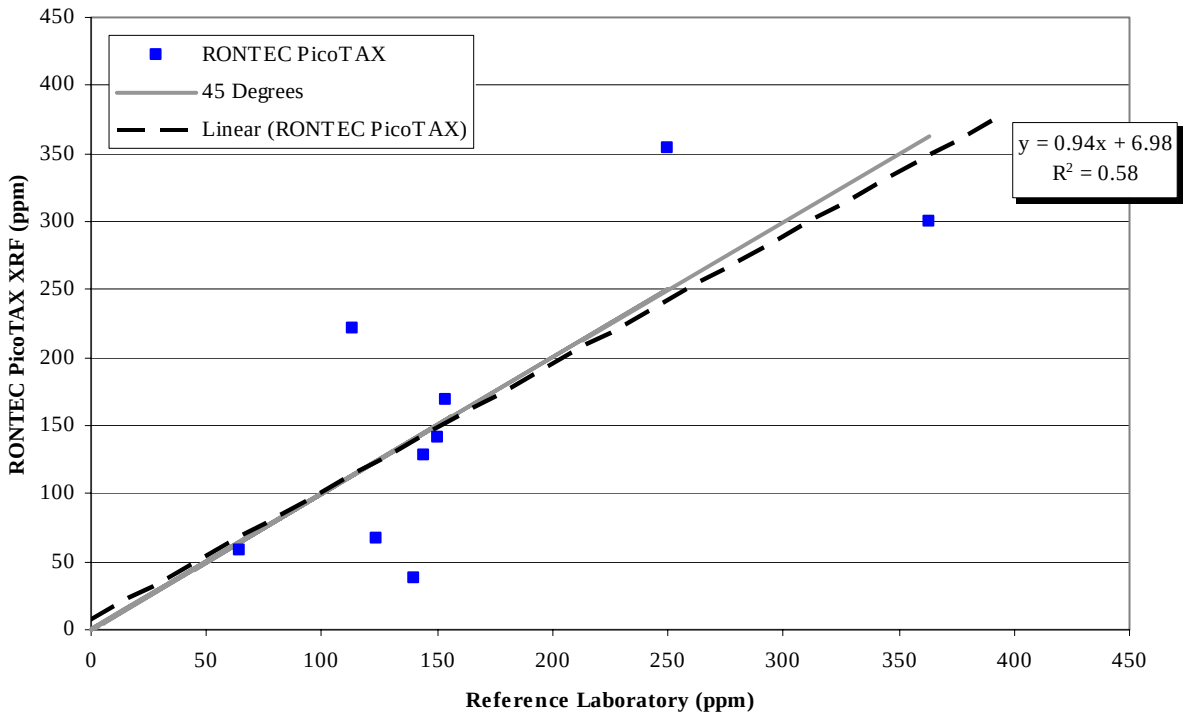


Figure E-11: Linear Correlation Plot for Vanadium

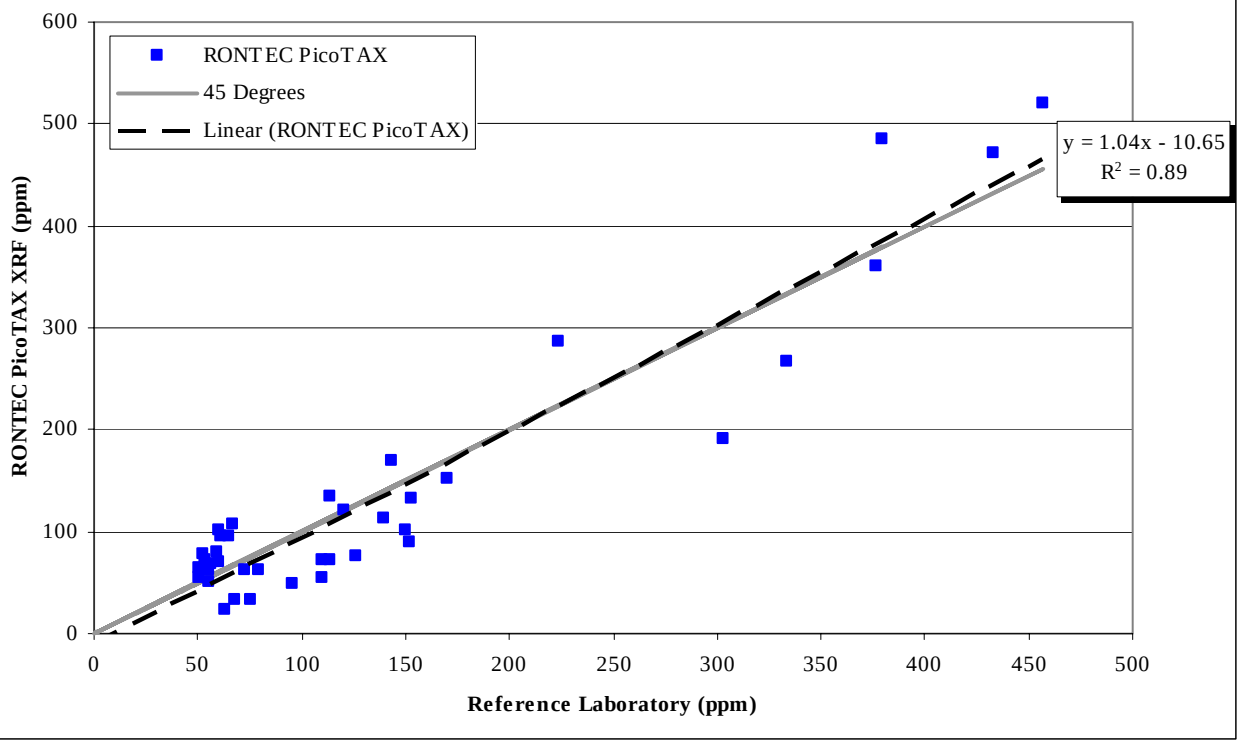
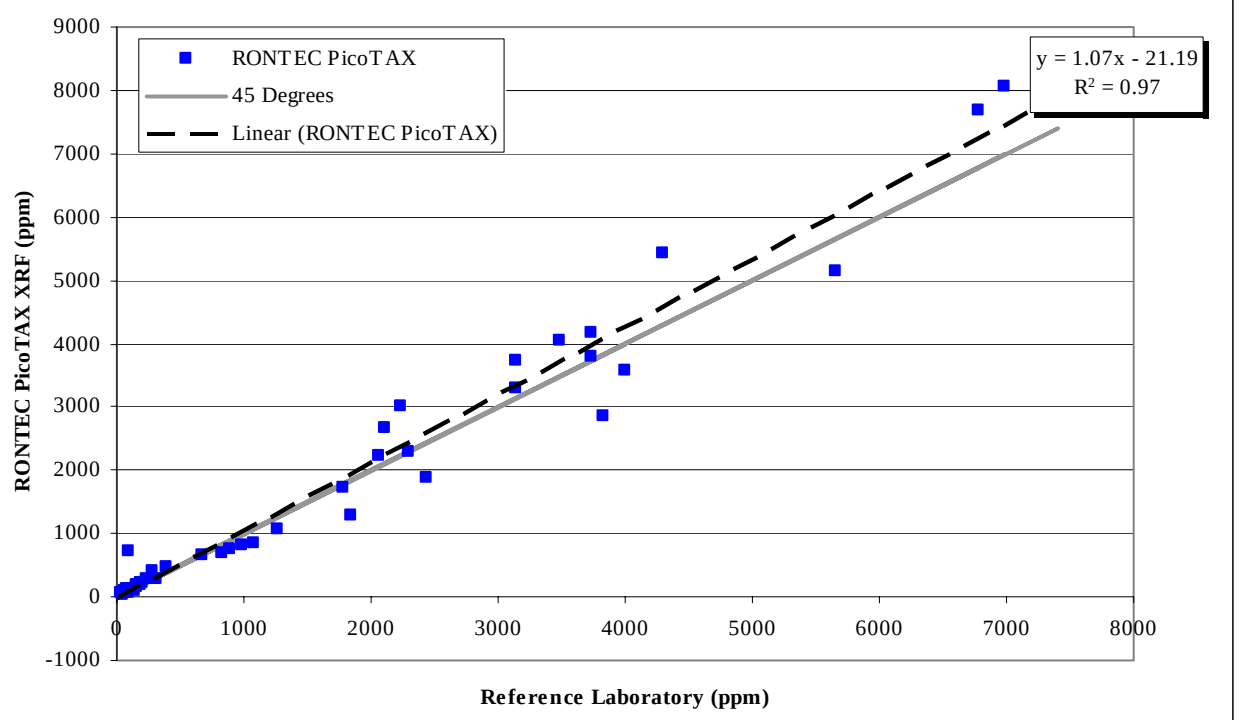
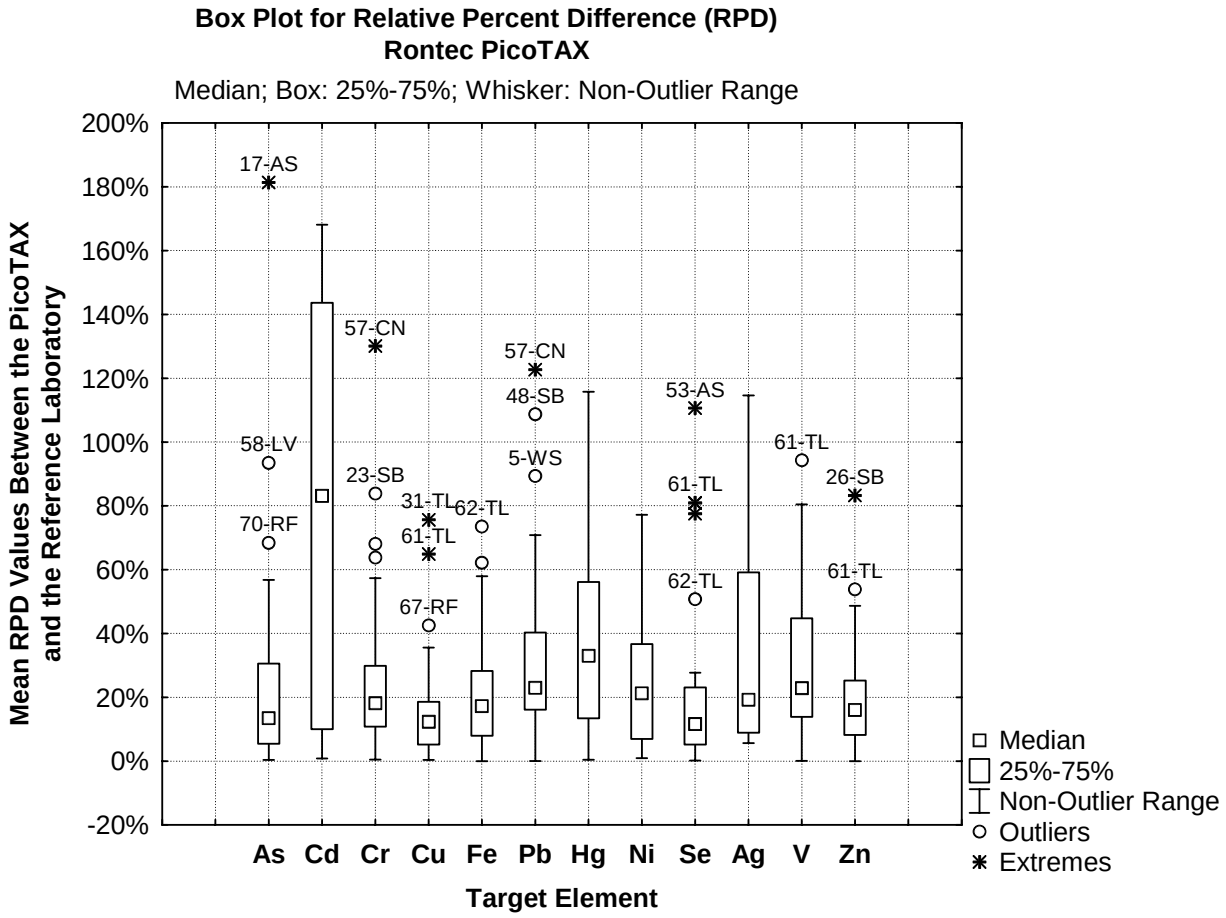


Figure E-12: Linear Correlation Plot for Zinc





Notes:

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

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

Figure E-13. Box and Whisker Plot for Mean RPD Values Showing Outliers and Extremes for Target Elements, Rontec PicoTAX Data Set.

Table E-1. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Rontec PicoTAX

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel
			Ref Lab	ERA Spike								
Soil	Level 1	Number	0	0	15	4	28	16	5	16	7	22
		Minimum	NC	NC	2.7%	138.5%	2.5%	1.0%	13.6%	9.1%	5.9%	1.0%
		Maximum	NC	NC	181.4%	168.1%	130.1%	34.8%	48.6%	122.7%	110.4%	62.8%
		Mean	NC	NC	32.9%	155.2%	32.0%	11.6%	23.9%	43.1%	41.9%	22.7%
		Median	NC	NC	20.7%	157.0%	20.7%	10.4%	20.5%	24.9%	19.3%	16.7%
	Level 2	Number	0	0	4	6	4	8	13	4	7	5
		Minimum	NC	NC	3.1%	0.8%	0.5%	0.5%	8.7%	2.2%	4.7%	6.2%
		Maximum	NC	NC	32.6%	84.2%	29.8%	26.8%	56.5%	25.7%	73.0%	74.6%
		Mean	NC	NC	11.2%	45.7%	13.0%	11.0%	26.0%	15.1%	40.6%	26.7%
		Median	NC	NC	4.6%	49.6%	10.9%	10.0%	23.2%	16.3%	43.3%	21.6%
	Level 3	Number	0	0	4	2	2	2	13	8	2	6
		Minimum	NC	NC	5.5%	6.3%	10.8%	14.1%	3.0%	7.3%	28.7%	0.9%
		Maximum	NC	NC	12.2%	13.7%	17.9%	35.6%	25.5%	42.2%	49.1%	18.6%
		Mean	NC	NC	8.7%	10.0%	14.4%	24.9%	12.0%	20.2%	38.9%	7.5%
		Median	NC	NC	8.7%	10.0%	14.4%	24.9%	12.0%	20.4%	38.9%	5.6%
	Level 4	Number	--	--	--	--	--	--	7	5	--	--
		Minimum	--	--	--	--	--	--	0.0%	15.3%	--	--
		Maximum	--	--	--	--	--	--	18.6%	55.4%	--	--
		Mean	--	--	--	--	--	--	5.9%	28.7%	--	--
		Median	--	--	--	--	--	--	2.4%	22.9%	--	--
	All Soil	Number	0	0	23	12	34	26	38	16	16	33
		Minimum	NC	NC	2.7%	0.8%	0.5%	0.5%	0.0%	11.2%	4.7%	0.9%
		Maximum	NC	NC	181.4%	168.1%	130.1%	35.6%	56.5%	70.9%	110.4%	74.6%
		Mean	NC	NC	25.0%	76.2%	28.8%	12.4%	17.2%	33.7%	40.9%	20.6%
		Median	NC	NC	14.3%	83.2%	17.9%	11.5%	14.9%	30.0%	33.0%	12.2%

Table E-1. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Rontec PicoTAX (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	22	4	0	13	20
		Minimum	1.0%	5.2%	NC	0.1%	0.0%
		Maximum	62.8%	80.8%	NC	68.7%	83.3%
		Mean	22.7%	42.3%	NC	30.0%	25.4%
		Median	16.7%	41.5%	NC	29.2%	21.0%
	Level 2	Number	5	5	0	4	6
		Minimum	6.2%	1.3%	NC	0.2%	3.4%
		Maximum	74.6%	110.7%	NC	20.7%	23.6%
		Mean	26.7%	27.1%	NC	14.0%	12.0%
		Median	21.6%	6.9%	NC	17.5%	11.3%
	Level 3	Number	6	4	4	4	9
		Minimum	0.9%	3.7%	5.6%	4.3%	0.9%
		Maximum	18.6%	27.7%	19.3%	24.7%	23.4%
		Mean	7.5%	13.4%	11.4%	14.8%	10.5%
		Median	5.6%	11.1%	10.4%	15.2%	10.9%
	Level 4	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Soil	Number	33	13	4	21	35
		Minimum	0.9%	1.3%	5.6%	0.1%	0.0%
		Maximum	74.6%	110.7%	19.3%	68.7%	83.3%
		Mean	20.6%	27.5%	11.4%	24.0%	19.3%
		Median	12.2%	6.9%	10.4%	20.7%	16.0%

Table E-1. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Rontec PicoTAX (Continued)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
			Ref Lab	ERA Spike							
Sediment	Level 1	Number	0	0	17	0	21	8	3	4	2
		Minimum	NC	NC	1.2%	NC	3.0%	0.7%	35.8%	0.1%	7.4%
		Maximum	NC	NC	93.5%	NC	63.8%	42.5%	62.2%	18.8%	47.7%
		Mean	NC	NC	24.0%	NC	19.8%	11.4%	45.2%	10.2%	27.5%
		Median	NC	NC	12.9%	NC	20.3%	5.6%	37.6%	10.9%	27.5%
	Level 2	Number	0	0	4	0	3	4	19	4	4
		Minimum	NC	NC	0.4%	NC	11.6%	6.2%	2.7%	0.1%	0.4%
		Maximum	NC	NC	38.4%	NC	31.8%	18.7%	73.5%	18.8%	115.8%
		Mean	NC	NC	14.0%	NC	18.9%	14.8%	28.6%	10.2%	49.4%
		Median	NC	NC	8.6%	NC	13.4%	17.2%	27.5%	10.9%	40.7%
	Level 3	Number	0	0	2	0	3	10	4	3	2
		Minimum	NC	NC	7.9%	NC	3.6%	0.4%	6.0%	21.8%	15.1%
		Maximum	NC	NC	30.6%	NC	34.7%	75.7%	54.7%	39.9%	48.3%
		Mean	NC	NC	19.2%	NC	21.1%	24.5%	30.7%	33.8%	31.7%
		Median	NC	NC	19.2%	NC	25.0%	18.5%	31.1%	39.7%	31.7%
	Level 4	Number	--	--	--	--	--	--	6	--	--
		Minimum	--	--	--	--	--	--	1.5%	--	--
		Maximum	--	--	--	--	--	--	10.5%	--	--
		Mean	--	--	--	--	--	--	6.9%	--	--
		Median	--	--	--	--	--	--	8.4%	--	--
	All Sediment	Number	0	0	23	0	27	22	32	23	8
		Minimum	NC	NC	0.4%	NC	3.0%	0.4%	1.5%	0.1%	0.4%
		Maximum	NC	NC	93.5%	NC	63.8%	75.7%	73.5%	70.9%	115.8%
		Mean	NC	NC	21.9%	NC	19.9%	18.0%	26.3%	29.7%	39.5%
		Median	NC	NC	12.9%	NC	20.3%	15.4%	21.6%	25.5%	37.8%

Table E-1. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Rontec PicoTAX (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Sediment	Level 1	Number	18	5	1	6	19
		Minimum	4.0%	4.8%	8.5%	15.0%	1.0%
		Maximum	77.2%	50.7%	8.5%	94.3%	53.9%
		Mean	35.5%	20.5%	8.5%	49.5%	19.2%
		Median	34.9%	17.4%	8.5%	45.0%	11.6%
	Level 2	Number	6	4	2	8	5
		Minimum	5.1%	4.4%	59.1%	11.4%	1.3%
		Maximum	53.8%	81.0%	114.6%	66.0%	25.5%
		Mean	27.7%	30.8%	86.9%	37.7%	16.3%
		Median	29.4%	18.9%	86.9%	43.8%	19.4%
	Level 3	Number	4	3	2	3	4
		Minimum	5.7%	0.2%	34.5%	8.4%	0.7%
		Maximum	24.6%	13.4%	64.8%	24.3%	35.7%
		Mean	14.4%	7.5%	49.7%	15.2%	16.1%
		Median	13.7%	8.8%	49.7%	12.9%	14.0%
	Level 4	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Sediment	Number	28	12	5	17	28
		Minimum	4.0%	0.2%	8.5%	8.4%	0.7%
		Maximum	77.2%	81.0%	114.6%	94.3%	53.9%
		Mean	30.8%	20.7%	56.3%	37.9%	18.2%
		Median	29.4%	14.1%	59.1%	24.3%	16.2%

Table E-1. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Rontec PicoTAX (Continued)

Matrix	Conc Range	Statistic	Antimony Ref Lab	Antimony ERA Spike	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
All Samples	Rontec PicoTAX	Number	0	0	46	12	61	48	70	56	24
		Minimum	NC	NC	0.4%	0.8%	0.5%	0.4%	0.0%	0.1%	0.4%
		Maximum	NC	NC	181.4%	168.1%	130.1%	75.7%	73.5%	122.7%	115.8%
		Mean	NC	NC	23.4%	76.2%	24.8%	15.0%	21.4%	31.0%	40.5%
		Median	NC	NC	13.4%	83.2%	18.1%	12.3%	17.3%	22.9%	33.0%
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 Differences Versus Reference Laboratory Data Calculated for the Rontec PicoTAX (Continued)

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
All Samples	Rontec PicoTAX	Number	61	25	9	38	63
		Minimum	0.9%	0.2%	5.6%	0.1%	0.0%
		Maximum	77.2%	110.7%	114.6%	94.3%	83.3%
		Mean	25.3%	24.2%	36.4%	30.2%	18.8%
		Median	21.2%	11.6%	19.3%	22.9%	16.0%
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%

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 Resouce Associstes, Inc.
NC	Not calculated due to lack of XRF data.
Number	Number of demonstration samples evaluated.
Ref Lab	Reference laboratory (Shealy Environmental Services, Inc.).
RPD	Relative percent difference.
SRF	X-ray fluorescence.

Table E-2. Evaluation of Precision - Relative Standard Deviations Calculated for the Rontec PicoTAX

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
Soil	Low	Number	0	15	4	28	16	5	16	7
		Minimum	NC	2.0%	17.4%	11.8%	0.1%	10.6%	2.2%	1.1%
		Maximum	NC	71.7%	31.0%	89.0%	25.9%	29.4%	78.7%	105.2%
		Mean	NC	18.2%	25.9%	34.8%	12.1%	19.9%	24.3%	42.7%
		Median	NC	13.8%	27.6%	26.3%	11.9%	19.2%	20.9%	25.2%
	Medium	Number	0	4	6	4	8	13	4	7
		Minimum	NC	6.5%	20.1%	3.3%	8.9%	4.4%	11.7%	17.7%
		Maximum	NC	17.3%	48.5%	15.1%	28.2%	20.5%	25.5%	88.2%
		Mean	NC	13.5%	33.4%	9.4%	17.0%	13.2%	15.6%	35.6%
		Median	NC	15.1%	31.5%	9.6%	16.7%	13.3%	12.6%	26.0%
	High	Number	0	4	2	2	2	13	8	2
		Minimum	NC	6.1%	18.7%	3.1%	9.6%	8.5%	1.7%	22.9%
		Maximum	NC	19.6%	33.6%	4.3%	22.1%	29.1%	23.0%	44.7%
		Mean	NC	12.8%	26.2%	3.7%	15.9%	17.3%	11.7%	33.8%
		Median	NC	12.7%	26.2%	3.7%	15.9%	17.9%	12.0%	33.8%
	Very High	Number	--	--	--	--	--	7	5	--
		Minimum	--	--	--	--	--	8.4%	3.8%	--
		Maximum	--	--	--	--	--	30.9%	18.6%	--
		Mean	--	--	--	--	--	18.9%	10.7%	--
		Median	--	--	--	--	--	15.7%	9.4%	--
	All Soil	Number	0	23	12	34	26	38	33	16
		Minimum	NC	2.0%	17.4%	3.1%	0.1%	4.4%	1.7%	1.1%
		Maximum	NC	71.7%	48.5%	89.0%	28.2%	30.9%	78.7%	105.2%
		Mean	NC	16.5%	29.7%	30.0%	13.9%	16.5%	18.1%	38.5%
		Median	NC	14.3%	30.0%	22.1%	13.3%	16.6%	14.4%	25.6%

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

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Low	Number	22	4	0	13	20
		Minimum	7.8%	1.4%	NC	14.7%	2.5%
		Maximum	70.0%	27.4%	NC	51.0%	42.0%
		Mean	22.7%	14.6%	NC	26.9%	15.7%
		Median	19.5%	14.9%	NC	27.1%	15.5%
	Medium	Number	5	5	0	4	6
		Minimum	5.0%	5.9%	NC	6.0%	4.3%
		Maximum	14.4%	22.3%	NC	35.7%	20.1%
		Mean	10.3%	15.1%	NC	16.9%	12.2%
		Median	11.6%	18.9%	NC	12.9%	13.8%
	High	Number	6	4	4	4	9
		Minimum	1.5%	3.7%	37.6%	0.4%	0.7%
		Maximum	35.8%	7.0%	125.3%	14.5%	37.4%
		Mean	17.8%	5.6%	66.4%	7.2%	15.1%
		Median	19.5%	6.0%	51.4%	7.0%	12.0%
	Very High	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Soil	Number	33	13	4	21	35
		Minimum	1.5%	1.4%	37.6%	0.4%	0.7%
		Maximum	70.0%	27.4%	125.3%	51.0%	42.0%
		Mean	19.9%	12.0%	66.4%	21.3%	14.9%
		Median	17.3%	7.0%	51.4%	18.2%	14.7%

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

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
Sediment	Low	Number	0	17	0	21	8	3	16	2
		Minimum	NC	6.4%	NC	12.2%	6.1%	9.7%	4.7%	16.8%
		Maximum	NC	63.1%	NC	116.3%	58.3%	20.6%	115.6%	94.7%
		Mean	NC	28.9%	NC	35.8%	17.0%	16.9%	28.0%	55.8%
		Median	NC	28.3%	NC	32.5%	11.1%	20.3%	24.4%	55.8%
	Medium	Number	0	4	0	3	4	19	4	4
		Minimum	NC	5.2%	NC	9.9%	2.7%	6.4%	4.8%	26.0%
		Maximum	NC	13.5%	NC	17.6%	31.3%	61.7%	75.9%	75.3%
		Mean	NC	8.7%	NC	12.5%	17.1%	21.3%	24.5%	40.2%
		Median	NC	8.1%	NC	9.9%	17.2%	14.0%	8.6%	29.8%
	High	Number	0	2	0	3	10	4	3	2
		Minimum	NC	4.5%	NC	6.9%	4.3%	8.2%	11.5%	9.0%
		Maximum	NC	15.8%	NC	20.4%	45.5%	29.9%	93.2%	16.8%
		Mean	NC	10.1%	NC	11.5%	24.6%	17.5%	38.9%	12.9%
		Median	NC	10.1%	NC	7.2%	22.5%	16.0%	11.9%	12.9%
	Very High	Number	--	--	--	--	--	6	--	--
		Minimum	--	--	--	--	--	11.2%	--	--
		Maximum	--	--	--	--	--	29.4%	--	--
		Mean	--	--	--	--	--	18.0%	--	--
		Median	--	--	--	--	--	17.5%	--	--
	All Sediment	Number	0	23	0	27	22	32	23	8
		Minimum	NC	4.5%	NC	6.9%	2.7%	6.4%	4.7%	9.0%
		Maximum	NC	63.1%	NC	116.3%	58.3%	61.7%	115.6%	94.7%
		Mean	NC	23.8%	NC	30.5%	20.5%	19.8%	28.8%	37.3%
		Median	NC	18.8%	NC	25.1%	16.2%	16.5%	21.3%	26.9%

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

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
Sediment	Low	Number	18	5	1	6	19
		Minimum	5.1%	3.8%	46.5%	15.3%	6.2%
		Maximum	77.6%	98.8%	46.5%	86.1%	38.5%
		Mean	31.6%	32.6%	46.5%	37.7%	21.6%
		Median	31.6%	5.6%	46.5%	31.0%	21.7%
	Medium	Number	6	4	2	8	5
		Minimum	7.0%	3.6%	50.2%	5.4%	6.9%
		Maximum	41.9%	28.1%	50.2%	47.6%	13.1%
		Mean	19.2%	15.8%	50.2%	24.4%	10.6%
		Median	17.0%	15.8%	50.2%	24.4%	11.2%
	High	Number	4	3	2	3	4
		Minimum	8.2%	2.8%	41.0%	5.6%	8.1%
		Maximum	25.9%	9.0%	73.9%	12.9%	24.2%
		Mean	14.0%	6.7%	57.4%	9.5%	16.3%
		Median	10.9%	8.2%	57.4%	9.9%	16.4%
	Very High	Number	--	--	--	--	--
		Minimum	--	--	--	--	--
		Maximum	--	--	--	--	--
		Mean	--	--	--	--	--
		Median	--	--	--	--	--
	All Sediment	Number	28	12	5	17	28
		Minimum	5.1%	2.8%	41.0%	5.4%	6.2%
		Maximum	77.6%	98.8%	73.9%	86.1%	38.5%
		Mean	26.4%	20.5%	52.4%	26.5%	18.8%
		Median	18.8%	8.6%	50.2%	27.0%	15.5%

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

Matrix	Conc Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury
All	All	Number	0	46	12	61	48	70	56	24
		Minimum	NC	2.0%	17.4%	3.1%	0.1%	4.4%	1.7%	1.1%
		Maximum	NC	71.7%	48.5%	116.3%	58.3%	61.7%	115.6%	105.2%
		Mean	NC	20.1%	29.7%	30.2%	16.9%	18.0%	22.5%	38.1%
		Median	NC	15.1%	30.0%	24.2%	15.2%	16.5%	14.5%	26.0%
All Samples	All Instruments	Number	421	498	440	492	531	560	507	435
		Minimum	0.5%	1.1%	1.3%	1.0%	0.3%	0.1%	0.4%	2.0%
		Maximum	23.2%	33.6%	29.5%	36.4%	32.8%	20.9%	29.6%	24.9%
		Mean	8.5%	9.1%	7.2%	12.1%	9.6%	3.5%	6.9%	7.5%
		Median	11.0%	11.5%	6.0%	15.6%	7.3%	2.2%	7.4%	11.3%

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

Matrix	Conc Range	Statistic	Nickel	Selenium	Silver	Vanadium	Zinc
All	All	Number	61	25	9	38	63
		Minimum	1.5%	1.4%	37.6%	0.4%	0.7%
		Maximum	77.6%	98.8%	125.3%	86.1%	42.0%
		Mean	22.9%	16.1%	58.6%	23.6%	16.7%
		Median	18.5%	8.2%	50.2%	19.8%	14.7%
All Samples	All Instruments	Number	509	433	441	462	535
		Minimum	1.6%	0.3%	1.5%	0.9%	0.1%
		Maximum	28.1%	18.5%	24.8%	24.3%	19.3%
		Mean	10.9%	5.9%	9.0%	8.8%	6.6%
		Median	8.4%	10.6%	12.9%	11.4%	5.8%

Notes:

--	No samples reported by the reference laboratory in this concentration range.
Conc	Concentration.
NC	Not calculated due to 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	Selenium	Silver	Vanadium	Zinc
All Soil	Number	13	13	21	35
	Minimum	0.0%	2.3%	0.0%	1.0%
	Maximum	22.7%	37.1%	18.1%	46.5%
	Mean	8.9%	12.4%	8.4%	10.4%
	Median	7.1%	7.5%	6.6%	9.1%
All Sediment	Number	12	10	17	27
	Minimum	1.3%	1.0%	2.2%	1.4%
	Maximum	37.3%	21.3%	21.9%	35.8%
	Mean	10.0%	9.4%	8.4%	8.9%
	Median	7.6%	6.6%	8.1%	6.9%
All Samples	Number	25	23	38	62
	Minimum	0.0%	1.0%	0.0%	1.0%
	Maximum	37.3%	37.1%	21.9%	46.5%
	Mean	9.4%	11.1%	8.4%	9.8%
	Median	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	42	5	14	39	1	8
RPD of Target Element[2]	Minimum	-41.9%	-5.5%	-93.5%	-29.2%	4.4%	6.2%	-35.6%	-16.1%	-42.5%
	Maximum	21.4%	32.6%	181.4%	69.4%	53.8%	77.2%	75.7%	-16.1%	14.3%
	Mean	-13.4%	4.1%	25.3%	11.2%	36.8%	43.6%	3.2%	-16.1%	-8.3%
	Median	-12.6%	-2.7%	24.6%	8.3%	42.2%	41.6%	-0.5%	-16.1%	-5.5%
RPD of Target Element (Absolute Value)[2]	Minimum	0.4%	2.7%	3.5%	0.9%	4.4%	6.2%	0.4%	16.1%	3.5%
	Maximum	41.9%	32.6%	181.4%	69.4%	53.8%	77.2%	75.7%	16.1%	42.5%
	Mean	15.8%	8.9%	55.7%	17.8%	36.8%	43.6%	14.8%	16.1%	15.5%
	Median	12.9%	5.5%	43.0%	13.8%	42.2%	41.6%	11.1%	16.1%	14.4%
Interferent Concentration Range	Minimum	ND	865	1108	ND	683	655	ND	288	890
	Maximum	1030	61480	23605	938	1393	7308	875	288	3201
	Mean	221	19418	6276	216	1000	2481	144	288	1943
	Median	89	9262	3434	123	871	1877	98	288	1906
Target Element Concentration Range	Minimum	25	119	1	42	88	23	56	92	81
	Maximum	2065	5133	1788	3201	136	251	7308	92	127
	Mean	302	1701	323	526	110	94	1209	92	105
	Median	135	1071	56	183	100	75	786	92	107

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	50	3	10
RPD of Target Element[2]	Minimum	-35.6%	-6.2%	-42.5%	-83.3%	-14.0%	-38.6%
	Maximum	75.7%	7.1%	34.8%	35.7%	-7.1%	53.9%
	Mean	1.5%	0.4%	-1.1%	-11.1%	-10.8%	-2.0%
	Median	-0.5%	0.4%	-1.0%	-12.0%	-11.4%	-8.5%
RPD of Target Element (Absolute Value)[2]	Minimum	0.4%	6.2%	1.0%	0.0%	7.1%	1.2%
	Maximum	75.7%	7.1%	42.5%	83.3%	14.0%	53.9%
	Mean	15.9%	6.6%	13.6%	18.4%	10.8%	23.2%
	Median	14.1%	6.6%	8.9%	16.8%	11.4%	17.2%
Interferent Concentration Range	Minimum	45	754	678	ND	829	683
	Maximum	7675	8170	8071	2491	1666	7308
	Mean	1084	4462	3143	468	1144	2830
	Median	177	4462	3015	169	938	2221
Target Element Concentration Range	Minimum	56	149	73	45	118	57
	Maximum	7308	1553	382	8170	223	411
	Mean	1280	851	144	1674	156	158
	Median	829	851	124	674	127	145

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 due to 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	1	1
			Minimum	--	--	--	--	181.4%	181.4%	-0.8%	0.8%
			Maximum	--	--	--	--	181.4%	181.4%	-0.8%	0.8%
			Mean	--	--	--	--	181.4%	181.4%	-0.8%	0.8%
			Median	--	--	--	--	181.4%	181.4%	-0.8%	0.8%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	--	--	--	--	7	7	5	5
			Minimum	--	--	--	--	-10.2%	2.7%	-13.7%	13.7%
			Maximum	--	--	--	--	49.0%	49.0%	138.5%	138.5%
			Mean	--	--	--	--	3.3%	11.8%	61.6%	67.1%
			Median	--	--	--	--	-3.1%	5.5%	82.5%	82.5%
Soil	CN	Sandy loam (burn pit residue)	Number	--	--	--	--	1	1	1	1
			Minimum	--	--	--	--	-17.6%	17.6%	83.8%	83.8%
			Maximum	--	--	--	--	-17.6%	17.6%	83.8%	83.8%
			Mean	--	--	--	--	-17.6%	17.6%	83.8%	83.8%
			Median	--	--	--	--	-17.6%	17.6%	83.8%	83.8%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	--	--	--	--	--	--
			Minimum	--	--	--	--	--	--	--	--
			Maximum	--	--	--	--	--	--	--	--
			Mean	--	--	--	--	--	--	--	--
			Median	--	--	--	--	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	--	--	--	--	11	11	1	1
			Minimum	--	--	--	--	-93.5%	0.4%	-6.1%	6.1%
			Maximum	--	--	--	--	1.6%	93.5%	-6.1%	6.1%
			Mean	--	--	--	--	-17.6%	18.1%	-6.1%	6.1%
			Median	--	--	--	--	-9.2%	9.2%	-6.1%	6.1%
Sediment	RF	Silty fine sand (tailings)	Number	--	--	--	--	12	12	--	--
			Minimum	--	--	--	--	-38.4%	1.4%	--	--
			Maximum	--	--	--	--	68.4%	68.4%	--	--
			Mean	--	--	--	--	-5.0%	21.8%	--	--
			Median	--	--	--	--	-12.7%	17.7%	--	--

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

Matrix	Site	Matrix Description	Statistic	Chromium		Copper		Iron		Lead	
				Reference Laboratory		Reference Laboratory		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	2	2	3	3	3	3	3	3
			Minimum	-12.1%	9.6%	-35.6%	8.9%	-56.2%	0.0%	-61.9%	21.4%
			Maximum	-9.6%	12.1%	34.8%	35.6%	18.6%	56.2%	-21.4%	61.9%
			Mean	-10.8%	10.8%	2.7%	26.5%	-12.5%	24.9%	-35.4%	35.4%
			Median	-10.8%	10.8%	8.9%	34.8%	0.0%	18.6%	-22.9%	22.9%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	7	7	6	6	7	7	7	7
			Minimum	-21.8%	10.2%	-18.1%	3.5%	-25.5%	8.7%	-22.7%	9.8%
			Maximum	28.6%	28.6%	3.9%	18.1%	-8.7%	25.5%	-9.8%	22.7%
			Mean	-1.6%	17.8%	-7.8%	10.2%	-16.0%	16.0%	-18.5%	18.5%
			Median	-10.9%	17.9%	-10.3%	10.3%	-15.8%	15.8%	-19.8%	19.8%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	3	3	3	3	3	3
			Minimum	-50.0%	50.0%	-20.7%	14.0%	-28.2%	2.4%	-27.2%	2.2%
			Maximum	130.1%	130.1%	26.8%	26.8%	-2.4%	28.2%	122.7%	122.7%
			Mean	40.1%	90.1%	-2.6%	20.5%	-17.1%	17.1%	32.6%	50.7%
			Median	40.1%	90.1%	-14.0%	20.7%	-20.8%	20.8%	2.2%	27.2%
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	-31.5%	7.1%	-11.1%	5.0%	-62.2%	13.6%	-31.9%	11.2%
			Maximum	7.1%	31.5%	5.0%	11.1%	-13.6%	62.2%	-11.2%	31.9%
			Mean	-14.3%	17.8%	-3.1%	8.0%	-33.2%	33.2%	-20.6%	20.6%
			Median	-16.3%	16.3%	-3.1%	8.0%	-30.3%	30.3%	-19.9%	19.9%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	11	11	4	4	12	12	6	6
			Minimum	-39.2%	3.0%	-23.4%	0.7%	-57.9%	2.1%	-70.9%	39.9%
			Maximum	11.4%	39.2%	7.0%	23.4%	35.8%	57.9%	-39.9%	70.9%
			Mean	-12.6%	16.0%	-2.8%	9.2%	-22.3%	28.2%	-47.6%	47.6%
			Median	-11.7%	11.7%	2.5%	6.4%	-19.0%	30.7%	-42.8%	42.8%
Sediment	RF	Silty fine sand (tailings)	Number	12	12	13	13	13	13	13	13
			Minimum	-63.8%	5.5%	-42.5%	1.2%	-46.7%	3.4%	-56.0%	0.1%
			Maximum	24.8%	63.8%	22.1%	42.5%	6.0%	46.7%	-0.1%	56.0%
			Mean	-15.9%	22.1%	-0.7%	14.6%	-22.3%	23.2%	-25.2%	25.2%
			Median	-16.5%	22.2%	4.6%	15.9%	-20.5%	20.5%	-23.0%	23.0%

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
Soil	AS	Fine to medium sand (steel processing)	Number	--	--	1	1	1	1	1	1
			Minimum	--	--	74.6%	74.6%	-110.7%	110.7%	19.3%	19.3%
			Maximum	--	--	74.6%	74.6%	-110.7%	110.7%	19.3%	19.3%
			Mean	--	--	74.6%	74.6%	-110.7%	110.7%	19.3%	19.3%
			Median	--	--	74.6%	74.6%	-110.7%	110.7%	19.3%	19.3%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	1	1	6	6	4	4	1	1
			Minimum	110.4%	110.4%	-4.2%	0.9%	-1.3%	1.3%	5.6%	5.6%
			Maximum	110.4%	110.4%	48.9%	48.9%	80.8%	80.8%	5.6%	5.6%
			Mean	110.4%	110.4%	16.3%	17.7%	40.2%	40.8%	5.6%	5.6%
			Median	110.4%	110.4%	10.6%	10.6%	40.6%	40.6%	5.6%	5.6%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	3	3	2	2	--	--
			Minimum	16.2%	16.2%	-21.2%	21.2%	-5.2%	5.2%	--	--
			Maximum	30.7%	30.7%	22.4%	22.4%	6.7%	6.7%	--	--
			Mean	23.4%	23.4%	7.6%	21.7%	0.8%	6.0%	--	--
			Median	23.4%	23.4%	21.6%	21.6%	0.8%	6.0%	--	--
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	3	3	--	--	--	--
			Minimum	--	--	-29.2%	10.1%	--	--	--	--
			Maximum	--	--	12.2%	29.2%	--	--	--	--
			Mean	--	--	-9.0%	17.1%	--	--	--	--
			Median	--	--	-10.1%	12.2%	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	11	11	5	5	1	1
			Minimum	11.7%	11.7%	-10.2%	4.0%	-27.7%	6.9%	-34.5%	34.5%
			Maximum	53.5%	53.5%	43.0%	43.0%	17.4%	27.7%	-34.5%	34.5%
			Mean	37.3%	37.3%	10.0%	14.5%	-8.2%	15.1%	-34.5%	34.5%
			Median	41.9%	41.9%	7.8%	10.2%	-8.8%	14.7%	-34.5%	34.5%
Sediment	RF	Silty fine sand (tailings)	Number	4	4	13	13	5	5	2	2
			Minimum	-0.4%	0.4%	5.1%	5.1%	-23.1%	0.2%	-64.8%	64.8%
			Maximum	27.9%	27.9%	69.4%	69.4%	11.6%	23.1%	114.6%	114.6%
			Mean	12.5%	12.7%	38.5%	38.5%	-6.0%	10.6%	24.9%	89.7%
			Median	11.2%	11.2%	36.7%	36.7%	-4.8%	11.6%	24.9%	89.7%

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

Matrix	Site	Matrix Description	Statistic	Vanadium		Zinc	
				Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	AS	Fine to medium sand (steel processing)	Number	1	1	3	3
			Minimum	68.7%	68.7%	-35.7%	14.5%
			Maximum	68.7%	68.7%	-14.5%	35.7%
			Mean	68.7%	68.7%	-21.9%	21.9%
			Median	68.7%	68.7%	-15.4%	15.4%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	7	7
			Minimum	-6.5%	0.1%	-23.6%	10.3%
			Maximum	22.3%	22.3%	-10.3%	23.6%
			Mean	9.1%	12.3%	-17.2%	17.2%
			Median	10.2%	13.5%	-17.9%	17.9%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	3	3
			Minimum	14.5%	14.5%	-27.3%	7.3%
			Maximum	14.5%	14.5%	-7.3%	27.3%
			Mean	14.5%	14.5%	-17.4%	17.4%
			Median	14.5%	14.5%	-17.6%	17.6%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	2	2
			Minimum	--	--	-14.0%	0.0%
			Maximum	--	--	0.0%	14.0%
			Mean	--	--	-7.0%	7.0%
			Median	--	--	-7.0%	7.0%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	9	9	10	10
			Minimum	-24.7%	0.2%	-44.0%	0.7%
			Maximum	66.0%	66.0%	0.7%	44.0%
			Mean	8.8%	22.9%	-20.9%	21.0%
			Median	-0.2%	15.0%	-19.0%	19.0%
Sediment	RF	Silty fine sand (tailings)	Number	3	3	13	13
			Minimum	-24.3%	16.7%	-11.6%	1.0%
			Maximum	-16.7%	24.3%	35.7%	35.7%
			Mean	-19.4%	19.4%	9.3%	14.3%
			Median	-17.2%	17.2%	8.2%	11.6%

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

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	SB	Coarse sand and gravel (ore and waste rock)	Number	--	--	--	--	5	5	1	1
			Minimum	--	--	--	--	-41.9%	8.4%	6.3%	6.3%
			Maximum	--	--	--	--	-8.4%	41.9%	6.3%	6.3%
			Mean	--	--	--	--	-26.0%	26.0%	6.3%	6.3%
			Median	--	--	--	--	-20.7%	20.7%	6.3%	6.3%
Sediment	TL	Silt and clay (slag-enriched)	Number	--	--	--	--	2	2	--	--
			Minimum	--	--	--	--	-27.4%	27.4%	--	--
			Maximum	--	--	--	--	56.8%	56.8%	--	--
			Mean	--	--	--	--	14.7%	42.1%	--	--
			Median	--	--	--	--	14.7%	42.1%	--	--
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	--	--	--	7	7	3	3
			Minimum	--	--	--	--	-21.3%	3.5%	-168.1%	148.8%
			Maximum	--	--	--	--	37.0%	37.0%	-148.8%	168.1%
			Mean	--	--	--	--	8.7%	17.3%	-160.7%	160.7%
			Median	--	--	--	--	9.2%	12.2%	-165.3%	165.3%
	All		Number	--	--	--	--	46	46	12	12
			Minimum	--	--	--	--	-93.5%	0.4%	-168.1%	0.8%
			Maximum	--	--	--	--	181.4%	181.4%	138.5%	168.1%
			Mean	--	--	--	--	-2.3%	23.4%	-7.6%	76.2%
			Median	--	--	--	--	-6.8%	13.4%	2.7%	83.2%

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

Matrix	Site	Matrix Description	Statistic	Chromium		Copper		Iron		Lead	
				Reference Laboratory		Reference Laboratory		Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	11	11	4	4	12	12	7	7
			Minimum	-83.8%	6.9%	-14.5%	1.0%	-25.5%	2.3%	-13.6%	9.1%
			Maximum	-6.9%	83.8%	14.3%	14.5%	7.9%	25.5%	108.8%	108.8%
			Mean	-40.8%	40.8%	-3.5%	10.7%	-11.1%	12.4%	35.1%	39.0%
			Median	-39.8%	39.8%	-7.0%	13.6%	-12.6%	12.6%	19.5%	19.5%
Sediment	TL	Silt and clay (slag-enriched)	Number	5	5	7	7	7	7	4	4
			Minimum	15.0%	15.0%	-25.6%	0.4%	-73.5%	1.5%	5.1%	5.1%
			Maximum	30.4%	30.4%	75.7%	75.7%	8.9%	73.5%	39.7%	39.7%
			Mean	22.7%	22.7%	21.0%	28.5%	-24.7%	27.7%	23.0%	23.0%
			Median	20.3%	20.3%	8.5%	19.2%	-7.6%	8.9%	23.5%	23.5%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	7	7	6	6	7	7	7	7
			Minimum	-17.8%	0.5%	-14.1%	0.5%	-27.3%	1.3%	-89.4%	7.3%
			Maximum	51.8%	51.8%	7.1%	14.1%	1.3%	27.3%	-7.3%	89.4%
			Mean	7.3%	16.0%	-2.9%	5.2%	-10.9%	11.2%	-35.5%	35.5%
			Median	0.5%	11.5%	-1.8%	4.2%	-7.6%	7.6%	-25.7%	25.7%
	All		Number	61	61	48	48	70	70	56	56
			Minimum	-83.8%	0.5%	-42.5%	0.4%	-73.5%	0.0%	-89.4%	0.1%
			Maximum	130.1%	130.1%	75.7%	75.7%	35.8%	73.5%	122.7%	122.7%
			Mean	-10.2%	24.8%	0.9%	15.0%	-19.1%	21.4%	-14.0%	31.0%
			Median	-11.2%	18.1%	-0.6%	12.3%	-16.4%	17.3%	-21.2%	22.9%

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
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	11	11	11	11	3	3	--	--
			Minimum	-94.0%	4.7%	-28.1%	1.0%	-17.4%	5.5%	--	--
			Maximum	73.0%	94.0%	18.6%	28.1%	5.5%	17.4%	--	--
			Mean	23.0%	41.0%	0.3%	9.1%	-7.2%	10.8%	--	--
			Median	28.7%	43.3%	1.0%	5.7%	-9.6%	9.6%	--	--
Sediment	TL	Silt and clay (slag-enriched)	Number	2	2	6	6	4	4	2	2
			Minimum	47.7%	47.7%	18.8%	18.8%	-50.7%	4.4%	8.5%	8.5%
			Maximum	115.8%	115.8%	77.2%	77.2%	81.0%	81.0%	59.1%	59.1%
			Mean	81.7%	81.7%	36.4%	36.4%	1.9%	38.5%	33.8%	33.8%
			Median	81.7%	81.7%	30.1%	30.1%	-11.2%	34.4%	33.8%	33.8%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	--	7	7	1	1	2	2
			Minimum	--	--	6.2%	6.2%	4.8%	4.8%	-8.9%	8.9%
			Maximum	--	--	62.8%	62.8%	4.8%	4.8%	12.0%	12.0%
			Mean	--	--	37.9%	37.9%	4.8%	4.8%	1.6%	10.4%
			Median	--	--	50.0%	50.0%	4.8%	4.8%	1.6%	10.4%
	All		Number	24	24	61	61	25	25	9	9
			Minimum	-94.0%	0.4%	-29.2%	0.9%	-110.7%	0.2%	-64.8%	5.6%
			Maximum	115.8%	115.8%	77.2%	77.2%	81.0%	110.7%	114.6%	114.6%
			Mean	32.2%	40.5%	20.7%	25.3%	-1.1%	24.2%	12.3%	36.4%
			Median	29.7%	33.0%	18.8%	21.2%	-4.8%	11.6%	8.5%	19.3%

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

Matrix	Site	Matrix Description	Statistic	Vanadium		Zinc	
				Reference Laboratory		Reference Laboratory	
				RPD	RPD ABS Val	RPD	RPD ABS Val
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	10	10	11	11
			Minimum	-50.4%	4.3%	-83.3%	10.9%
			Maximum	20.7%	50.4%	11.1%	83.3%
			Mean	-26.7%	31.7%	-25.7%	27.8%
			Median	-33.7%	33.7%	-22.9%	22.9%
Sediment	TL	Silt and clay (slag-enriched)	Number	7	7	7	7
			Minimum	23.5%	23.5%	-38.6%	1.2%
			Maximum	94.3%	94.3%	53.9%	53.9%
			Mean	57.8%	57.8%	7.5%	22.8%
			Median	52.4%	52.4%	1.2%	7.9%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	3	7	7
			Minimum	-13.9%	7.9%	-18.0%	0.9%
			Maximum	8.2%	13.9%	16.0%	18.0%
			Mean	0.7%	10.0%	-5.3%	10.1%
			Median	7.9%	8.2%	-4.5%	9.9%
	All		Number	38	38	63	63
			Minimum	-50.4%	0.1%	-83.3%	0.0%
			Maximum	94.3%	94.3%	53.9%	83.3%
			Mean	7.3%	30.2%	-9.7%	18.8%
			Median	2.1%	22.9%	-11.5%	16.0%

Site Abbreviations:

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

Other Notes:

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