



United States
Environmental Protection
Agency

Office of Research and
Development
Washington, DC 20460

EPA/540/R-06/002
February 2006

Innovative Technology Verification Report

XRF Technologies for Measuring Trace Elements in Soil and Sediment

Innov-X XT400 Series XRF Analyzer



Innovative Technology Verification Report

Innov-X XT400 Series 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.

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Abstract

The Innov-X XT400 Series (XT400) 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 XT400 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 XT400 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 Innov-X XT400 portable XRF analyzer features a miniature, rugged x-ray tube excitation source for analyzing a wide variety of elements and sample materials, including alloys, environmental solids, and other analytical samples. The x-ray tube source and Light Element Analysis Program (LEAP) technology analyzes elements that would require three isotope sources in an isotope-based XRF analyzer. Other features of the XT400 include: multiple x-ray beam filters, multiple calibration methods, and adjustable tube voltages and currents.

The analyzer weighs 4.5 pounds and can be powered in the field with a lithium-ion battery or 110-volt alternating current (AC). The XT400 XRF analyzer utilizes a Hewlett-Packard (HP) iPAQ personal data assistant (PDA) for data storage of up to 10,000 tests with spectra in its 64 megabyte memory. The iPAQ has a color, high resolution display with variable backlighting and can be fitted with Bluetooth® wireless printing and data downloading, an integrated bar-code reader, and wireless data and file transfer accessories. The XT400 analyzer can analyze elements from potassium to uranium in suites of 25 elements simultaneously.

This report describes the results of the evaluation of the XT400 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 XT400 analyzer is compiled and compared to both fixed laboratory costs and average XRF instrument costs.

Contents

<u>Chapter</u>	<u>Page</u>
Notice.....	ii
Foreword.....	iii
Abstract.....	iv
Acronyms, Abbreviations, and Symbols.....	x
Acknowledgements.....	xiv
 1.0 INTRODUCTION	 1
1.1 Organization of this Report.....	1
1.2 Description of the SITE Program	2
1.3 Scope of the Demonstration.....	2
1.4 General Description of XRF Technology	3
1.5 Properties of the Target Elements.....	4
1.5.1 Antimony	5
1.5.2 Arsenic	5
1.5.3 Cadmium.....	5
1.5.4 Chromium.....	5
1.5.5 Copper.....	5
1.5.6 Iron.....	5
1.5.7 Lead	6
1.5.8 Mercury.....	6
1.5.9 Nickel.....	6
1.5.10 Selenium	6
1.5.11 Silver.....	7
1.5.12 Vanadium.....	7
1.5.13 Zinc	7
 2.0 FIELD SAMPLE COLLECTION LOCATIONS.....	 9
2.1 Alton Steel Mill Site	9
2.2 Burlington Northern-ASARCO Smelter Site.....	11
2.3 Kennedy Athletic, Recreational and Social Park Site.....	11
2.4 Leviathan Mine Site.....	12
2.5 Navy Surface Warfare Center, Crane Division Site	12
2.6 Ramsay Flats–Silver Bow Creek Site	13
2.7 Sulphur Bank Mercury Mine Site	13
2.8 Torch Lake Superfund Site	14
2.9 Wickes Smelter Site.....	14
 3.0 FIELD DEMONSTRATION	 15
3.1 Bulk Sample Processing	15
3.1.1 Bulk Sample Collection and Shipping.....	15
3.1.2 Bulk Sample Preparation and Homogenization.....	15
3.2 Demonstration Samples	17
3.2.1 Environmental Samples	17
3.2.2 Spiked Samples.....	17
3.2.3 Demonstration Sample Set.....	17

Contents (Continued)

<u>Chapter</u>		<u>Page</u>
3.3	Demonstration Site and Logistics	20
3.3.1	Demonstration Site Selection.....	20
3.3.2	Demonstration Site Logistics.....	20
3.3.3	EPA Demonstration Team and Developer Field Team Responsibilities	21
3.3.4	Sample Management During the Field Demonstration.....	21
3.3.5	Data Management.....	22
4.0	EVALUATION DESIGN.....	23
4.1	Evaluation Objectives	23
4.2	Experimental Design.....	23
4.2.1	Primary Objective 1 – Method Detection Limits.....	24
4.2.2	Primary Objective 2 – Accuracy	25
4.2.3	Primary Objective 3 – Precision	26
4.2.4	Primary Objective 4 – Impact of Chemical and Spectral Interferences	27
4.2.5	Primary Objective 5 – Effects of Soil Characteristics	28
4.2.6	Primary Objective 6 – Sample Throughput	28
4.2.7	Primary Objective 7 – Technology Costs	28
4.2.8	Secondary Objective 1 – Training Requirements	28
4.2.9	Secondary Objective 2 – Health and Safety	29
4.2.10	Secondary Objective 3 – Portability	29
4.2.11	Secondary Objective 4 – Durability.....	29
4.2.12	Secondary Objective 5 – Availability	29
4.3	Deviations from the Demonstration Plan.....	29
5.0	REFERENCE LABORATORY	31
5.1	Selection of Reference Methods	31
5.2	Selection of Reference Laboratory	32
5.3	QA/QC Results for Reference Laboratory.....	33
5.3.1	Reference Laboratory Data Validation	33
5.3.2	Reference Laboratory Technical Systems Audit	34
5.3.3	Other Reference Laboratory Data Evaluations	34
5.4	Summary of Data Quality and Usability.....	36
6.0	TECHNOLOGY DESCRIPTION	39
6.1	General Description	39
6.2	Instrument Operations during the Demonstration.....	41
6.2.1	Setup and Calibration.....	41
6.2.2	Demonstration Sample Processing	41
6.3	General Demonstration Results	42
6.4	Contact Information.....	42

Contents (Continued)

<u>Chapter</u>	<u>Page</u>
7.0 PERFORMANCE EVALUATION	43
7.1 Primary Objective 1 – Method Detection Limits	43
7.2 Primary Objective 2 – Accuracy and Comparability	46
7.3 Primary Objective 3 – Precision	52
7.4 Primary Objective 4 – Impact of Chemical and Spectral Interferences	55
7.5 Primary Objective 5 – Effects of Soil Characteristics	55
7.6 Primary Objective 6 – Sample Throughput	59
7.7 Primary Objective 7 – Technology Cost	60
7.8 Secondary Objective 1 – Training Requirements	60
7.9 Secondary Objective 2 – Health and Safety	60
7.10 Secondary Objective 3 – Portability	60
7.11 Secondary Objective 4 – Durability	61
7.12 Secondary Objective 5 – Availability	61
8.0 ECONOMIC ANALYSIS	63
8.1 Equipment Costs	63
8.2 Supply Costs	63
8.3 Labor Costs	63
8.4 Comparison of XRF Analysis and Reference Laboratory Costs	65
9.0 SUMMARY OF TECHNOLOGY PERFORMANCE	67
10.0 REFERENCES	73

APPENDICES

Appendix A:	Verification Statement
Appendix B:	Developer Discussion
Appendix C:	Data Validation Summary Report
Appendix D:	Developer and Reference Laboratory Data
Appendix E:	Statistical Data Summaries

Contents (Continued)

<u>TABLES</u>	<u>Page</u>
1-1 Participating Technology Developers and Instruments	1
2-1 Nature of Contamination in Soil and Sediment at Sample Collection Sites	10
2-2 Historical Analytical Data, Alton Steel Mill Site	11
2-3 Historical Analytical Data, BN-ASARCO Smelter Site	11
2-4 Historical Analytical Data, KARS Park Site	11
2-5 Historical Analytical Data, Leviathan Mine Site	12
2-6 Historical Analytical Data, NSW Crane Division-Old Burn Pit	13
2-7 Historical Analytical Data, Ramsay Flats-Silver Bow Creek Site	13
2-8 Historical Analytical Data, Sulphur Bank Mercury Mine Site	14
2-9 Historical Analytical Data, Torch Lake Superfund Site	14
2-10 Historical Analytical Data, Wickes Smelter Site-Roaster Slag Pile	14
3-1 Concentration Levels for Target Elements in Soil and Sediment	18
3-2 Number of Environmental Sample Blends and Demonstration Samples	19
3-3 Number of Spiked Sample Blends and Demonstration Samples	19
4-1 Evaluation Objectives	24
5-1 Number of Validation Qualifiers	35
5-2 Percent Recovery for Reference Laboratory Results in Comparison to ERA Certified Spike Values for Blends 46 through 70	37
5-3 Precision of Reference Laboratory Results for Blends 1 through 70	38
6-1 Innov-X XT400 XRF Analyzer Technical Specifications	40
7-1 Evaluation of Sensitivity – Method Detection Limits for the Innov-X XT400	44
7-2 Comparison of Mean XT400 MDLs to All-Instrument Mean MDLs and EPA Method 6200 Data	46
7-3 Evaluation of Accuracy – Relative Percent Differences versus Reference Laboratory Data for the Innov-X XT400	48
7-4 Summary of Correlation Evaluation for the XT400	50
7-5 Evaluation of Precision – Relative Standard Deviations for the Innov-X XT400	53
7-6 Evaluation of Precision – Relative Standard Deviations for the Reference Laboratory versus the XT400 and All Demonstration Instruments	54
7-7 Effects of Interferent Elements on the RPDs (Accuracy) for Other Target Elements, Innov-X XT400	56
7-8 Effect of Soil Type on the RPDs (Accuracy) for Target Elements, Innov-X XT400	57
7-9 RPDs Calculated for Wickes Smelter Sample Blends for the Innov-X XT400	59
8-1 Equipment Costs	63
8-2 Time Required to Complete Analytical Activities	64
8-3 Comparison of XRF Technology and Reference Method Costs	66
9-1 Summary of Innov-X XT400 Performance – Primary Objectives	68
9-2 Summary of Innov-X XT400 Performance – Secondary Objectives	70

Contents (Continued)

<u>FIGURES</u>	<u>Page</u>
1-1 The XRF Process	4
3-1 Bulk Sample Processing Diagram	16
3-2 KARS Park Recreation Building	20
3-3 Work Areas for the XRF Instruments in the Recreation Building.....	21
3-4 Visitors Day Presentation	21
3-5 Sample Storage Room.....	22
6-1 Innov-X XT400 Analyzer Set Up for Portable In Situ Analysis	39
6-2 Innov-X XT400 XRF Analyzer Set Up for Bench-Top Analysis.....	39
6-3 INNOV-x Technician Preparing Samples for Analysis.....	41
7-1 Linear Correlation Plot for Innov-X XT400 Showing High Correlation for Cadmium	49
7-2 Linear Correlation Plot for Innov-X ST400 Showing Low Correlation and Variable Bias for Vanadium	51
8-1 Comparison of Labor Requirements for the XT400 versus Other XRF Instruments	65
9-1 Method Detection Limits (sensitivity), Accuracy, and Precision of the XT400 in Comparison to the Average of All Eight XRF Instruments.....	71

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. Don Sackett and Rose Koch of Innov-X Systems; 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 Innov-X XT400 Series XRF analyzer based on the results of the demonstration.

1.1 Organization of this Report

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

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

Table 1-1. Participating Technology Developers and Instruments

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

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

1.2 Description of the SITE Program

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

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

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

- **Remediation Technology Program** – Demonstrates innovative treatment technologies to provide reliable data on performance, cost, and applicability for site cleanups.
- **Technology Transfer Program** – Provides and disseminates technical information in the form of updates, brochures, and other publications that promote the SITE Program and the participating technologies.

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

1.3 Scope of the Demonstration

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

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

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

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

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

Each developer analyzed all 326 samples during the field demonstration using its XRF instrument and in accordance with its standard operating procedure. The field demonstration was conducted during the week of January 24, 2005, at the Kennedy Athletic, Recreational and Social (KARS) Park, which is part of the Kennedy Space Center on Merritt Island, Florida. Observers were assigned to each XRF instrument during the field demonstration to collect detailed information on the instrument and operating procedures, including sample processing times, for

subsequent evaluation. The reference laboratory also analyzed a complete set of the demonstration samples for the target elements using acid digestion and inductively coupled plasma-atomic emission spectrometry (ICP-AES), in accordance with EPA Method 3050B/6010B, and using cold vapor atomic absorption (CVAA) spectroscopy (for mercury only) in accordance with EPA Method 7471A. By assuming that the results from the reference laboratory were essentially “true” values, instrument accuracy was assessed by comparing the results obtained using the XRF instrument with the results from the reference laboratory. The data obtained using the XRF instrument were also assessed in other ways, in accordance with the objectives of the demonstration, to provide information on instrument precision, detection limits, and interferences.

1.4 General Description of XRF Technology

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

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

As illustrated in Figure 1-1, characteristic radiation arises when the energy from the x-ray source exceeds the absorption edge energy of inner-shell electrons, ejecting one or more electrons. The vacancies are

filled by electrons that cascade in from the outer shells. The energy states of the electrons in the outer shells are higher than those of the inner-shell electrons, and the outer-shell electrons emit energy in the form of x-rays as they cascade down. The energy of this x-ray radiation is unique for each element.

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

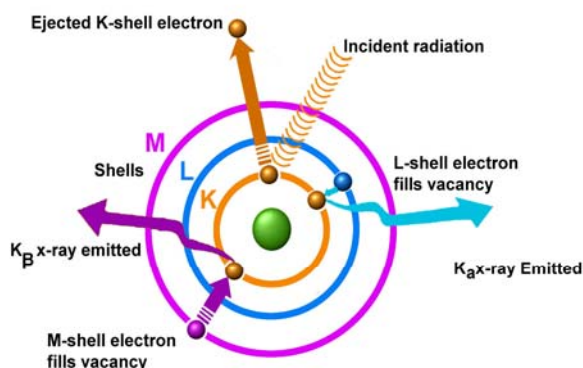


Figure 1-1. The XRF process.

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

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

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

2. Chemical matrix effects (absorption and enhancement phenomena) and Spectral interferences (peak overlaps).
3. Moisture content above 10 percent, which affects x-ray transmission.

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

1.5 Properties of the Target Elements

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

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

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

1.5.1 Antimony

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

1.5.2 Arsenic

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

1.5.3 Cadmium

Naturally occurring cadmium in surface soils typically ranges from 0.6 to 1.1 mg/kg; concentrations greater than 4 mg/kg are potentially phytotoxic. Concentrations of cadmium that exceed 37 mg/kg may result in adverse effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 50 mg/kg. Elevated

concentrations of cadmium are associated with mine wastes and industrial facilities. Cadmium is successfully analyzed by both ICP-AES and field-portable XRF; however, action levels for cadmium may be lower than the detection limits of field-portable XRF instruments.

1.5.4 Chromium

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

1.5.5 Copper

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

1.5.6 Iron

Although iron is not considered an element that poses a significant environmental consequence, it interferes with measurement of other elements and was

therefore included in the study. Furthermore, iron is often used as a target reference element in XRF analysis.

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

1.5.7 Lead

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

1.5.8 Mercury

Naturally occurring mercury in surface soils typically ranges from 0.01 to 0.3 mg/kg; concentrations greater than 0.3 mg/kg are potentially phytotoxic. Concentrations of mercury greater than 23 mg/kg and

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

1.5.9 Nickel

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

1.5.10 Selenium

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

ecological risk-based screening levels for soil. Analytical results for selenium using ICP-AES and XRF are expected to be comparable.

1.5.11 Silver

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

1.5.12 Vanadium

Naturally occurring vanadium in surface soils typically ranges from 20 to 500 mg/kg; concentrations greater than 2 mg/kg are potentially phytotoxic, although specific phytotoxicity levels for

naturally occurring vanadium have not been documented. Concentrations above 550 mg/kg may result in adverse health effects in humans. Typical detection limits for field-portable XRF instruments range from 10 to 50 mg/kg. Vanadium can be associated with manganese, potassium, and organic matter and is typically concentrated in organic shales, coal, and crude oil. It is successfully analyzed by both ICP-AES and XRF with little interference.

1.5.13 Zinc

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

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Chapter 2

Field Sample Collection Locations

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

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

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

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

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

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

2.1 Alton Steel Mill Site

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

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

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

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

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

Notes (in order of appearance in table):

Sb: Antimony

Cr: Chromium

Pb: Lead

Se: Selenium

As: Arsenic

Cu: Copper

Hg: Mercury

Ag: Silver

Cd: Cadmium

Fe: Iron

Ni: Nickel

Zn: Zinc

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

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

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

2.2 Burlington Northern-ASARCO Smelter Site

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

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

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

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

2.3 Kennedy Athletic, Recreational and Social Park Site

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

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

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

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

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

2.4 Leviathan Mine Site

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

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

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

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

2.5 Navy Surface Warfare Center, Crane Division Site

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

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

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

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

2.6 Ramsay Flats-Silver Bow Creek Site

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

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

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

Two sediment samples were collected from the Ramsay Flats tailings area and were analyzed for a suite of metals using a field-portable XRF. The contaminated sediment sample was collected in Silver Bow Creek adjacent to the mine tailings. The matrix of this sediment sample was orange-brown

silty fine sand with interlayered black organic material. The background sediment sample was collected upstream of Butte, Montana. The matrix of this sample was organic rich clayey silt with approximately 25 percent fine sand. The maximum concentrations of the target elements in the samples are summarized in Table 2-7.

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

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

2.7 Sulphur Bank Mercury Mine Site

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

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

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

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

2.8 Torch Lake Superfund Site

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

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

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

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

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

2.9 Wickes Smelter Site

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

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

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

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

Chapter 3

Field Demonstration

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

3.1 Bulk Sample Processing

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

3.1.1 Bulk Sample Collection and Shipping

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

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

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

3.1.2 Bulk Sample Preparation and Homogenization

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

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

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

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

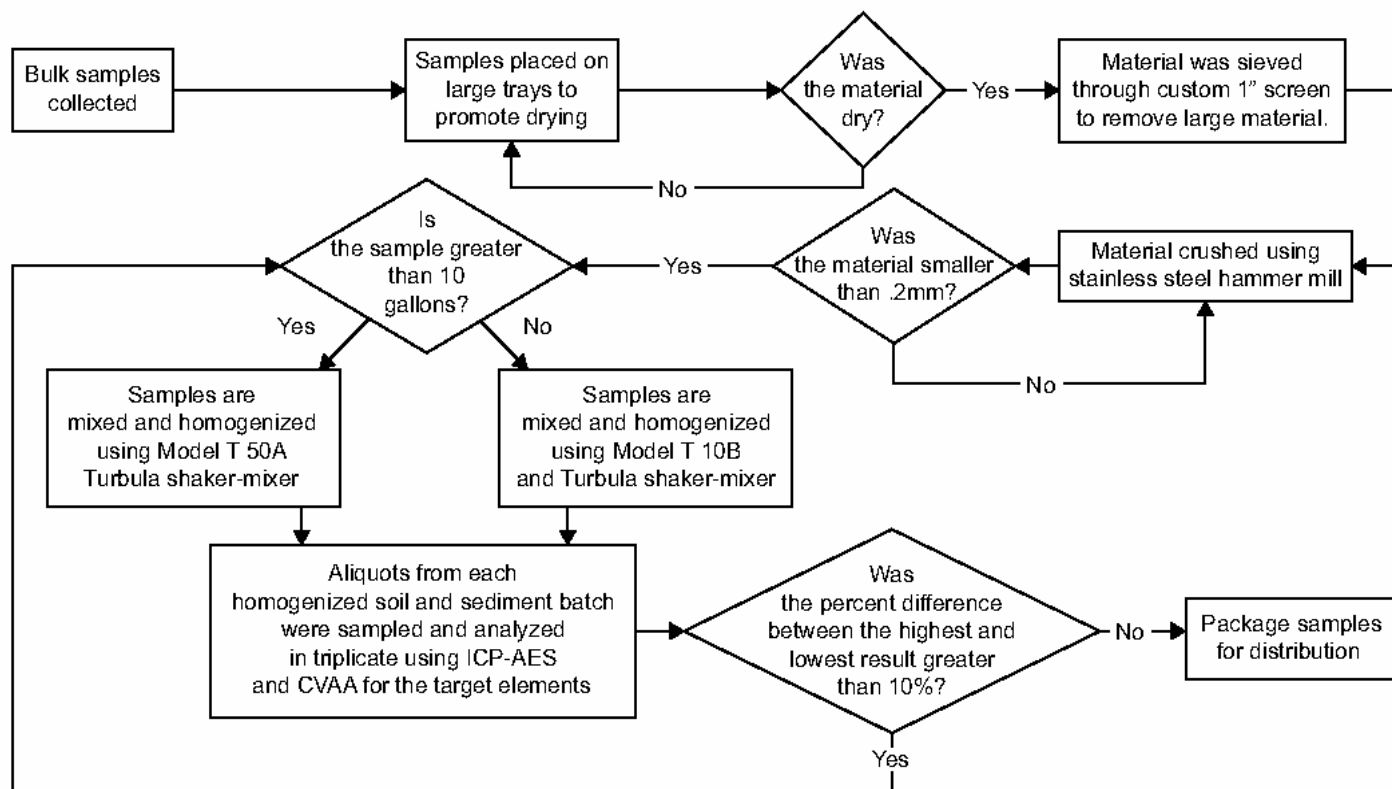


Figure 3-1. Bulk sample processing diagram.

3.2 Demonstration Samples

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

3.2.1 Environmental Samples

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

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

3.2.2 Spiked Samples

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

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

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

3.2.3 Demonstration Sample Set

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

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

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

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

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

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

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

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

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

3.3 Demonstration Site and Logistics

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

3.3.1 Demonstration Site Selection

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

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

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

- Access and Site Owner Support — Representatives from NASA were willing to support the field demonstration by providing access to the site, assisting in logistical support during the demonstration, and hosting a visitors day.
- Facilities Requirements and Feasibility — The recreation building was available and was of sufficient size to accommodate all the demonstration participants. Furthermore, the

recreation building had adequate power to operate all the XRF instruments simultaneously and all the amenities to fully support the demonstration participants, as well as visitors, in reasonable comfort.

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

3.3.2 Demonstration Site Logistics

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

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



Figure 3-2. KARS Park recreation building.



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



Figure 3-4. Visitors day presentation.

3.3.3 EPA Demonstration Team and Developer Field Team Responsibilities

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

The EPA/Tetra Tech demonstration team assigned an observer to each instrument. The observer sat beside the developer's field team, or was nearby, throughout the field demonstration and observed all activities

involved in setup and operation of the instrument. The observer's specific responsibilities included:

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

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

3.3.4 Sample Management during the Field Demonstration

The developer's field team analyzed the demonstration sample set with its XRF instrument during the field demonstration. Each demonstration sample set was shipped to the demonstration site with only a reference number on each bottle as an identifier. The reference number was tied to the source information in the EPA/Tetra Tech database, but no information was provided on the sample label

that might provide the developer's field team any insight as to the nature or content of the sample. Spiked samples were integrated with the environmental samples in a random manner so that the spiked samples could not be distinguished.

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

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

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



Figure 3-5. Sample storage room.

3.3.5 Data Management

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

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

Once the EPA/Tetra Tech field team returned to their offices, the demonstration data were transferred to a 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.

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.

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.

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

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

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

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

detection limit for iron because all the blends contained substantial native concentrations of iron.

4.2.2 Primary Objective 2 — Accuracy

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

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

1. The results for replicate samples within a blend were averaged for both the data from the XRF instrument and the reference laboratory. Since there were 70 sample blends, this step created a maximum of 70 paired results for the assessment.
2. A blend that exhibited one or more non-detect values in either the XRF instrument or the reference laboratory analysis was excluded from the evaluation.
3. A blend was excluded from the evaluation when the average result from the reference laboratory was below a minimum concentration. The minimum concentration for exclusion from the accuracy assessment was identified as the lower limit of the lowest concentration range (Level 1 in Table 3-1), which is about 50 ppm for most elements.
4. The mean result for a blend obtained with the XRF instrument was compared with the corresponding mean result from the reference laboratory by calculating a relative percent difference (RPD). This comparison was carried out for each of the paired XRF and reference laboratory results included in the evaluation (up to 70 pairs) as follows:

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

where

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

5. Steps 1 through 4 provided a set of up to 70 RPDs for each element (70 sample blends minus the number excluded in steps 1 and 2). The absolute value of each of the RPDs was taken and summary statistics (minimum, maximum, mean and median) were then calculated.
6. The accuracy of the XRF instrument for each target element was then categorized, based on the median of the absolute values of the RPDs, as either excellent (RPD less than 10 percent), good (RPD between 10 percent and 25 percent), fair (RPD between 25 percent and 50 percent), or poor (RPD above 50 percent).
7. The set of absolute values of the RPDs for each instrument and element was further evaluated to assess any trends in accuracy versus concentration. These evaluations involved grouping the RPDs by concentration range (Levels 1 through 3 and 4, as presented in Table 3-1), preparing summary statistics for each range, and assessing differences among the grouped RPDs.

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

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

bias between the data sets for the XRF instrument and the reference laboratory.

4.2.3 Primary Objective 3 — Precision

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

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

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

where

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

The standard deviation was calculated using the equation:

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

where

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

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

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

-
2. A blend that exhibited one or more non-detect values in either the XRF or the reference laboratory analysis was excluded from the evaluation.
 3. A blend was excluded from the evaluation when the average result from the reference laboratory was below a minimum concentration. The minimum concentration for exclusion from the precision assessment was identified as the lower limit of the lowest concentration range (Level 1 in Table 3-1), which was about 50 ppm for most elements.
 4. The RSDs for the various blends for both the XRF instrument and the reference laboratory were treated as a statistical population. Summary statistics (minimum, maximum, mean and median) were then calculated and compared for the data set as a whole and for the different concentration ranges (Levels 1 through 3 or 4).
 5. The precision of the XRF instrument for each target element was then categorized, based on the median RSDs, as either excellent (RSD less than 5 percent), good (RSD between 5 percent and 10 percent), fair (RSD between 10 percent and 20 percent), or poor (RSD above 20 percent).

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

4.2.4 Primary Objective 4 — Impact of Chemical and Spectral Interferences

The potential in the XRF analysis for spectral interference between adjacent elements on the periodic table was evaluated for the following element pairs: lead/arsenic, nickel/copper, and copper/zinc. The demonstration sample set included multiple blends where the concentration of one of

these elements was greater than 10 times the concentration of the other element in the pair to facilitate this evaluation. Interference effects were identified through evaluation of the RPDs for these sample blends, which were calculated according to the equation in Section 4.2.2, since spectral interferences would occur only in the XRF data and not in the reference laboratory data.

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

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

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

4.2.5 Primary Objective 5 — Effects of Soil Characteristics

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

4.2.6 Primary Objective 6 — Sample Throughput

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

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

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

4.2.7 Primary Objective 7 — Technology Costs

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

Based on input from each technology developer and from distributors, the instrument cost was established for purchase of the equipment and for daily, weekly,

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

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

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

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

4.2.8 Secondary Objective 1 — Training Requirements

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

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

4.2.9 Secondary Objective 2 — Health and Safety

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

4.2.10 Secondary Objective 3 — Portability

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

4.2.11 Secondary Objective 4 — Durability

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

4.2.12 Secondary Objective 5 — Availability

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

4.3 Deviations from the Demonstration Plan

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

1. An in-process audit of the reference laboratory was originally planned while the laboratory was analyzing the demonstration samples. However, the reference laboratory completed all analysis earlier than expected, during the week of the field demonstration, and thereby created a schedule conflict. Furthermore, it was decided that the original pre-award audit was adequate for assessing the laboratory's procedures and competence.
2. The plan suggested that each result for spiked samples from the reference laboratory would be replaced by the "certified analysis" result, which was quantitative based on the amount of each element spiked, whenever the RPD between these two results was greater than 10 percent. The project team agreed that 10 percent was too stringent for this evaluation, however, and decided to use 25 percent RPD as the criterion for assessing reference laboratory accuracy against the spiked samples. Furthermore, it was found during the data evaluations that replacing individual reference laboratory results using this criterion would result in a mixed data set. Therefore, the 25 percent criterion was applied to the overall mean RPD for each element, and the "certified analysis" data set for a specific target element was used as a supplement to the reference laboratory result when this criterion was exceeded.
3. Instrument accuracy and comparability in relation to the reference laboratory (Primary Objective 2) was originally planned to be assessed based on a combination of percent recovery (instrument result divided by reference laboratory result) and RPD. It was decided during the data analysis, however, that the RPD was a much better parameter for this assessment. Specifically, it was found that the mean or median of the absolute values of the RPD for each blend was a good discriminator of instrument performance for this objective.
4. Although this step was not described in the plan, some quantitative results for each instrument were compared with the overall average of all XRF instruments. Since there were eight instruments, it was believed that a comparison of

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

5. The plan proposed statistical testing in support of Primary Objectives 4 and 5. Specifically, the Wilcoxon Rank Sum (WRS) test was proposed to assist in evaluating interference effects, and the

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

Chapter 5

Reference Laboratory

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

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

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

5.1 Selection of Reference Methods

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

- Inductively coupled plasma-atomic emission spectroscopy (ICP-AES), in accordance with

EPA SW-846 Method 3050B/6010B, for all target elements except mercury.

- Cold vapor atomic absorption (CVAA) spectroscopy, in accordance with EPA SW-846 Method 7471A, for mercury only.

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

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

Inductively coupled plasma-mass spectrometry (ICP-MS) was considered as a possible analytical technique; however, fewer data were available to support the claims of accuracy and precision. Furthermore, it was available in less than one-third of the laboratories solicited for this project. Finally, ICP-MS is a technique for analysis of trace elements and often requires serial dilutions to mitigate the effect of high concentrations of interfering ions or other matrix interferences. These dilutions can introduce the possibility of error and contaminants that might bias the results. Since the matrices (soil

and sediment) for this demonstration are designed to contain high concentrations of elements and interfering ions, ICP-AES was selected over ICP-MS as the instrumental method best suited to meet the project objectives. The cost per analysis is also higher for ICP-MS in most cases than for ICP-AES.

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

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

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

Soil/Sediment Sample Preparation for Analysis of Mercury by CVAA. Method 7471A (CVAA) is the only method approved by EPA and promulgated for analysis of mercury. Method 7471A includes its own digestion procedure because more vigorous digestion

of samples, like that incorporated in Method 3050B, would volatilize mercury and produce inaccurate results. This technique is widely available, and extensive data are available that support the ability of this method to meet the objectives of the demonstration.

5.2 Selection of Reference Laboratory

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

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

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

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

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

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

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

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

Based on the results of the evaluation process, Shealy Environmental Services, Inc. (Shealy), of Cayce, South Carolina, received the highest score and was therefore selected as the reference laboratory. Shealy is accredited by the National Environmental Laboratory Accreditation Conference (NELAC). Once selected, Shealy analyzed all demonstration samples (both environmental and spiked samples) concurrently with the developers' analysis during the field demonstration. Shealy analyzed the samples by

ICP-AES using EPA SW-846 Method 3050B/6010B and by CVAA using EPA SW-846 Method 7471A.

5.3 QA/QC Results for Reference Laboratory

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

5.3.1 Reference Laboratory Data Validation

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

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

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

5.3.2 Reference Laboratory Technical Systems Audit

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

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

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

5.3.3 Other Reference Laboratory Data Evaluations

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

Blends 46 through 70 of the demonstration sample set consisted of certified spiked samples that were used to assess the accuracy of the reference laboratory data. The summary statistics from

comparing the “certified values” for the spiked samples with the reference laboratory results are shown in Table 5-2. The target for percent recovery was 75 to 125 percent. The mean percent recoveries for 12 of the 13 target elements were well within this accuracy goal. Only the mean recovery for antimony was outside the goal (26.8 percent). The low mean percent recovery for antimony supported the recommendation made by the project team to conduct

a secondary comparison of XRF data to ERA-certified spike values for antimony. This secondary evaluation was intended to better understand the impacts on the evaluation of the low bias for antimony in the reference laboratory data. All other recoveries were acceptable. Thus, this evaluation further supports the conclusion that the reference data set is of high quality.

Table 5-1. Number of Validation Qualifiers

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

Notes:

MS Matrix spike.

MSD Matrix spike duplicate.

QC Quality control.

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

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

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

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

A review of the ARDL characterization data in comparison to the reference laboratory data indicated that ARDL obtained lower recoveries of several elements. When expressed as a percent of the average reference laboratory result (percent recovery), the median ARDL result was below the lower QC limit of 75 percent recovery for three elements — chromium, nickel, and selenium. This discrepancy between data from the reference laboratory and ARDL was determined to have no

significant impact on reference laboratory data quality for three reasons: (1) the ARDL data were obtained on a rapid turnaround basis to evaluate homogeneity — accuracy was not a specific goal, (2) the ARDL data were not validated, and (3) all other quality measurement for the reference laboratory data indicated a high level of quality.

5.4 Summary of Data Quality and Usability

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

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

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

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

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

Notes:

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

ERA = Environmental Resource Associates, Inc.

NC = Not calculated.

%R = Percent recovery.

Source of certified values: Environmental Resource Associates, Inc.

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

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

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

Notes:

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

%RSD = Percent relative standard deviation.

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

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

Chapter 6

Technology Description

The XT400 Series XRF analyzer is manufactured by Innov-X Systems. This chapter provides a technical description of the XT400 based on information obtained from Innov-X and observation of this analyzer during the field demonstration. This chapter also identifies an Innov-X company contact, where additional technical information may be obtained.

6.1 General Description

The Innov-X XT400 features a miniature x-ray tube excitation source for analyzing a wide variety of elements and sample materials, including alloys, environmental solids, and other analytical samples (Figure 6-1). The x-ray tube source and Light Element Analysis Program (LEAP) technology can be used to analyze for elements that would otherwise require three isotope sources in an isotope-based XRF analyzer. The XT400 features:

- Multiple x-ray beam filters.
- Adjustable tube voltages and currents.
- Multiple calibration methods.
 - a) Fundamental parameters.
 - b) Compton normalization.
 - c) Empirical – factory and user generated linear, quadratic and exponential calibrations.
 - d) Scatter normalization (trace elements and low-density matrices.
 - e) Spectral matching (rapid material sorting and product authentication).

The XT400 uses a Hewlett-Packard (HP) iPAQ personal data assistant (PDA) for data storage of up to 10,000 tests with spectra in its 64 megabyte (MB) memory. The iPAQ has a color, high-resolution display with variable backlighting, and it can be fitted with Bluetooth® wireless printing and data downloading, an integrated bar-code reader, and wireless data and file transfer accessories.

The XT400 can analyze elements from potassium to uranium in suites of 25 elements simultaneously. Typical applications are:

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

The technical specifications for the XT400 are presented in Table 6-1. The analyzer can be set up either as a hand-held instrument for portable in situ analysis (Figure 6-1) or as a bench-top instrument using a triangular testing stand and protective hood (Figure 6-2).



Figure 6-1. Innov-X XT400 XRF analyzer set up for portable in situ analysis.



Figure 6-2. Innov-X XT400 XRF analyzer set up for bench-top analysis.

To analyze soil samples in the in situ mode, the instrument x-ray window is placed directly on the ground or on soils in a plastic bag. In situ testing with the XT400 allows for semi-quantitative assessment of element concentrations at multiple locations or over large areas in a short time. For ex situ analysis, samples are prepared in x-ray sample cups and placed over the analyzer x-ray window in the test stand. Quantitative ex situ testing involves properly preparing the samples, placing the samples in x-ray sample cups, and analyzing them in a controlled area, typically free from dust and weather

extremes. Most field-portable XRF analyses use a combination of in situ and ex situ sample testing. The XT400 comes with Compton Normalization calibration algorithms for soil analysis. Fundamental parameters calibration algorithms are used for metals and alloy analysis. The analyzer can also be empirically calibrated for site-specific analyses. Before performing analytical tests, the type of analysis is selected and the analyzer standardized using a metal clip made of a known alloy that compares a variety of parameters to values stored when the analyzer was calibrated at the factory.

Table 6-1. Innov-X XT400 XRF Analyzer Technical Specifications

Weight:	4.5 pounds (2 kg).
Dimensions:	Hand-held.
Excitation Source:	X-ray tube, silver anode, 10-35 kilovolt (kV), 10-50 microamps (μ A).
Detector:	Si-PiN diode detector, less than 250 electron volts (eV) full width half maximum (FWHM) at 5.95 kiloelectron volts (keV), Manganese K-alpha line.
Software:	Modes include soil, wipe/filter, lead paint, empirical, many others.
Element Range:	Potassium to uranium. With LEAP software, suite of elements can include Cr, Ba, Fe, V, Ti, P, S, Cl, Ca, K.
Number of Elements:	Standard package includes 20 elements. Customer may specify five additional, or use multiple suites of 25 elements each.
Operating Environment:	Temperature: -10 °C to +50 °C.
Operation:	Trigger or Start/Stop Icon for in situ analysis. Optional control from external PC.
Batteries:	Li-ion batteries, rechargeable (charger included). Powers analyzer and iPAQ simultaneously. AC adapter optional.
Battery Life:	Eight hours (typical duty cycle), 3 hours continuous (tube on) operation.
Display:	Color, high-resolution touch screen. Variable brightness provides easy viewing in all ambient lighting conditions.
Data Display:	Concentrations in parts per million (ppm), spectra or peak intensities (count rate) or user-specified units, depending on software mode selected.
Memory, Data Storage:	Minimum 20,000 test results with spectra, upgradeable to 100,000 test results with upgrade to 1 gigabyte (GB) flash card. 128 MB standard memory.
Processor:	Intel 400 megahertz (MHz) StrongArm processor.
Operating System:	Microsoft Windows [®] CE (portable system) or Windows (PC-based).

The Innov-X software allows for visual observance and the identification of spectra. Data from the analyzer can be fully compliant with EPA Method 6200, “Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.” Since XRF analysis is nondestructive, samples analyzed by XRF can be sent to a fixed analytical laboratory for confirmation of results.

Innov-X does not have formal published standard operating procedures for the XT400 but does have an Instruction Manual describing operations for both alloy and soil analysis. Innov-X recommends that users follow EPA Method 6200 and the instruction manual as appropriate protocols for analysis of environmental samples.

The XT400 can be shipped via regular ground or air transportation. The analyzer can be transported on aircraft as carry-on or checked baggage because the x-ray tube emits radiation only while it is operating.

6.2 Instrument Operations during the Demonstration

Innov-X elected to ship the XT400 to the demonstration site using a national shipping carrier. The analyzer and associated analytical supplies were shipped in a padded Pelican case placed inside a cardboard box. Two additional boxes were needed to hold all the accessories and supplies for routine analysis. A laptop PC is not required for analysis, but was used during the field demonstration for data downloading, manipulation, and storage.

6.2.1 Set up and Calibration

The XT400 was set up and operated in the bench-top mode for the demonstration (Figure 6-2). Setting up the XT400 involved removing the triangular testing stand from the box, assembling the stand, and placing it on the table. The analyzer was inserted in the stand and connected to a 110-volt power supply and to the HP iPAQ PDA. The laptop PC was also connected to the 110-volt power supply and turned on.

The standard “Soil” test mode was selected, which uses the preprogrammed Compton Normalization calibration algorithm for soil. The standard LEAP suite of analytes was also included to test for

titanium, barium, and chromium. The standard soil and LEAP suite testing were set up to run sequentially. The instrument was standardized by attaching the alloy standardization clip to the top of the analyzer and analyzing the clip. The standard Soil and LEAP calibration models were used during the demonstration.

6.2.2 Demonstration Sample Processing

Innov-X sent a two-person field team to the demonstration site. These two field team members shared all sample processing tasks in completing the analysis of the demonstration sample set using the XT400.

Sample preparation consisted of filling 32-millimeter sample cups with soil from the sample jar. Mylar® X-ray film was used to cover the top of each sample cup. The cups were filled with soil, and the bottom of the cup was snapped on and labeled with the sample number (see Figure 6-3). Samples were analyzed by placing the prepared sample cup over the analyzer x-ray window, closing the testing stand cover, and pushing the start button. The analyzer was programmed to analyze each sample for 2 minutes for regular soil analysis and then sequentially analyze for 2 additional minutes for the LEAP suite of elements. The analyzer was re-standardized every 15 to 20 samples. At the end of each day, the data from the PDA were transferred to the laptop PC using a comma separated value (CSV) format.



Figure 6-3. Innov-X technician preparing samples for analysis.

Samples were also analyzed on a second XT400 with an experimental 40 kV x-ray tube in the same manner and technique as for the first instrument. Only the analysis and results for three elements were of interest for the second instrument. The 40-kV x-ray tube analyzer was programmed to test each sample for 2 minutes using the standard soil analysis. No LEAP analysis was completed using the second 40-kV analyzer. The 40-kV instrument was also re-standardized every 15 to 20 samples.

Innov-X has not published a formal standard operating procedure (SOP) for the XT400. As a result, the analyzer was operated in accordance with the XT400 Instrument Manual and EPA Method 6200.

6.3 General Demonstration Results

Innov-X analyzed all 326 soil and sediment samples using the regular soil analysis and LEAP soil analysis modules. All samples were analyzed using the 35-kV analyzer set up in the bench-top mode. After each sample was analyzed with the 35-kV analyzer, the

same sample was also analyzed using the experimental 40-kV analyzer. Only three elements (of the 14 demonstration elements) were evaluated with the experimental 40-kV analyzer; the results were not merged with the original data from the 35-kV analyzer. All evaluations of the XT400 relative to the primary and secondary objectives of the demonstration were based on the results obtained using the currently available 35-kV analyzer. The data reported from the 40-kV analyzer is provided for informational purposes only in the Appendixes to this report. .

6.4 Contact Information

Additional information on the Innov-X XT400 Series XRF analyzer is available from the following source:

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Chapter 7

Performance Evaluation

As discussed in Chapter 6, Innov-X analyzed all 326 demonstration samples of soil and sediment at the field demonstration site between January 25 and 28, 2005. The demonstration data set for the 13 target elements was generated on an Innov-X XT400 instrument equipped with a 35-kV x-ray tube. A second data set was generated for three of the target elements (antimony, cadmium, and silver) on an experimental XT400 unit equipped with a 40-kV x-ray tube. Electronic data sets for both instruments were delivered to the Tetra Tech field team in Excel spreadsheet format before Innov-X demobilized from the site on January 28, 2005. All data Innov-X provided at the close of the demonstration are tabulated and compared with the reference laboratory data and the ERA-certified spike concentrations, as applicable, in Appendix D. Table D-1 presents the main demonstration data reported from the 35-kV instrument, and Table D-2 presents the experimental data set reported from the 40-kV instrument.

The data set for the Innov-X XT400 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. The evaluations below focus on the data set for the 35-kV instrument, and do not discuss the 40-kV instrument. However, summary statistics in support of the primary objectives are included for the 40-kV instrument in Appendix E..

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. Innov-X reported the instrument response for each target

element in each sample; in some cases, the instrument response was a negative value. Whether positive or negative, however; the instrument response was used to calculate the MDL.

The MDLs calculated for the Innov-X XT400 with the 35-kV x-ray tube are presented in Table 7-1. As shown, the data for the MDL blends allowed between eight and 12 individual MDLs to be calculated for each target element. (Iron was not included in the MDL evaluation, as was discussed in Section 4.2.1.) Also shown in Table 7-1 are the mean MDLs calculated for each target element, which are classified as follows:

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

Instrument response remained fairly consistent in the MDL blends, with few extremes. Blend 8 from the Wickes Smelter site produced the maximum soil MDLs for antimony, mercury, nickel, selenium, and silver. Blend 8 was a roaster slag matrix that contained high concentrations of other elements, such as arsenic, copper, lead, and zinc. Other instruments participating in the demonstration also showed poor relative instrument performance for this blend. No other trends could be discerned in the calculated MDLs in terms of sample matrix (soil versus sediment) or blend.

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Innov-X XT400¹

Matrix	Blend No.	Antimony			Arsenic			Cadmium			Chromium		
		Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴
Soil	2	47	4	17	17	1	1.5	31	-30	ND	42	265	167
Soil	5	49	-9	ND	5	36	47	41	-25	1.9	51	120	121
Soil	6	45	-4	8	NC	349	477	36	-11	12	54	136	133
Soil	8	104	83	118	NC	8,398	3,943	40	223	91	44	151	55
Soil	10	34	-9	ND	6	28	39	55	-19	0.96	40	147	116
Soil	12	43	99	62	NC	534	559	18	292	263	61	146	101
Soil	18	38	-13	ND	7	9	9	62	-27	ND	113	252	150
Sediment	29	27	-12	ND	6	10	10	45	-32	ND	69	64	63
Sediment	31	31	-3	ND	5	11	11	39	-27	ND	87	237	133
Sediment	32	28	-10	ND	8	25	31	46	-25	ND	31	79	75
Sediment	39	46	-11	ND	9	12	14	36	-24	ND	72	122	102
Sediment	65	44	15	11	NC	213	250	48	18	44	155	444	303
Mean		45			8			41			60		
Matrix	Blend No.	Copper			Lead			Mercury			Nickel		
		Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴
Soil	2	10	32	47	NC	1,023	1,200	7	-1	ND	21	126	83
Soil	5	16	43	49	8	75	78	9	-4	ND	35	35	60
Soil	6	20	144	160	NC	3,910	3,986	10	0	0.83	39	41	70
Soil	8	NC	2,680	1,243	NC	66,981	33,429	58	9	15	100	107	57
Soil	10	10	30	31	10	64	72	11	3	0.14	26	47	60
Soil	12	NC	848	747	NC	4,552	4,214	21	14	1.8	30	98	91
Soil	18	8	46	50	7	16	17	17	71	56	68	246	213
Sediment	29	NC	1,996	1,986	11	42	33	11	0	0.24	55	54	72
Sediment	31	NC	1,561	1,514	12	58	51	18	46	ND	126	292	196
Sediment	32	11	47	36	10	33	26	12	42	ND	32	176	174
Sediment	39	19	104	94	12	44	27	7	56	ND	64	252	202
Sediment	65	16	92	69	57	44	25	32	87	32	89	322	214
Mean		14			16			18			57		

Table 7-1. Evaluation of Sensitivity — Method Detection Limits for the Innov-X XT400¹ (Continued)

Matrix	Blend No.	Selenium			Silver			Vanadium			Zinc		
		Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴	Calc. MDL ²	XT400 Conc. ³	Ref. Lab Conc. ⁴
Soil	2	5	-1	ND	27	-15	ND	9	-0.4	1.2	10	21	24
Soil	5	3	0	ND	33	-53	0.93	23	81	55	26	226	229
Soil	6	9	-4	ND	57	-27	14	16	82	56	NC	784	886
Soil	8	23	-59	ND	129	20	144	26	94	34	NC	11,862	5,657
Soil	10	2	1	ND	27	15	ND	21	78	51	10	94	92
Soil	12	10	15	15	48	59	38	20	77	45	NC	2,560	2,114
Soil	18	3	3	ND	29	-2	ND	33	122	67	12	97	90
Sediment	29	4	1	ND	30	28	ND	19	155	96	32	198	160
Sediment	31	3	6	ND	33	69	6.2	30	123	76	36	172	137
Sediment	32	4	7	4.6	36	13	ND	48	98	57	8	94	69
Sediment	39	3	7	ND	36	16	ND	18	62	38	22	162	137
Sediment	65	6	30	22	22	58	41	14	55	31	NC	2,062	1,843
Mean		6			42			23			19		

Notes:

- Detection limits and concentrations are in milligrams per kilogram (mg/kg), or parts per million (ppm). Innov-X reported the instrument response for each target element in each sample without an instrument lower limit of detection. In some cases, therefore, the instrument response and corresponding concentration were negative values for some low-level samples. Whether positive or negative, however, the concentration as reported by Innov-X was used to calculate the MDL.
 - MDLs calculated from the 12 MDL sample blends in this technology demonstration (in bold typeface for emphasis).
 - This column lists the mean concentration reported for this MDL sample blend by the XT400.
 - This column lists the mean concentration reported for this MDL sample blend by the reference laboratory.
- Calc. Calculated.
Conc. Concentration.
MDL Method detection limit.
NC The MDL was not calculated because reference laboratory concentrations exceeded 250 ppm or an insufficient number of detected concentrations were reported.
ND One or more results for this blend were reported as “Not Detected.” Blends with one or more ND result as reported by the XRF were not used for calculating the MDL for this element.
Ref. Lab. Reference laboratory.

The mean MDLs calculated for the XT400 are compared in Table 7-2 with the mean MDLs for all XRF instruments that participated in the demonstration and with the mean MDLs derived from performance data presented in EPA Method 6200 (EPA 1998e). As shown, the mean MDLs for the XT400 are significantly lower than were calculated from EPA Method 6200 data for all elements. When compared with the average results for all eight XRF instruments that participated in the demonstration, the XT400 exhibited a high relative mean MDL only for nickel. Mean MDLs for the XT400 were less than one-half the all-instrument means for arsenic and lead.

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 adequate for all the target elements and ranged from 22 (silver) to 70 (iron). RPDs between the mean concentrations obtained from the XT400 and the reference laboratory were calculated for each blend that met the criteria for an element. Table 7-3 presents the median RPDs, along with the number of RPD results used to calculate the median, for each target element. These statistics are provided for all demonstration samples as well as for subpopulations grouped by medium (soil versus sediment) and concentration level (Levels 1 through 4, as documented in Table 3-1). Additional summary statistics for the RPDs (minimum, maximum, and mean) are provided in Appendix E (Table E-2).

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

- Very good (median RPD less than 10 percent): cadmium.

Table 7-2. Comparison of XT400 Mean MDLs to All-Instrument Mean MDLs and EPA Method 6200 Data¹

Element	XT400 Mean MDLs ²	All XRF Instrument Mean MDLs ³	EPA Method 6200 Mean Detection Limits ⁴
Antimony	45	61	55 ⁵
Arsenic	8	26	92
Cadmium	41	70	NR
Chromium	60	83	376
Copper	14	23	171
Lead	16	40	78
Mercury	18	23	NR
Nickel	57	50	100 ⁵
Selenium	6	8	NR
Silver	42	42	NR
Vanadium	23	28	NR
Zinc	19	38	89

Notes:

- ¹ Detection limits are in units of milligrams per kilogram (mg/kg), or parts per million (ppm).
 - ² The mean MDLs calculated for this technology demonstration, as presented in Table 7-1.
 - ³ The mean MDLs calculated for all eight XRF instruments that participated in this technology demonstration.
 - ⁴ Mean values calculated from Table 4 of Method 6200 (EPA 1998e, www.epa.gov/sw-846).
 - ⁵ Only one value reported.
- EPA U.S. Environmental Protection Agency.
MDL Method detection limit.
NR Not reported; no MDLs reported for this element.

-
- Good (median RPD between 10 percent and 25 percent): arsenic, copper, lead, selenium, and zinc.
 - Fair (median RPD between 25 percent and 50 percent): antimony, chromium, iron, mercury, nickel, and silver.
 - Poor (median RPD greater than 50 percent): vanadium (54.2 percent).

The median RPD was used for this evaluation because it is less affected by extreme values than is the mean. (The initial evaluation of the RPD populations showed that they were right-skewed or lognormal.) However, the classification of the elements based on accuracy generally stayed the same when the mean rather than the median RPD was used for the evaluation (Table E-2). The ability to evaluate the classification by medium (soil versus sediment) or by concentration range was limited by the variability of the data set.

The only significant difference or trend noted in terms of sample matrix or concentration levels were high median RPDs observed in the Level 3 samples for arsenic (with concentrations that exceeded 2,000 ppm) and Level 1 samples for cadmium (with concentrations between 50 and 500 ppm). At 51.9 percent for arsenic (“poor”) and 31.6 percent for

cadmium (“fair”), these median RPDs were much higher than were observed for other concentration ranges. A smaller effect of this nature was also observed in the Level 2 soil samples for copper (where the mean RPD increased into the “fair” range, at 27.7 percent). In all three of these cases, the median RPDs appeared to be skewed high by the results for sample Blends 8 and 9, which contained high concentrations of a number of target elements (arsenic, lead, copper, zinc, and iron).

Section 5.3.3 discussed how the reference laboratory data for antimony were consistently biased low when compared with the ERA-certified spike concentrations. This effect may be caused by volatilization of the antimony compounds used for spiking, resulting in loss of antimony during the sample digestion process at the reference laboratory. Therefore, Table 7-3 includes a second evaluation of accuracy for antimony, comparing the results from the XT400 with the ERA-certified values. Unlike most of the other XRF instruments that participated in the demonstration, however, use of these values did not improve the RPDs for antimony. The mean RPD for the antimony data set actually increased from 27.0 percent (“fair”) to 92.9 percent (“poor”) when the ERA-certified values were used. The XT400 data displayed a consistent low bias for antimony when compared with the certified spike concentrations.

Table 7-3. Evaluation of Accuracy — Relative Percent Differences versus Reference Laboratory Data for the Innov-X XT400

Matrix	Sample		Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
	Group	Statistic	Ref Lab	ERA Spike												
Soil	Level 1	Number	6	--	152	5	28	16	5	15	6	19	4	2	13	20
		Median	34.1%	--	23.0%	31.6%	50.2%	12.7%	13.0%	20.1%	45.5%	23.7%	7.9%	29.0%	66.0%	11.1%
	Level 2	Number	5	1	4	7	4	8	13	4	7	5	5	3	4	6
		Median	18.6%	79.9%	5.0%	9.9%	42.6%	27.7%	36.9%	17.5%	38.8%	38.5%	4.7%	9.4%	21.5%	14.1%
	Level 3	Number	4	3	4	2	2	2	13	8	2	6	4	5	4	9
		Median	18.8%	90.6%	51.9%	7.8%	24.0%	15.7%	16.5%	12.1%	28.9%	37.6%	9.4%	13.3%	55.6%	19.2%
	Level 4	Number	--	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	--	21.4%	26.0%	--	--	--	--	--	--
	All Soil	Number	15	4	23	14	34	26	38	32	15	30	13	10	21	35
		Median	20.0%	86.2%	23.1%	11.3%	44.9%	15.6%	22.4%	18.9%	40.6%	26.6%	5.7%	19.8%	56.6%	12.2%
Sediment	Level 1	Number	2	2	16	3	13	8	3	16	3	12	5	5	6	19
		Median	55.0%	107.8%	25.5%	6.9%	18.2%	12.0%	36.8%	19.4%	91.8%	17.6%	23.9%	34.5%	47.2%	20.8%
	Level 2	Number	4	4	4	4	3	4	19	4	4	6	4	4	8	5
		Median	44.4%	96.7%	27.5%	10.7%	39.8%	4.2%	31.1%	4.1%	42.5%	37.2%	17.9%	12.8%	47.2%	4.3%
	Level 3	Number	3	3	2	3	3	10	4	3	3	4	3	3	3	4
		Median	15.1%	92.9%	13.3%	5.7%	29.6%	3.1%	48.4%	13.1%	13.1%	35.9%	13.5%	46.0%	64.7%	7.5%
	Level 4	Number	--	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	--	39.5%	--	--	--	--	--	--	--
	All Sediment	Number	9	9	22	10	19	22	32	23	10	22	12	12	17	28
		Median	43.4%	102.7%	22.4%	7.2%	29.6%	5.1%	33.6%	13.1%	42.5%	25.5%	17.9%	32.6%	53.8%	17.2%
All Samples	XT400	Number	24	13	45	24	53	48	70	55	25	52	25	22	38	63
		Median	27.0%	92.9%	23.1%	9.7%	36.9%	11.2%	30.7%	16.0%	40.6%	26.5%	10.6%	27.8%	54.2%	15.8%
All Samples	All XRF Instruments	Number	206	110	320	209	338	363	558	392	192	403	195	177	218	471
		Median	84.3%	70.6%	26.2%	16.7%	26.0%	16.2%	26.0%	21.5%	58.6%	25.4%	16.7%	28.7%	38.3%	19.4%

Notes:

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

-- No samples reported by the reference laboratory in this concentration ranges.

ERA Environmental Resource Associates, Inc.

NC Not calculated.

Number Number of samples appropriate for accuracy evaluation.

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

RPD Relative percent difference.

As an additional comparison, Table 7-3 also presents the overall average of the median RPDs for all eight XRF instruments. Complete summary statistics for the RPDs across all eight XRF instruments are included in Appendix E (Tables E-2 and E-3). Table 7-3 indicates that the median RPDs for the XT400 were equivalent to or below the all-instrument medians for all target elements except chromium, iron, and vanadium. The median RPDs for chromium and iron were close to the all-instrument medians. Table 7-3 also shows that the median RPD for antimony obtained with the XT400 and calculated using the reference laboratory data was significantly lower than the corresponding all-instrument median. Conversely, it shows that the median RPD calculated using the ERA-certified spike value was higher.

In addition to calculating RPDs, the evaluation of accuracy included preparing linear correlation plots. These plots are presented for the individual target

elements in Figures E-1 through E-13 of Appendix E. The plots include a 45-degree line showing the “ideal” relationship between the XT400 data and the reference laboratory data, as well as a “best fit” linear equation ($y = mx + b$, where m is the slope of the line and b is the y-intercept of the line) and correlation coefficient (r^2) to help illustrate the “actual” relationship between the two methods. To be considered accurate, the correlation coefficient should be greater than 0.9, the slope (m) should be between 0.75 and 1.25, and the y-intercept (b) should be relatively (plus or minus the detection limit) close to zero. The summary of the evaluation of correlation in Table 7-4 shows that results for antimony, cadmium, mercury, and selenium met these criteria (these elements are shown in bold highlights). The correlation plot for cadmium is displayed in Figure 7-1 as an example of the correlations obtained for these elements.

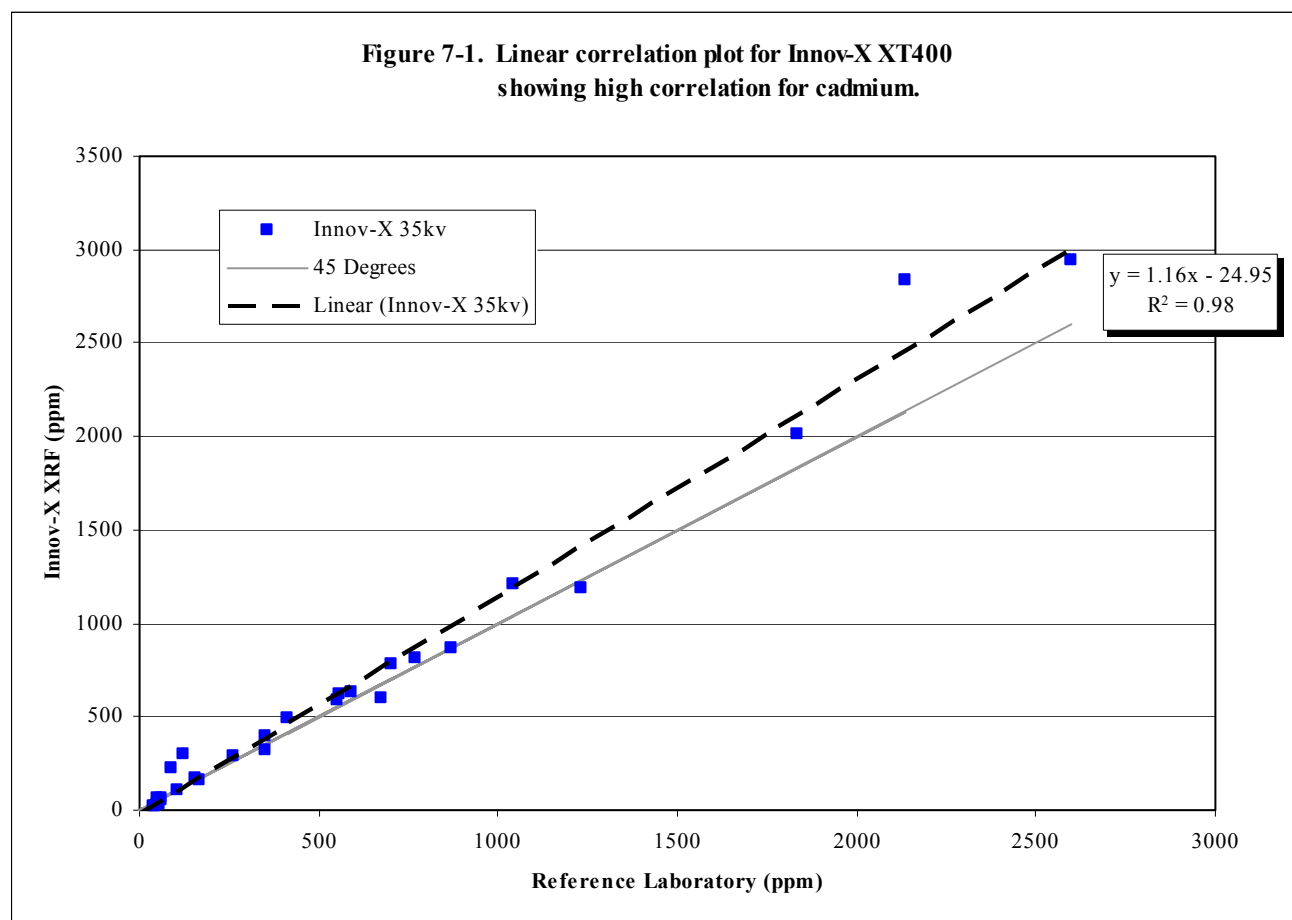


Table 7-4. Summary of Correlation Evaluation for XT400

Target Element	m	b	r ²	Correlation	Bias
Antimony (Ref. Lab) ¹	1.11	7.35 ²	0.92	High	--
Antimony (Cert. Val.) ¹	0.38	-11.64 ²	0.98	High	Low
Arsenic	2.01	-174.16	0.88	Moderate	High
Cadmium	1.16	-24.95 ²	0.98	High	--
Chromium	1.36	17.81 ²	0.97	High	High
Copper	1.09	44.05	0.91	High	--
Iron	1.98	-12,589	0.90	High	High
Lead	1.98	-981.45	0.91	High	High
Mercury	0.82	30.91	0.98	High	--
Nickel	1.48	-25.35 ²	0.98	High	High
Selenium	1.22	0.65 ²	0.99	High	--
Silver	1.03	25.46 ²	0.75	Moderate	High
Vanadium	0.41	73.62	0.39	Low	Variable ³
Zinc	1.61	-294.27	0.82	Moderate	High

Notes:

- ¹ For antimony, correlation was analyzed for the XT400 versus the reference laboratory data ("Ref. Lab") as well as versus the ERA-certified spike values ("Cert. Val.") for the spiked sample blends.
- ² For this element, the absolute value of the y-intercept is below the mean MDL presented in Table 7-1. The accuracy evaluation interpreted this value as meaning that the y-intercept is not significantly different from zero.
- ³ The high intercept and low slope produce a bias that varies with concentration for vanadium. The bias in the results for the XT400 versus the reference laboratory changes from high at low concentrations to low at high concentrations (see Figure E-12).
- No bias observed.
- b Y-intercept of correlation line.
- m Slope of correlation line.
- r² Correlation coefficient of correlation line.

Other general observations from the correlation plots are as follows:

- Correlations for arsenic, lead, and zinc were moderately high, with r² values between 0.82 and 0.91. However, slopes significantly greater than 1 indicated a positive bias in the XRF data for these elements relative to the reference laboratory. Further review of the data indicated that removal of high outliers from complex blends 8 and 9 (Wickes Smelter slag) improved the r² values and eliminated the positive bias for these elements.
- Mercury exhibited a high r² value (0.98). However, this correlation was affected by two extreme Level 4 concentrations (blends 21 and 22) that were more than three times higher than the next-highest concentrations in the data set for mercury (see Figure E-8). Removing these extreme concentrations from the plots produced a much poorer correlation coefficient, in the range of 0.84.
- Large, negative y-intercepts were calculated for arsenic, lead, iron, and zinc. Examination of the

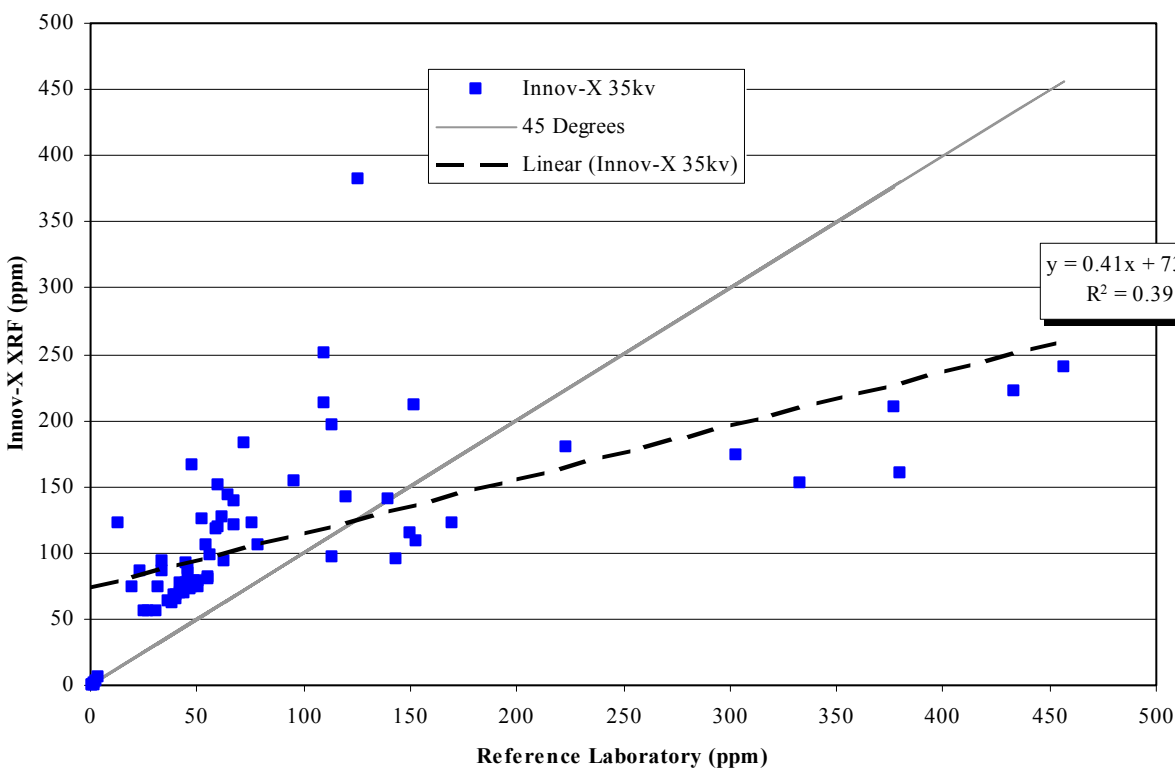
plots for these elements (Figures E-2, E-6 and E-7) reveals that these y-intercepts are small relative to the extreme range of concentrations reported in the demonstration for these elements. In fact, these negative y-intercepts are caused by outliers and non-linearity in the plots at high concentrations that skew the correlation line.

- A high degree of correlation was observed between the data for the XT400 and the reference laboratory for antimony. Table 7-4 and Figure E-1 show a second correlation analysis for antimony, comparing the mean XT400 concentrations for spiked blends with the ERA-certified values. Although a slightly better correlation was observed relative to the ERA-certified values ($r^2 = 0.98$), a low slope of 0.38 indicates a significant low bias in the XT400 data when compared with these values. This observation was consistent with the RPD

evaluation and showed that, unlike many other XRF instruments in the demonstration, comparisons to ERA-certified concentrations did not improve the apparent accuracy of the XT400 data for antimony.

- The lowest degree of correlation between the XT400 and the reference laboratory was observed for vanadium, with an r^2 of 0.39. A slope much less than 1 (0.41), combined with a high intercept, produced a significant positive bias in low-concentration data that changed to a significant negative bias as concentrations increased. This finding agreed with the RPD evaluation, which found poor performance for vanadium. The correlation plot for vanadium is displayed in Figure 7-2.

Figure 7-2. Linear correlation plot for Innov-X XT400 showing low correlation and variable bias for vanadium.



In conclusion, the evaluations of accuracy were similar to the MDL evaluation in Section 7.1 in showing an acceptable overall level of performance by the XT400 for the target elements. Correlations with the reference laboratory were generally high and, for most elements, the median RPDs were better than the average for all eight XRF instruments. Innov-X's proven calibration and quantification algorithms for environmental media may have contributed to the high relative level of accuracy attained. However, the XT400 encountered difficulty in accurately analyzing a few complex slag matrixes and showed poor overall performance for vanadium. In addition, XT400 results for antimony did not agree with the certified spike concentrations in the spiked sample blends, showing a low bias.

7.3 Primary Objective 3 — Precision

As described in Section 4.2.3, the precision of the XT400 instrument 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. Additional summary statistics for the RSDs (including minimum, maximum, and mean) are provided in Appendix E (Table E-4).

The RSD calculation found a high level of precision for the XT400 across all target elements. The ranges into which the median RSDs fell are summarized below:

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

The median RSDs for the full sample set, as well as for the soil and sediment subsets, were less than 5 percent (very low) for eight of the target elements and were between 5 and 10 percent (low) for the remaining five target elements. No significant differences were observed between the RSDs for soil and sediment. Use of the mean as opposed to the median RSDs (Table E-4) indicated a similarly high level of precision in the results from the XT400, although the means for a few elements (antimony and chromium) were slightly above 10 percent. The high overall level of precision may have been facilitated by the level of processing (homogenizing, sieving, crushing, and drying) on the sample blends before the demonstration (Chapter 3). This observation is consistent with the previous SITE MMT program demonstration of XRF technologies that occurred in 1995 (EPA 1996a, 1996b, 1998a, 1998b, 1998c, and 1998d). The high level of sample processing applied during both XRF technology demonstrations was necessary to minimize the effects of sample heterogeneity on the demonstration results and on comparability with the reference laboratories. During project design, site investigation teams that intend to compare XRF and laboratory data should similarly assess the need for sample processing steps to manage sample heterogeneity and improve data comparability.

Further review of the median RSDs in Table 7-5 based on concentration range reveals slightly higher RSDs (in other words, lower precision) for the target elements in Level 1 samples when compared with the rest of the data set. This effect was observed for all the target elements in both soil and sediment, but was greatest for antimony, cadmium and iron. This observation indicates that, to a minor extent, analytical precision for the XT400 results depends on concentration.

As an additional comparison, Table 7-5 also presents the median RSDs calculated for all XRF instruments that participated in the demonstration. Additional summary statistics for the RSDs calculated across all eight XRF instruments are included in Table E-4. Table 7-5 indicates that the median RSDs for the XT400 were equivalent to or below the all-instrument medians for all target elements except silver.

Table 7-5. Evaluation of Precision — Relative Standard Deviations for the Innov-X XT400

Matrix	Sample Group	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	6	15	5	28	16	5	15	6	19	4	2	13	20
		Median	22.1%	6.8%	8.2%	11.8%	7.3%	3.9%	4.7%	7.0%	9.7%	5.0%	10.0%	8.5%	4.8%
	Level 2	Number	5	4	7	4	8	13	4	7	5	5	3	4	6
		Median	6.1%	2.9%	2.0%	3.5%	2.3%	1.4%	1.4%	4.4%	4.4%	2.3%	7.1%	6.0%	2.9%
	Level 3	Number	4	4	2	2	2	13	8	2	6	4	5	4	9
		Median	2.4%	2.4%	2.3%	3.1%	2.6%	1.3%	2.0%	2.1%	2.4%	2.3%	4.9%	4.9%	1.9%
	Level 4	Number	--	--	--	--	--	7	5	--	--	--	--	--	--
		Median	--	--	--	--	--	1.7%	2.1%	--	--	--	--	--	--
Sediment	Level 1	Number	2	16	3	13	8	3	16	3	12	5	5	6	19
		Median	34.1%	5.0%	12.9%	12.6%	6.0%	8.5%	5.0%	7.8%	9.0%	3.2%	11.6%	9.6%	4.3%
	Level 2	Number	4	4	4	3	4	19	4	4	6	4	4	8	5
		Median	5.5%	3.6%	3.1%	6.3%	2.2%	1.8%	2.9%	3.6%	2.6%	1.7%	5.8%	6.7%	3.0%
	Level 3	Number	3	2	3	3	10	4	3	3	4	3	3	3	4
		Median	4.2%	1.1%	3.1%	1.9%	2.3%	1.3%	1.2%	3.1%	2.7%	1.8%	4.4%	3.5%	3.0%
	Level 4	Number	--	--	--	--	--	6	--	--	--	--	--	--	--
		Median	--	--	--	--	--	1.2%	--	--	--	--	--	--	--
All Samples	XT400	Number	24	45	24	53	48	70	55	25	52	25	22	38	63
		Median	6.2%	4.5%	2.8%	9.3%	3.6%	1.5%	3.5%	4.2%	7.4%	2.3%	7.0%	7.1%	3.7%
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 XT400 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	XT400	Number	24	45	24	53	48	70	55	25	52	25	22	38	63
		Median	6.2%	4.5%	2.8%	9.3%	3.6%	1.5%	3.5%	4.2%	7.4%	2.3%	7.0%	7.1%	3.7%
All Samples	All Instruments	Number	206	320	209	338	363	558	392	192	403	195	177	218	471
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%	7.0%	4.5%	5.2%	8.5%	5.3%

Notes:

Number Number of samples appropriate for precision evaluation.
Ref. Lab Reference laboratory
RSD Relative standard deviation

Table 7-6 presents median RSD statistics for the reference laboratory and compares these to the summary data for the XT400. These reference laboratory median RSD statistics were calculated using the same blends as were used in the RSD statistics for the XT400. (Additional summary statistics are provided in Table E-6 of Appendix E.) Table 7-6 indicates that the median RSDs for the XT400 were equivalent to or lower than the RSDs for the reference laboratory for all target elements except chromium. Moreover, the median RSD for chromium using the XT400 was less than 1 percent higher than the median RSD for the reference laboratory. Thus, the XT400 exhibited better precision overall than the reference laboratory on average. In comparison, the median RSDs for all XRF instruments were equivalent to or lower than for the reference laboratory for all target elements except chromium and vanadium.

7.4 Primary Objective 4 — Impact of Chemical and Spectral Interferences

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

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

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

Interferent-to-element ratios were calculated using the mean concentrations the reference laboratory reported for each blend, classified as low (less than 5X), moderate (5 to 10X), or high (greater than 10X). Table 7-7 presents median RPD data for arsenic, nickel, copper, and zinc that are grouped based on this classification scheme. Additional summary statistics are presented in Appendix E (Table E-7).

The table indicates an increase in the median RPD for arsenic at the higher lead-to-arsenic ratios. Specifically, a median RPD of 20.7 percent at low interferent ratios increases to 39.9 percent at high interferent ratios. Using the criteria applied in Section 7.2, high concentrations of lead therefore diminished the accuracy of the XT400 from “good” to “fair” for arsenic. In presenting statistics for the raw RPDs as well as the absolute values of the RPDs, Table E-7 further shows that the interferences by lead tended to produce a more positive bias for the results for arsenic (as indicated by more negative raw RPDs).

Trends in results for copper, nickel, and zinc in response to higher interferent concentrations were difficult to discern due to low numbers of samples with moderate (5 to 10X) interferent-to-element ratios. Although slight increases in RPDs were observed for zinc as copper concentrations increased, the overall accuracy for zinc remained “good” at the highest copper-to-zinc ratios (above 10X) with a median RPD of 21.9 percent.

7.5 Primary Objective 5 — Effects of Soil Characteristics

The population of RPDs between the results obtained from the XT400 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 Appendix E (Table E-8).

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

Table 7-7. Effects of Interferent Elements on the RPDs (Accuracy) for Other Target Elements for the Innov-X XT400¹

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	9	39	5	8	40	1	8	35	2	11	50	3	10
Median RPD of Target Element ²	20.7%	23.0%	39.9%	26.9%	16.6%	28.8%	10.5%	13.4%	17.7%	10.5%	51.7%	14.3%	10.7%	29.9%	21.9%
Median Interferent Concentration	50	8536	2695	99	1106	2515	167	546	3254	197	10790	3692	120	1225	2242
Median Target Element Concentration	86	1006	135	246	198	193	903	89	124	1022	2061	131	570	154	185

Notes:

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

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

< Less than.

> Greater than.

RPD Relative percent difference.

Table 7-8. Effect of Soil Type on the RPDs (Accuracy) for Target Elements, Innov-X XT4000

Matrix	Site	Matrix Description	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead
Soil	AS	Fine to medium sand (steel processing)	Number	--	1	2	2	3	3	3
			Median	--	141.6%	8.8%	43.3%	9.5%	35.1%	10.7%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	5	5	7	6	7	7
			Median	52.2%	5.5%	6.9%	36.0%	17.0%	28.0%	10.9%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	1	2	3	3	3
			Median	18.6%	3.9%	12.2%	69.6%	21.1%	32.4%	33.8%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	1	--	--	4	2	6	6
			Median	127.5%	--	--	43.7%	21.7%	22.7%	13.2%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	4	11	5	7	4	12	6
			Median	36.4%	30.7%	6.9%	30.4%	11.6%	46.6%	28.1%
Sediment	RF	Silty fine sand (tailings)	Number	3	12	5	12	13	13	13
			Median	15.1%	19.0%	7.2%	27.9%	10.5%	27.1%	13.1%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	5	5	1	11	4	12	6
			Median	11.8%	20.7%	12.5%	43.6%	8.8%	14.1%	19.8%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	1	2	1	7	7	4
			Median	64.5%	16.9%	7.0%	56.2%	3.1%	48.6%	12.7%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	7	3	7	6	7	7
			Median	19.1%	38.6%	83.5%	50.6%	29.5%	39.5%	19.1%
	All		Number	24	45	24	53	48	70	55
			Median	27.0%	23.1%	9.7%	36.9%	11.2%	30.7%	16.0%

Table 7-8. Effect of Soil Type on RPDs (Accuracy) of Target Elements, Innov-X XT400 (Continued)

Matrix	Site	Matrix Description	Statistic	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Soil	AS	Fine to medium sand (steel processing)	Number	--	3	1	1	1	3
			Median	--	34.3%	10.6%	6.1%	68.7%	23.6%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	--	5	4	4	4	7
			Median	--	30.9%	5.1%	18.8%	39.5%	17.1%
Soil	CN	Sandy loam (burn pit residue)	Number	2	3	2	2	1	3
			Median	22.3%	22.2%	7.7%	28.2%	33.4%	9.1%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	3	--	--	--	2
			Median	--	34.5%	--	--	--	19.7%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitates)	Number	4	8	5	4	9	10
			Median	33.0%	32.7%	12.2%	27.4%	62.0%	25.8%
Sediment	RF	Silty fine sand (tailings)	Number	5	13	5	5	3	13
			Median	48.0%	24.2%	22.1%	27.5%	40.4%	9.8%
Soil	SB	Coarse sand and gravel (ore and waste rock)	Number	11	11	3	1	10	11
			Median	40.6%	14.6%	5.1%	66.4%	66.4%	7.4%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	3	4	4	7	7
			Median	52.5%	26.7%	16.3%	40.7%	47.0%	18.9%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	3	1	1	3	7
			Median	--	46.8%	13.4%	13.3%	38.2%	32.4%
	All		Number	25	52	25	22	38	63
			Median	40.6%	26.5%	10.6%	27.8%	54.2%	15.8%

Notes:

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

Other notes:

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

Review of Table 7-8 indicates that the median RPDs were highly variable and that trends or differences between sample sites were difficult to discern. Evaluations relative to sampling site were further complicated by the low numbers of samples for many target elements. (Table 7-8 indicates that only 1 to 3 samples were available from many sampling sites for evaluation of specific target elements.) However, high relative median RPDs were observed in the Wickes Smelter blends for a number of elements, including arsenic, cadmium, copper, nickel, and zinc. Evaluation of the outliers in Figure E-14 confirms this observation, indicating that two or more of the highest mean RPDs for these elements are from Wickes Smelter Blends 7, 8, or 9. 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. In addition to arsenic, copper, and zinc, this matrix was found to contain high concentrations of lead and iron. These same Wickes Smelter blends were identified as resulting in outliers for some target elements in the evaluation of accuracy (Section 7.2). Thus, the data presented in Table 7-8 and Figure E-14 confirm that the effects of the complex slag matrixes of Blends 7, 8, and 9 on the accuracy of the XT400 are significant for some elements. RPDs for several target elements are presented for these blends in Table 7-9 below.

High RPD outliers were also observed on a more limited basis in data plots for Blends 55 and 58 from the Leviathan Mine site (Figure E-14). Chapter 2 indicates that the matrices from the Leviathan Mine were clay soils that also included precipitates and solids from acid mine leachate and wastewater

retention ponds. Many of the blends contained extreme concentrations of iron (in the range of 100,000 to 250,000 ppm). Other sampling sites and blends appeared to produce high RPDs for some elements on a more isolated basis, or produced minor increases in mean or median RPDs. These effects appeared to be minor, however, and no other generalized trends in accuracy of the XRF versus the sample collection site or soil and sediment type could be discerned.

7.6 Primary Objective 6 — Sample Throughput

The Innov-X two-person field team was able to analyze all 326 demonstration samples in 5 days at the demonstration site. Once the XT400 instrument had been set up and operations had been streamlined, the Innov-X field team was able to analyze a maximum of 123 samples during an extended work day. This sample throughput was achieved by using the two members of the field team to separately perform sample preparation and instrumental analysis. Without an extended work day, it was estimated that the Innov-X field team would have averaged about 86 samples per day.

This estimated sample throughput for a normal working day was higher than that observed for the other instruments that participated in the demonstration (average of 66 samples per day). The higher sample throughput was primarily the result of the lower-than-average times required to complete each analytical step. A detailed discussion of the time required to complete the various steps of sample analysis using the XT400 is included as part of the labor cost analysis in Section 8.3.

Table 7-9. RPDs Calculated for Wickes Smelter Sample Blends for the Innov-X XT400

Blend	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Zinc
7	31.6%	31.6%	55.4%	37.2%	55.9%	26.0%	37.0%
8	72.2%	83.5%	93.8%	73.3%	92.1%	66.8%	70.8%
9	89.4%	83.5%	107.0%	92.6%	108.6%	88.4%	95.0%
Median for all blends	23.1%	9.7%	36.9%	11.2%	30.7%	16.0%	15.8%

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.

Innov-X recommends that the operator have a high school diploma and basic on-site operational training. Field or laboratory technicians are generally qualified to operate the XT400. One Innov-X staff member who operated the instrument during the demonstration held a Ph.D. in physics, while the other held an M.S. degree in chemistry. Both individuals had multiple years of experience in operating Innov-X XRF analyzers. The skill level of these operators was higher than is required to operate the XT400.

Innov-X has not established standard operating procedures (SOPs) for the analysis of soil or sediment samples using the XT400. However, the instrument is accompanied by a clear and detailed operating manual that presents the general steps in analyzing soil and other media. Instrument software is also helpful in directing users with intuitive operating menus. Innov-X and its distributors offer on-site training and telephone support to instrument users on an informal, as needed basis.

In addition to the general instrument operational instruction and training, the operator and data manager must be familiar with using a PDA and a personal computer (PC) to acquire and manage analytical data obtained from the instrument. The XT400 features direct transfer of analytical results from its iPAQ operating unit to a PC through a hardwired or wireless (Bluetooth) connection, thereby minimizing the potential for lost data.

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 XT400 contains a miniature silver anode x-ray tube. Each instrument is equipped with a fail-safe mechanism (a trigger) to manually control tube operation and analysis. However, the developer reports that risks from exposure to radiation are minimal; direct exposure to the x-rays generated by the instrument for the entire life of the battery would not cause limits on radiation exposure to be exceeded.

The second potential source of risk to XRF instrument operators is exposure to reagent chemicals used in sample preparation. However, for the XT400, there are no risks from this source because no chemical reagents are required for sample preparation.

7.10 Secondary Objective 3 — Portability

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

- (1) The instrument was considered fully portable if the dimensions were such that the instrument could be easily brought directly to the sample location by one person.
- (2) The instrument was considered transportable if the dimensions and power requirements were such that the instrument could be moved to a location near the sampling location, but required a larger and more stable environment (for example, a site trailer with AC power and stable conditions).

Based on its dimensions and power requirements, the XT400 is defined as fully portable. It is a hand-held unit that can be carried directly to the sampling location for analysis of samples. The XT400 is suitable for all types of field analysis, ranging from “point-and-shoot” readings on undisturbed soil surfaces to processed soil samples in plastic bags or sample cups. With an additional instrument stand, the XT400 can also be used in a hands-free, bench-top mode. This instrument stand was used during the demonstration.

7.11 Secondary Objective 4 — Durability

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

The outer construction of the XT400 consists of a high-density plastic and metal shell, which is weatherproof and impact resistant. The iPAQ operating system is attached to the top of the instrument and is held in place with a spring-loaded latch. This connection to the iPAQ constitutes a potential reliability concern, because it could be

compromised by water or dust. The developer recommends placing the instrument inside a large plastic bag when it will be used in wet or dusty conditions. However, this mode of operation was not assessed during the demonstration.

Innov-X provides a 2-year limited warranty for the instrument and x-ray tube. Since x-ray tube sources are new to the world of portable instrumentation, no clear data have been obtained on the useful life that can be assumed. The average lifespan of an x-ray tube in a traditional bench-top device is 3 to 5 years. In comparison, Innov-X estimates that the useful life of an x-ray tube in the XT400 is about 2 to 4 years. The use of a commercially available iPAQ PDA and associated Windows-based Pocket PC software allows for easy upgrades and updates of instrument software and hardware.

7.12 Secondary Objective 5 — Availability

The XT400 is available for purchase or rental from a nationwide network of 20 distributors, and many also can provide on-site training. The instrument can be repaired, maintained, and calibrated by the distributors or at the factory in Woburn, Massachusetts. Innov-X also operates a telephone helpline from 8:00 a.m. to 6:00 p.m. eastern time, Monday through Friday.

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Chapter 8

Economic Analysis

This chapter provides cost information for the Innov-X XT400 XRF analyzer. Cost elements that were addressed include 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 XT400.

8.1 Equipment Costs

Capital equipment costs include either purchase or rental of the XT400 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 Innov-X.

The XT400 used at the demonstration costs \$32,500. The cost includes peripherals such as the Hewlett Packard iPAQ PDA-based operating system, instrument stand, and 110-volt adapter. A laptop computer (not included with instrument) is also recommended to manipulate the data. The instrument is available for rental by Innov-X or by field equipment rental companies. Purchased models include a 2-year parts and labor warranty. The lifespan of the x-ray tube is about 2,000 hours of operation or 2 to 4 years for normal usage.

The purchase price, rental cost, and shipping cost for the XT400 compares favorably with the average costs for all XRF instruments that participated in the demonstration, as shown in Table 8-1. Purchase of the instrument could be justified as more cost effective than rental only for field activities that involve more than about 6 months of total field analysis time.

8.2 Supply Costs

The supplies that were included in the cost estimate include sample containers, Mylar[®] film, spatulas or

Table 8-1. Equipment Costs

Cost Element	Innov-X XT400	XRF Demonstration Average ¹
Shipping	\$200	\$410
Capital Cost (Purchase)	\$32,500	\$54,300
Weekly Rental	\$2,000	\$2,813
Autosampler (for Overnight Analysis)	N/A	N/A

Notes:

¹ Average for all eight instruments in the demonstration

N/A Not available or not applicable for this comparison

scoops, wipes, and disposable gloves. The rate of consumption for these supplies was based on observations during the field demonstration. Unit prices for these supplies were based on price quotes from independent vendors of field equipment.

The XT400 was operated over 4 working days to complete the analysis of all 326 samples during the field demonstration. The supplies required to process samples were similar for all XRF instruments that participated in the demonstration and were estimated to cost about \$245 for 326 samples or \$0.75 per sample.

8.3 Labor Costs

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

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

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

The estimated time required to complete each of these activities using the XT400 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 XT400 is compared with the average of all XRF instruments in Table 8-2 and with the range of all XRF instruments in Figure 8-1. In comparison to other XRF analyzers, the XT400 exhibited lower-than-average times for all activities except for daily shutdown and startup.

The Innov-X field team expended about 45 labor hours to complete all sample processing activities during the field demonstration using the XT400. This was significantly lower than the overall average of 69 labor hours for all instruments that participated in the demonstration. The labor hours were lower for the XT400 because essentially all analytical activities took less time using the XT400, as shown in Figure 8-1.

8.4 Comparison of XRF Analysis and Reference Laboratory Costs

Two scenarios were evaluated to compare the cost for XRF analysis using the XT400 with the cost of fixed-laboratory analysis using the reference methods. Both scenarios assumed that 326 samples were to be analyzed, as in the field demonstration. The first scenario assumed that only one element was to be measured in a metal-specific project or application (for example, lead in soil, paint, or other solids) for comparison to laboratory per-metal unit costs. The second scenario assumed that 13 elements were to be analyzed, as in the field demonstration, for comparison to laboratory costs for a full suite of metals.

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

Table 8-2. Time Required to Complete Analytical Activities¹

Activity	XT400	Average ²
Initial Setup and Calibration	10	54
Sample Preparation	1.2	3.1
Sample Analysis	4.6	6.7
Daily Shutdown/Startup	10	10
End of Project Packing	8	43
Total Processing Time per Sample	5.9	10.0

Notes:

¹ All estimates are in minutes

² Average for all eight XRF instruments in the demonstration

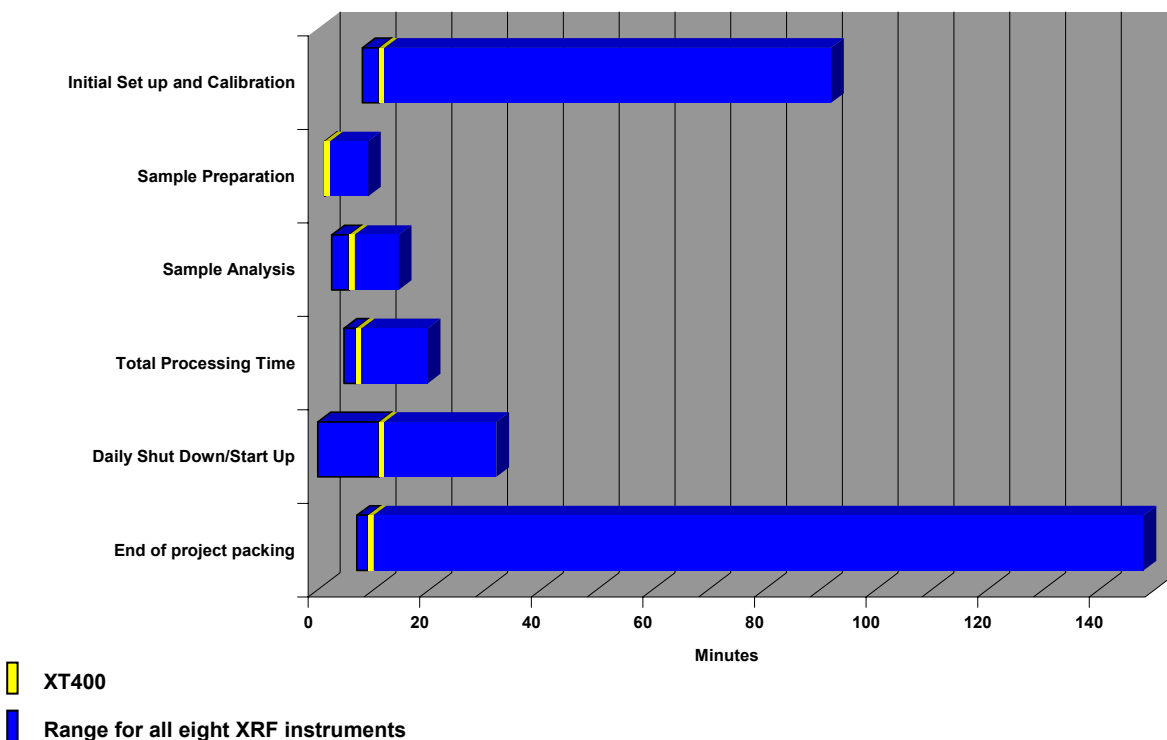


Figure 8-1. Comparison of activity times for the XT400 versus other XRF instruments.

The cost for XRF analysis using the XT400 was based on equipment rental for 1 week, along with labor and supplies estimates from the field demonstration. Labor costs were added for drying, grinding, and homogenizing the samples (estimated at 10 minutes per sample) since these additional steps in sample preparation are required for XRF analysis but not for analysis in a fixed laboratory. A typical cost for managing investigation-derived waste (IDW), including general trash, personal protective equipment, wipes, and soil, was also added to the cost of XRF analysis because IDW costs are included in the unit cost for fixed-laboratory analysis. The IDW management cost was fixed, based on the average IDW disposal cost per instrument during the demonstration, because IDW generation did not vary significantly between instruments during the demonstration. Since the cost for XRF analysis of one element or multiple elements does not vary significantly (all target elements are determined simultaneously when a sample is analyzed), the XRF analysis cost was not adjusted for one element versus 13 elements.

Table 8-3 compares the costs for the XT400 versus the cost for analysis in a fixed laboratory. As shown, the XT400 compares favorably to a fixed laboratory in terms of overall cost, particularly when a large number of elements are to be determined. Use of the XT400 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 XT400 in the example scenario (326 samples) was estimated at \$6,898, 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, which typically cost more than hand-held instruments like the XT400, were included in the calculation of the average cost for all XRF instruments. In comparison to other hand-held XRF instruments, the XT400 cost for the example scenario was similar.

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

Analytical Approach	Quantity	Item	Unit Rate	Total
Innov-X Systems XT400 (1 to 13 elements)				
Shipping	1	Roundtrip	\$200	\$200
Weekly Rental	1	Week	\$2,000	\$2,000
Supplies	326	Sample	\$0.75	\$245
Labor	100	Hours	\$43.75	\$4,363
IDW	N/A	N/A	N/A	\$90
Total XT400 Analysis Cost (1 to 13 elements)				\$6,898
Fixed Laboratory (1 element)				
(EPA Method 6010, ICP-AES)	326	Sample	\$21	\$6,846
Total Fixed Laboratory Costs (1 element)				\$6,846
Fixed Laboratory (13 elements)				
Mercury (EPA Method 7471, CVAA)	326	Sample	\$36	\$11,736
All other Elements (EPA Method 6010, ICP-AES)	326	Sample	\$160	\$52,160
Total Fixed Laboratory Costs (13 elements)				\$63,896

Chapter 9

Summary of Technology Performance

The preceding chapters of this report document that the evaluation design succeeded in providing detailed performance data for the Innov-X XT400 XRF analyzer. The evaluation design incorporated 13 target elements, 70 distinct sample blends, and a total of 326 samples. The blends included both soil and sediment samples from nine sampling locations. A rigorous program of sample preparation and characterization, reference laboratory analysis, QA/QC oversight, and data reduction supported the evaluation of XRF instrument performance.

One important aspect of the demonstration was the sample blending and processing procedures (including drying, sieving, grinding, and homogenization) that significantly reduced uncertainties associated with the demonstration sample set. These procedures minimized the impacts of heterogeneity on method precision and on the comparability between data obtained by the XRF and reference laboratory. 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 calibration protocols, in accordance with EPA Method 6200, that adjust instrument algorithms to compensate for matrix effects.

The findings of the evaluation of the XT400 analyzer for each primary and secondary objective are summarized in Tables 9-1 and 9-2. The XT400 and the combined performance of all eight instruments that participated in the XRF technology demonstration are compared in Figure 9-1. Figure 9-1 indicates that, when compared with the mean performance of all eight XRF instruments, the XT400 showed:

- Equivalent or better MDLs for all target elements except nickel (iron was not included in the MDL evaluation).
- Better accuracy (lower RPDs) for 10 of the 13 target elements (exceptions include chromium, iron, and vanadium). However, when RPDs for antimony are calculated versus sample spike levels rather than reference laboratory data (which may be biased low), accuracy for antimony is lower than for the program as whole.
- Equivalent or better precision (lower RSDs) for all target elements except silver.

As a hand-held instrument, the XT400 is fully portable and can be operated in the hand-held mode at a sampling site. The reasons for the better-than-average sensitivity, accuracy, and precision are not known with any certainty but may relate to Innov-X's proven calibration protocols and quantification algorithms for environmental applications.

Table 9-1. Summary of Innov-X XT400 Performance – Primary Objectives

Objective	Performance Summary
P1: Method Detection Limits	<ul style="list-style-type: none"> • Mean MDLs for the target elements ranged as follows: <ul style="list-style-type: none"> ○ MDLs of 1 to 20 ppm: arsenic, copper, lead, mercury, selenium, and zinc. ○ MDLs of 20 to 50 ppm: antimony, cadmium, silver, and vanadium. ○ MDLs of 50 to 100 ppm: chromium and nickel. ○ MDLs greater than 100 ppm: none. (Iron was not included in the MDL evaluation.) • Blend of roaster slag from the Wickes Smelter site produced the highest MDLs for many target elements, ranging above 100 ppm for antimony, nickel, and silver. • For all the target elements, the MDLs calculated for the XT400 were significantly lower than reference MDL data from EPA Method 6200.
P2: Accuracy and Comparability	<ul style="list-style-type: none"> • Median RPDs between the XRF and reference laboratory data revealed the following, with lower RPDs indicating greater accuracy: <ul style="list-style-type: none"> ○ RPDs less than 10 percent: cadmium. ○ RPDs of 10 to 25 percent: arsenic, copper, lead, selenium, and zinc. ○ RPDs of 25 to 50 percent: antimony, chromium, iron, mercury, nickel, and silver. ○ RPDs greater than 50 percent: vanadium (54.2 percent). • Data review indicated that the reference laboratory results for some spiked demonstration samples may be biased low for antimony due to the volatility of the spiking compounds used. RPDs for antimony were moderate when the XT400 data were compared with the reference laboratory data (with a median RPD of 27 percent) but increased considerably when compared with certified spike values (where the median RPD was 92.9 percent). Thus, unlike other instruments in the XRF technology demonstration, comparison to the spike concentrations did not improve the apparent accuracy of the XT400 for antimony. • Correlation plots relative to reference laboratory data indicated: <ul style="list-style-type: none"> ○ High correlation coefficients (greater than 0.9 for 9 of the 13 target elements). ○ Low to moderate correlation coefficients for arsenic, silver, vanadium, and zinc. The correlations for arsenic and zinc were reduced by extreme values again associated with the roaster slag matrix. ○ High biases in the XRF data versus the laboratory data for seven of the 13 elements; for vanadium, a bias that changed from high to low as concentrations increased.
P3: Precision	<ul style="list-style-type: none"> • Median RSDs were good for all elements, as follows: <ul style="list-style-type: none"> ○ RSDs were 0 to 5 percent for arsenic, cadmium, copper, iron, lead, mercury, selenium, and zinc. ○ RSDs were between 5 and 10 percent for antimony, chromium, nickel, silver, and vanadium. • RSDs were higher (that is, precision was lower) in the lowest- concentration sample blends for all of the target elements, with the greatest effects seen for antimony, cadmium, and iron. • For all target elements except chromium, median RSDs for the XT400 were lower than the RSDs calculated for the reference laboratory data, indicating better precision for the XT400 instrument.

Table 9-1. Summary of Innov-X XT400 Performance – Primary Objectives (continued)

Objective	Performance Summary
P4: Effects of Sample Interferences	<ul style="list-style-type: none"> • High relative concentrations of lead slightly reduced accuracy for arsenic; median RPDs for arsenic increased from 21 percent to 40 percent as the concentration of lead increased. • No significant trends in the results for copper, nickel, or zinc were observed in response to potential interferent concentrations.
P5: Effects of Soil Type	<ul style="list-style-type: none"> • As was initially noted in the MDL and accuracy evaluations, low relative accuracy was observed for multiple elements in blends of roaster slag from the Wickes Smelter site, which contained high overall element concentrations. • High RPD outliers were also observed on a more limited basis in soils from the Leviathan Mine site, which were impacted by high-iron precipitates and solids from acid mine leachate. • Evaluation of RPD values based on the different soil and sediment sampling sites revealed no other significant matrix effects or outliers.
P6: Sample Throughput	<ul style="list-style-type: none"> • With an average sample preparation time of 1.2 minutes and an instrument analysis time of 4.6 minutes per sample, the total sample processing time was 5.9 minutes per sample. • A maximum sample throughput of 123 samples per day was achieved during an extended work day. A more typical sample throughput was estimated to be 86 samples per day for an 8-hour work day.
P7: Costs	<ul style="list-style-type: none"> • Instrument purchase cost is \$32,500 with a weekly rental cost of \$2,000. These costs are for the instrument equipped as applied in the demonstration, including the Hewlett Packard iPAQ PDA-based operating system, instrument stand, and 110-volt AC adapter. • The Innov-X field team expended approximately 45 labor hours to complete the processing of the demonstration sample set (326 samples). In comparison, the average for all participating XRF instruments was 69 labor hours. • By adding labor and shipping/supplies costs to the 1-week instrument rental cost, a total project cost of \$6,898 was estimated for a project the size of the demonstration using the XT400. In comparison, the average project cost for all participating XRF instruments was \$8,932 and the cost for fixed-laboratory analysis of all 13 elements was \$63,896.

Table 9-2. Summary of Innov-X XT400 Performance – Secondary Objectives

Objective	Performance Summary
S1: Training Requirements	<ul style="list-style-type: none"> Field or laboratory technicians with a high school diploma and basic operational training are generally qualified to operate the XT400. Innov-X and its distributors offer on-site training on an informal, as needed bases, and provide telephone support through a toll-free number.
S2: Health and Safety	<ul style="list-style-type: none"> The XT400 has a manual trigger that must be engaged and held to operate the x-ray tube and analyze a sample. Further, Innov-X states that direct exposure to x-ray tube emissions for the entire life of the battery would not exceed exposure limits. No chemicals are used during sample preparation or analysis that would pose potential hazards.
S3: Portability	<ul style="list-style-type: none"> Based on dimensions, weight, and power requirements, the XT400 is a fully portable instrument. It can be used as a hand-held unit to analyze undisturbed soil or bagged samples. With an additional instrument stand, the XT400 can be used in a hands-free, bench-top mode.
S4: Durability	<ul style="list-style-type: none"> The XT400 has a 2-year limited warranty that includes the x-ray tube. The expected lifespan of the x-ray tube is 2-4 years. The instrument is encased in durable hard-tool plastic and metal, and is largely weatherproof and impact-resistant. However, the demonstration found that the connection between the instrument and its iPAQ operating unit could be compromised by water or dust. Innov-X recommends placing the instrument inside a large plastic bag when it will be used in wet or dusty conditions.
S5: Availability	<ul style="list-style-type: none"> The XT400 is available for purchase or rental from a nationwide network of 20 distributors. Instrument repairs, maintenance, and calibration can be performed by the distributors or at the factory in Woburn, Massachusetts.

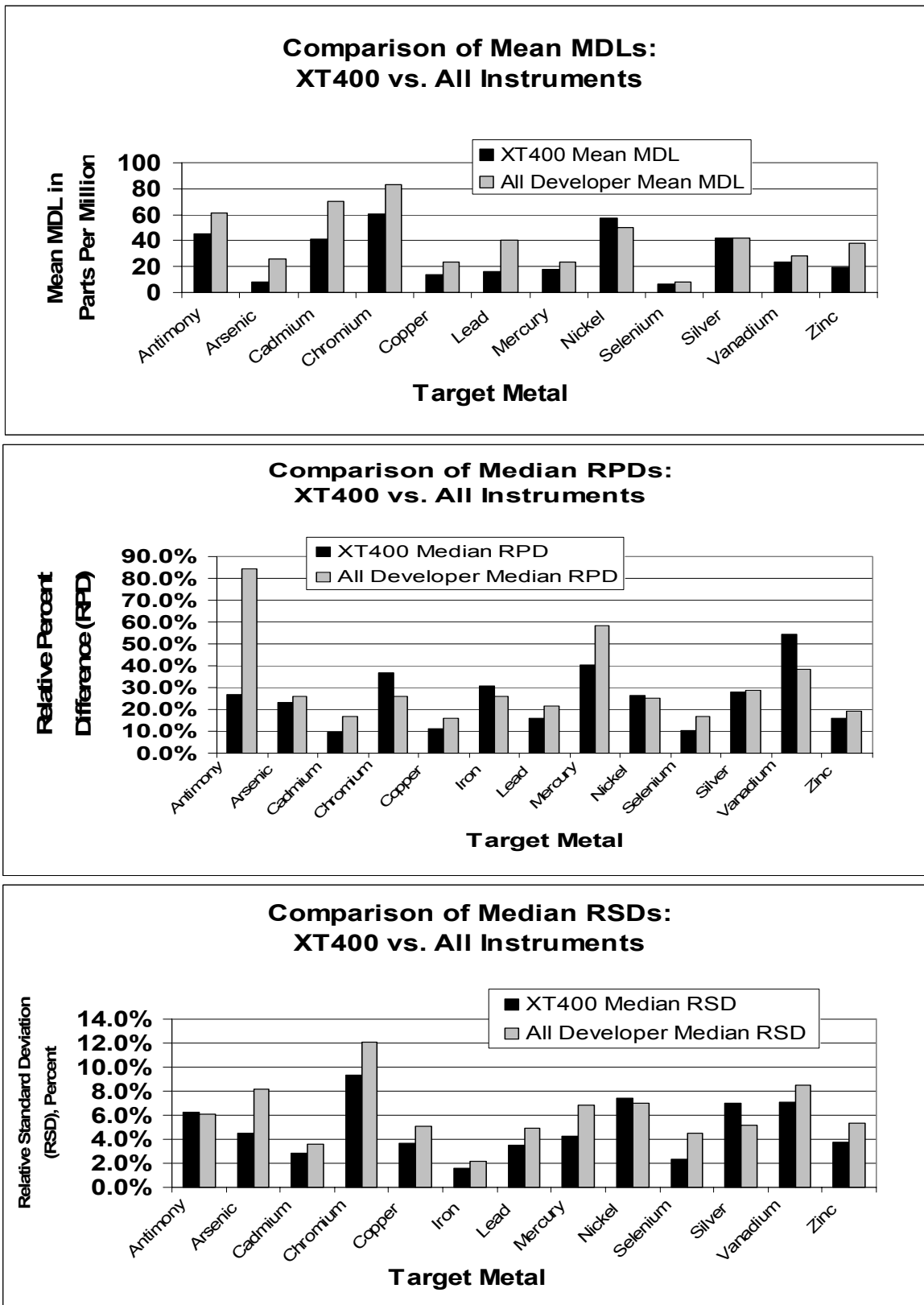


Figure 9-1. Method detection limits (sensitivity), accuracy, and precision of the XT400 in comparison to the average of all eight XRF instruments.

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Chapter 10

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APPENDIX A

VERIFICATION STATEMENT

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, DC 20460



SITE Monitoring and Measurement Technology Program Verification Statement

TECHNOLOGY TYPE:	X-ray Fluorescence (XRF) Analyzer
APPLICATION:	MEASUREMENT OF TRACE ELEMENTS IN SOIL AND SEDIMENT
TECHNOLOGY NAME:	XT400 XRF Analyzer
COMPANY:	Innov-X Systems, Inc.
ADDRESS:	10 Gill Street, Suite Q Woburn, Massachusetts 01801
PHONE:	781-938-5005
WEB SITE:	www.innov-xsys.com
E-MAIL:	info@innov-xsys.com

VERIFICATION PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Superfund Innovative Technology Evaluation (SITE) Monitoring and Measurement Technology (MMT) Program to facilitate deployment of innovative technologies through performance verification and information dissemination. The goal of this program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The program assists and informs those involved in designing, distributing, permitting, and purchasing environmental technologies. This document summarizes the results of a demonstration of the Innov-X Systems, Inc., XT400 portable x-ray fluorescence (XRF) analyzer for the analysis of 13 target elements in soil and sediment, including antimony, arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, vanadium, and zinc.

PROGRAM OPERATION

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

DEMONSTRATION DESCRIPTION

The field demonstration of eight XRF instruments to measure trace elements in soil and sediment was conducted from January 24 through 28, 2005, at the Kennedy Athletic, Recreational and Social (KARS) Park, which is part of the Kennedy Space Center on Merritt Island, Florida. A total of 326 samples were analyzed by each XRF instrument, including the Innov-X XT400, 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 Innov-X XT400 XRF instrument. More detailed discussion can be found in the *Innovative Technology Verification Report – XRF Technologies for Measuring Trace Elements in Soil and Sediment: Innov-X XT400 XRF Analyzer* (EPA/540/R-06/002).

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 Innov-X XT400 hand-held XRF analyzer features a miniature, rugged x-ray tube excitation source for analyzing a wide variety of elements and sample materials, including alloys, environmental solids, and other samples. The combined x-ray tube source and Light Element Analysis Program (LEAP) technology analyzes elements that would require three isotope sources in an isotope-based XRF analyzer. Other features of the XT400 include multiple x-ray beam filters, multiple calibration methods (fundamental parameters, Compton normalization, empirical, scatter normalization, and spectral matching), and adjustable tube voltages and currents.

The XT400 can be powered in the field with a lithium-ion battery or 110-volt alternating current (AC). It utilizes a removable Hewlett-Packard (HP) iPAQ personal data assistant (PDA) for data storage of up to 10,000 tests with spectra in its 64 MB memory. The iPAQ has a color, high resolution display and can be fitted with Bluetooth[®] wireless printing and data downloading, an integrated bar-code reader, and wireless data and file transfer accessories. The XT400 can analyze elements from potassium (atomic number [Z] = 19) to uranium (Z = 92) in suites of 25 elements simultaneously.

VERIFICATION OF PERFORMANCE

Method Detection Limit: MDLs were calculated using seven replicate analyses from each of 12 low-concentration sample blends, according to the procedure described in Title 40 Code of Federal Regulations (CFR) Part 136, Appendix B, Revision 1.11. A mean MDL was further calculated for each element. The ranges into which the mean MDLs fell for the XT400 are listed below (lower MDL values indicate higher sensitivity).

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

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

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

XRF and the mean reference laboratory concentration for each blend. Accuracy of the XT400 was classified from high to very low for the various target elements, as indicated in the table below, based on the overall median RPDs calculated for the demonstration.

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

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

Accuracy was also assessed through correlation plots between the mean XT400 and mean reference laboratory concentrations for the various sample blends. Correlation coefficients (r^2) for linear regression analysis of the plots are summarized below, along with any significant biases apparent from the plots in the XRF data versus the reference laboratory data.

	Antimony*	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver	Vanadium	Zinc
Correlation	0.92	0.88	0.98	0.97	0.91	0.90	0.91	0.98	0.98	0.99	0.75	0.39	0.82
Bias	--	High	--	High	--	High	High	--	High	--	High	Var.	High

Note: -- = No significant bias. Var. = Bias varies from high to low as concentration increases.

* Correlation is 0.98 and bias is low when assessed versus sample spike concentrations.

Precision: Replicates were analyzed for all sample blends. Precision was evaluated by calculating the standard deviation of the replicates, dividing by the average concentration of the replicates, and multiplying by 100 percent to yield the relative standard deviation (RSD) for each blend. Precision of the XT400 was classified from high to very low for each target element, as indicated in the table below, based on the overall median RSDs. These results indicated a higher level of precision in the XT400 data than in the reference laboratory data for all target elements except chromium.

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

Effects of Interferences: The RPDs from the evaluation of accuracy were further grouped and compared for a few elements of concern (arsenic, nickel, copper, and zinc) based on the relative concentrations of potentially interfering elements. Accuracy for arsenic was reduced from “moderate” (median RPDs less than 25 percent) to “low” (median RPDs between 25 and 50 percent) by high relative concentrations of lead (greater than 10X the arsenic concentration). However, no significant trends in the results for copper, nickel, or zinc results were observed in response to potential interferent concentrations.

Effects of Soil Characteristics: The RPDs from the evaluation of accuracy were also further evaluated in terms of sampling site and soil type. This evaluation found outlier RPD values, indicating low relative accuracy, for multiple elements in blends of roaster slag from the Wickes Smelter site. These blends contained high overall element concentrations.

Sample Throughput: The total processing time per sample was estimated at 5.9 minutes, which included 1.2 minutes of sample preparation and 4.6 minutes of instrument analysis time. A sample throughput of 86

samples per 8-hour work day was estimated using a two-person field team. As noted above, however, the sample blends had undergone rigorous pre-processing before the demonstration. Sample throughput would have decreased if these sample preparation steps (grinding, drying, sieving) had been performed during the demonstration; these steps can add from 10 minutes to 2 hours to the sample processing time.

Costs: A cost assessment for the XT400 identified a purchase cost of \$32,500 and a weekly rental cost of \$2,000, plus \$200 shipping, as equipped for the demonstration. A total cost of \$6,898 (with a labor cost of \$4,363 at \$43.75/hr) associated with sample preparation and analysis was estimated for a project similar to the demonstration (326 samples of soil and sediment). In comparison, the project cost averaged \$8,932 for all eight XRF instruments participating in the demonstration, and \$63,896 for fixed-laboratory analysis of all samples for the 13 target elements.

Skills and Training Required: Field or laboratory technicians with a high school diploma are generally qualified to operate the XT400. Innov-X and its distributors offer on-site training on an informal, as-needed basis, and provide telephone support through a toll-free number.

Health and Safety Aspects: The manual trigger on the XT400 must be engaged and held to operate the x-ray tube and analyze a sample. Further, Innov-X states that direct exposure to x-ray tube emissions for the entire life of the battery would not exceed exposure limits. No chemicals are used during sample preparation or analysis that would pose potential hazards.

Portability: Based on dimensions, weight (4.5 pounds), and power requirements, the XT400 is a fully portable instrument. It can be used as a hand-held unit to analyze undisturbed soil or bagged samples. With the instrument stand, the XT400 can be used in a hands-free, bench-top mode.

Durability: The 2-year limited warranty on the XT400 includes the x-ray tube. The expected lifespan of an x-ray tube is 2 to 4 years. The instrument is encased in durable hard-tool plastic and metal. However, Innov-X recommends placing the instrument inside a large plastic bag when it will be used in wet or dusty conditions so that the connection between the instrument and its iPAQ PDA is not compromised.

Availability: The XT400 can be purchased, rented, and serviced from the factory in Woburn, Massachusetts, or through a nationwide network of 20 distributors.

RELATIVE PERFORMANCE

The overall performance of the XT400 analyzer relative to the average of all eight XRF instruments that participated in the demonstration is shown below:

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

Key: ● Better ○ Worse

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

APPENDIX B

DEVELOPER DISCUSSION

DEVELOPER DISCUSSION

The data obtained in these trials has allowed Innov-X to make improvements to the calibration procedure. The most noted improvement is in Chromium performance. Detection limits and precision for Cr are expected to be better for current production units than was demonstrated in this study.

Further improvements to peak deconvolution algorithms are expected in the near future which should have a positive impact on the Ni performance.

Since this study encompassed a large range of samples, it is possible to get improved accuracy for a specific site if known standards are available. Customers are able to fine tune calibrations for a specific matrix using type specific standards.

APPENDIX C

DATA VALIDATION SUMMARY REPORT

Contents

<u>Chapter</u>	<u>Page</u>
Acronyms, Abbreviations, and Symbols	iii
1.0 INTRODUCTION	C-1
2.0 VALIDATION METHODOLOGY	C-1
3.0 DATA VALIDATION	C-3
3.1 Holding Time	C-3
3.2 Calibration	C-3
3.3 Laboratory Blanks	C-4
3.4 Laboratory Control Samples	C-5
3.5 Matrix Spike Samples	C-5
3.6 Serial Dilution Results	C-5
3.7 ICP Interference Check Samples	C-6
3.8 Target Analyte Identification and Quantitation	C-6
3.9 Quantitation Limit Verification	C-6
4.0 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY EVALUATION SUMMARY	C-6
4.1 Precision	C-7
4.2 Accuracy	C-7
4.3 Representativeness	C-7
4.4 Completeness	C-7
4.5 Comparability	C-7
5.0 CONCLUSIONS FOR DATA QUALITY AND DATA USABILITY	C-8
6.0 REFERENCES	C-8

APPENDIX

DATA VALIDATION REPORTS

ABBREVIATIONS AND ACRONYMS

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

1.0 INTRODUCTION

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

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

2.0 VALIDATION METHODOLOGY

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

- “USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review” (EPA 2004), hereinafter referred to as the “EPA guidance.”
- “Demonstration and Quality Assurance Project Plan, XRF Technologies for Measuring Trace Elements in Soil and Sediment” (Tetra Tech 2005), hereinafter referred to as “the QAPP.”

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

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

- Holding times

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

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

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

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

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

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

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

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

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

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

3.0 DATA VALIDATION RESULTS

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

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

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

3.1 Holding Time

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

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

3.2 Calibration

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

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

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

3.3 Laboratory Blanks

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

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

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

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

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

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

3.4 Laboratory Control Samples

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

3.5 Matrix Spike Samples

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

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

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

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

3.6 Serial Dilution Results

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

3.7 ICP Interference Check Samples

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

3.8 Target Analyte Identification and Quantitation

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

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

3.9 Quantitation Limit Verification

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

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

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

4.1 Precision

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

4.2 Accuracy

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

4.3 Representativeness

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

4.4 Completeness

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

4.5 Comparability

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

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

5.0 CONCLUSIONS FOR DATA QUALITY AND DATA USABILITY

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

6.0 REFERENCES

- Tetra Tech EM, Inc. (Tetra Tech). 2005. "Demonstration and Quality Assurance Project Plan, XRF Technologies for Measuring Trace Elements in Soil and Sediment." March.
- U.S. Environmental Protection Agency (EPA). 1996. "Test Methods for Evaluating Solid Waste", Third Edition (SW-846). With promulgated revisions. December.
- EPA. 2004. "USEPA Contract Laboratory Program National Functional Guidelines For Inorganic Data Review". October.

TABLES

TABLE 1: DATA VALIDATION QUALIFIERS AND COMMENT CODES

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

TABLE 2: QC CRITERIA

Parameter	Method	QC Check	Frequency	Criterion	Corrective Action
Reference Method					
Target Metals (12 ICP metals and Hg)	3050B/6010B and 7471A	Method and instrument blanks	One per analytical batch of 20 or less	Less than the reporting limit	<ol style="list-style-type: none"> 1. Check calculations 2. Assess and eliminate source of contamination 3. Reanalyze blank 4. Inform Tetra Tech project manager 5. Flag affected results
		MS/MSD	One per analytical batch of 20 or less	75 to 125 percent recovery $RPD \leq 25$	<ol style="list-style-type: none"> 1. Check calculations 2. Check LCS/LCSD and digest duplicate results to determine whether they meet criterion 3. Inform Tetra Tech project manager 4. Flag affected results
		LCS/LCSD	One per analytical batch of 20 or less	80 to 120 percent recovery $RPD \leq 20$	<ol style="list-style-type: none"> 1. Check calculations 2. Check instrument operating conditions and adjust as necessary 3. Check MS/MSD and digest duplicate results to determine whether they meet criterion 4. Inform Tetra Tech project manager 5. Redigest and reanalyze the entire batch of samples 6. Flag affected results
		Performance audit samples	One per analytical batch of 20 or less	Within acceptance limits	<ol style="list-style-type: none"> 1. Evaluated by Tetra Tech QA chemist 2. Inform laboratory and recommend changes 3. Flag affected results
Percent moisture		Laboratory duplicates	One per analytical batch of 20 or less	$RPD \leq 20$	<ol style="list-style-type: none"> 1. Check calculations 2. Reanalyze sample batch 3. Inform Tetra Tech project manager 4. Flag affected results

TABLE 3: DATA QUALIFICATION: LABORATORY METHOD BLANK CONTAMINATION

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

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

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

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

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

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

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

Notes:

mg/kg = Milligrams per kilogram

b = Data were qualified based on blank contamination

e = Data were additionally qualified based on matrix spike/matrix spike duplicate exceedances

J+ = Result is estimated and potentially biased high

J- = Result is estimated and potentially biased low

UJ = Result is undetected at estimated quantitation limits

TABLE 4: DATA QUALIFICATION: MATRIX SPIKE RECOVERY EXCEEDANCES

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

**TABLE 4: DATA QUALIFICATION: 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 QUALIFICATION: SERIAL DILUTION EXCEEDANCES
(Continued)**

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

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

Sample ID	Analyte	Result	Unit	Validation Qualifier	Comment Code
SB-SO-15-XX	Iron	51000	mg/kg	J-	j
SB-SO-15-XX	Lead	40	mg/kg	J-	j
SB-SO-15-XX	Nickel	100	mg/kg	J-	j
SB-SO-15-XX	Vanadium	52	mg/kg	J-	j
SB-SO-15-XX	Zinc	36	mg/kg	J-	j
SB-SO-22-XX	Antimony	10	mg/kg	J	e, j
SB-SO-22-XX	Zinc	64	mg/kg	J-	j
SB-SO-31-XX	Arsenic	8	mg/kg	J-	j
SB-SO-31-XX	Nickel	3200	mg/kg	J-	j
SB-SO-31-XX	Selenium	28	mg/kg	J-	j
SB-SO-31-XX	Silver	160	mg/kg	J-	e, j
SB-SO-31-XX	Zinc	3900	mg/kg	J-	j
TL-SE-13-XX	Antimony	95	mg/kg	J+	j, e
TL-SE-13-XX	Chromium	36	mg/kg	J+	j
TL-SE-13-XX	Copper	4400	mg/kg	J+	j
TL-SE-13-XX	Iron	22000	mg/kg	J+	j
TL-SE-13-XX	Lead	1100	mg/kg	J+	j
TL-SE-13-XX	Silver	160	mg/kg	J	j, e
TL-SE-13-XX	Vanadium	59	mg/kg	J+	j
WS-SO-01-XX	Mercury	5.8	mg/kg	J	e, j
WS-SO-33-XX	Arsenic	450	mg/kg	J-	j
WS-SO-33-XX	Cadmium	11	mg/kg	J-	j
WS-SO-33-XX	Chromium	120	mg/kg	J-	j
WS-SO-33-XX	Copper	150	mg/kg	J-	j
WS-SO-33-XX	Iron	28000	mg/kg	J-	j
WS-SO-33-XX	Lead	3700	mg/kg	J-	j
WS-SO-33-XX	Nickel	65	mg/kg	J-	j
WS-SO-33-XX	Silver	13	mg/kg	J-	j
WS-SO-33-XX	Vanadium	53	mg/kg	J-	j
WS-SO-33-XX	Zinc	830	mg/kg	J-	j

Notes:

mg/kg = Milligram per kilogram

e = Data were additionally qualified based on matrix spike/matrix spike duplicate exceedances

j = Data were qualified based on serial dilution exceedances

J = Result is estimated and biased could not be determined

J+ = Result is estimated and potentially biased high

J- = Result is estimated and potentially biased low

APPENDIX D

DEVELOPER AND REFERENCE LABORATORY DATA

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	10	0	-7	527	5	1,636	429	1
1	KP-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	-4	3	-56	533	12	1,746	462	1
1	KP-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	-22	3	7	481	16	1,609	464	-1
1	KP-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	-8	2	-19	473	10	1,512	440	-4
1	KP-SO-22-IX	Innov-X Systems, Inc. 35kv lamp	1	5	-45	459	9	1,588	461	-3
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-IX	Innov-X Systems, Inc. 35kv lamp	-6	1	-22	258	30	1,225	1,018	-1
2	KP-SO-13-IX	Innov-X Systems, Inc. 35kv lamp	12	1	-14	268	35	1,115	998	-5
2	KP-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	7	0	-32	261	37	1,108	1,037	2
2	KP-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	-17	12	-42	247	30	1,118	1,031	-3
2	KP-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	27	-7	-25	275	31	1,087	1,020	0
2	KP-SO-29-IX	Innov-X Systems, Inc. 35kv lamp	13	0	-39	289	29	1,151	1,033	1
2	KP-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	-8	1	-36	259	35	1,116	1,021	-2
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-IX	Innov-X Systems, Inc. 35kv lamp	13	2	-37	239	192	1,409	5,242	-4
3	KP-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	3	10	-28	240	214	1,344	4,880	4
3	KP-SO-23-IX	Innov-X Systems, Inc. 35kv lamp	34	23	-57	234	167	1,313	4,671	-7
3	KP-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	22	23	-22	252	175	1,352	4,962	2
3	KP-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	21	19	-48	260	186	1,379	5,062	4

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	201	0	-21	2	12
1	KP-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	211	-1	-6	5	5
1	KP-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	199	-2	-11	-2	11
1	KP-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	200	-1	-17	0	11
1	KP-SO-22-IX	Innov-X Systems, Inc. 35kv lamp	195	0	-33	3	12
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-IX	Innov-X Systems, Inc. 35kv lamp	126	-1	-22	1	20
2	KP-SO-13-IX	Innov-X Systems, Inc. 35kv lamp	131	-3	-12	-2	24
2	KP-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	134	-2	-4	5	25
2	KP-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	133	1	-21	-4	20
2	KP-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	123	-2	-17	1	22
2	KP-SO-29-IX	Innov-X Systems, Inc. 35kv lamp	120	1	-27	-3	16
2	KP-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	116	-2	-6	-1	20
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-IX	Innov-X Systems, Inc. 35kv lamp	114	-3	8	0	45
3	KP-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	118	-4	-7	-2	36
3	KP-SO-23-IX	Innov-X Systems, Inc. 35kv lamp	122	-4	-18	3	34
3	KP-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	115	-6	-26	0	41
3	KP-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	118	-6	-6	2	47

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	89	240	-31	20	1,273	2,361	21,534	-13
4	KP-SO-03-IX	Innov-X Systems, Inc. 35kv lamp	80	262	11	22	925	2,194	21,626	-27
4	KP-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	75	199	-10	37	998	2,235	23,083	-2
4	KP-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	112	233	-20	34	925	2,395	22,263	7
4	KP-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	81	391	34	31	989	2,368	23,346	-4
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-IX	Innov-X Systems, Inc. 35kv lamp	-25	34	-24	126	43	31,283	78	-5
5	WS-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	8	35	-19	95	39	30,559	76	-3
5	WS-SO-12-IX	Innov-X Systems, Inc. 35kv lamp	-13	38	-20	127	43	30,071	75	-2
5	WS-SO-17-IX	Innov-X Systems, Inc. 35kv lamp	-3	38	-31	121	43	30,314	78	-6
5	WS-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	7	36	-7	126	34	30,249	71	0
5	WS-SO-30-IX	Innov-X Systems, Inc. 35kv lamp	-33	36	-25	143	48	30,348	75	-2
5	WS-SO-35-IX	Innov-X Systems, Inc. 35kv lamp	-5	38	-49	103	48	29,743	74	-8
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-IX	Innov-X Systems, Inc. 35kv lamp	19	338	-16	119	132	35,708	3,899	1
6	WS-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	-20	328	-24	141	147	35,889	3,831	-3
6	WS-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	-11	347	2	118	153	35,348	3,977	-2
6	WS-SO-14-IX	Innov-X Systems, Inc. 35kv lamp	-1	370	-11	168	142	35,592	3,899	0
6	WS-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	-21	346	-4	128	143	36,499	3,968	6
6	WS-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	4	418	1	134	142	35,784	3,937	-2
6	WS-SO-33-IX	Innov-X Systems, Inc. 35kv lamp	4	296	-25	143	146	34,978	3,863	1

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	-19	-13	-15	-2	153
4	KP-SO-03-IX	Innov-X Systems, Inc. 35kv lamp	-18	-13	-6	1	136
4	KP-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	-12	-22	-11	-4	145
4	KP-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	-9	-17	1	0	115
4	KP-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	-23	-15	5	0	142
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-IX	Innov-X Systems, Inc. 35kv lamp	31	-1	-45	85	238
5	WS-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	49	0	-62	92	233
5	WS-SO-12-IX	Innov-X Systems, Inc. 35kv lamp	48	0	-52	81	226
5	WS-SO-17-IX	Innov-X Systems, Inc. 35kv lamp	37	0	-67	78	220
5	WS-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	22	1	-62	69	228
5	WS-SO-30-IX	Innov-X Systems, Inc. 35kv lamp	39	1	-41	79	219
5	WS-SO-35-IX	Innov-X Systems, Inc. 35kv lamp	21	-2	-43	80	215
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-IX	Innov-X Systems, Inc. 35kv lamp	55	-3	-46	87	801
6	WS-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	48	-5	-24	81	790
6	WS-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	54	-5	-9	90	787
6	WS-SO-14-IX	Innov-X Systems, Inc. 35kv lamp	44	-7	-59	78	791
6	WS-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	27	-5	-15	82	785
6	WS-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	33	2	-17	75	769
6	WS-SO-33-IX	Innov-X Systems, Inc. 35kv lamp	26	-4	-22	80	765

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	90	2,661	65	165	907	60,069	25,344	10
7	WS-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	42	2,786	65	106	945	60,490	25,817	33
7	WS-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	51	2,894	67	252	956	62,776	27,164	-1
7	WS-SO-22-IX	Innov-X Systems, Inc. 35kv lamp	42	2,810	93	143	966	61,052	26,550	19
7	WS-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	41	2,737	54	130	931	61,114	26,270	-6
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-IX	Innov-X Systems, Inc. 35kv lamp	75	8,291	230	178	2,687	112,563	67,243	7
8	WS-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	65	8,410	235	144	2,797	113,815	67,080	30
8	WS-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	81	8,256	209	156	2,571	109,585	65,084	8
8	WS-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	81	8,391	235	153	2,741	113,541	68,862	-10
8	WS-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	122	8,379	214	133	2,620	111,782	65,924	-18
8	WS-SO-29-IX	Innov-X Systems, Inc. 35kv lamp	29	8,768	206	147	2,674	114,179	68,568	26
8	WS-SO-37-IX	Innov-X Systems, Inc. 35kv lamp	125	8,295	230	146	2,671	109,790	66,103	22
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-IX	Innov-X Systems, Inc. 35kv lamp	158	12,613	317	168	3,980	131,132	90,459	46
9	WS-SO-19-IX	Innov-X Systems, Inc. 35kv lamp	133	12,708	275	189	4,028	130,880	90,554	-65
9	WS-SO-28-IX	Innov-X Systems, Inc. 35kv lamp	94	12,479	290	206	3,977	127,196	88,783	-46
9	WS-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	161	12,538	277	178	3,888	128,078	88,480	-17
9	WS-SO-36-IX	Innov-X Systems, Inc. 35kv lamp	202	13,214	329	177	4,022	134,047	91,422	-80

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	37	-19	10	101	4,361
7	WS-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	77	-17	17	107	4,473
7	WS-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	47	-32	10	81	4,793
7	WS-SO-22-IX	Innov-X Systems, Inc. 35kv lamp	62	-27	7	84	4,681
7	WS-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	57	-24	1	68	4,510
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-IX	Innov-X Systems, Inc. 35kv lamp	144	-61	19	106	11,840
8	WS-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	113	-59	33	83	11,945
8	WS-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	126	-56	4	96	11,577
8	WS-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	130	-71	104	84	12,116
8	WS-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	107	-61	-23	100	11,646
8	WS-SO-29-IX	Innov-X Systems, Inc. 35kv lamp	73	-56	-3	91	12,308
8	WS-SO-37-IX	Innov-X Systems, Inc. 35kv lamp	55	-47	5	98	11,601
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-IX	Innov-X Systems, Inc. 35kv lamp	66	-134	-67	79	20,630
9	WS-SO-19-IX	Innov-X Systems, Inc. 35kv lamp	18	-114	-46	84	20,942
9	WS-SO-28-IX	Innov-X Systems, Inc. 35kv lamp	109	-100	-123	55	20,777
9	WS-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	18	-133	-99	87	20,331
9	WS-SO-36-IX	Innov-X Systems, Inc. 35kv lamp	33	-132	0	66	21,305

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	-4	28	-19	144	32	26,560	66	4
10	BN-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	-7	25	-54	163	30	27,213	66	5
10	BN-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	-16	28	-22	155	29	26,714	64	-2
10	BN-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	-6	30	-1	149	25	26,725	60	2
10	BN-SO-28-IX	Innov-X Systems, Inc. 35kv lamp	9	27	-24	157	31	27,505	64	6
10	BN-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	-21	26	-7	126	32	27,420	61	5
10	BN-SO-35-IX	Innov-X Systems, Inc. 35kv lamp	-21	31	-8	136	35	26,957	68	-1
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-IX	Innov-X Systems, Inc. 35kv lamp	43	97	10	107	132	28,164	671	9
11	BN-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	40	88	1	100	113	27,044	657	-1
11	BN-SO-17-IX	Innov-X Systems, Inc. 35kv lamp	18	92	20	76	128	28,011	660	8
11	BN-SO-22-IX	Innov-X Systems, Inc. 35kv lamp	-12	93	22	102	141	27,687	676	6
11	BN-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	17	89	32	103	124	27,155	670	1
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-IX	Innov-X Systems, Inc. 35kv lamp	73	534	295	151	839	32,292	4,441	18
12	BN-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	103	546	285	127	874	31,354	4,603	8
12	BN-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	101	520	290	158	844	31,887	4,567	22
12	BN-SO-13-IX	Innov-X Systems, Inc. 35kv lamp	102	556	296	115	851	31,675	4,577	12
12	BN-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	98	527	289	170	821	31,605	4,581	20
12	BN-SO-30-IX	Innov-X Systems, Inc. 35kv lamp	99	515	286	157	842	31,086	4,537	16
12	BN-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	119	541	301	143	867	31,407	4,559	4

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	51	1	7	75	91
10	BN-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	46	1	21	77	96
10	BN-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	63	0	14	72	91
10	BN-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	40	1	2	70	93
10	BN-SO-28-IX	Innov-X Systems, Inc. 35kv lamp	38	0	11	83	92
10	BN-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	43	1	26	82	97
10	BN-SO-35-IX	Innov-X Systems, Inc. 35kv lamp	49	1	21	88	99
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-IX	Innov-X Systems, Inc. 35kv lamp	42	1	12	71	395
11	BN-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	40	1	8	72	380
11	BN-SO-17-IX	Innov-X Systems, Inc. 35kv lamp	41	2	16	82	403
11	BN-SO-22-IX	Innov-X Systems, Inc. 35kv lamp	31	5	20	75	398
11	BN-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	35	1	17	69	382
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-IX	Innov-X Systems, Inc. 35kv lamp	97	15	44	75	2,545
12	BN-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	91	13	53	67	2,625
12	BN-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	105	21	55	81	2,537
12	BN-SO-13-IX	Innov-X Systems, Inc. 35kv lamp	111	11	87	73	2,543
12	BN-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	85	16	65	83	2,582
12	BN-SO-30-IX	Innov-X Systems, Inc. 35kv lamp	105	15	42	84	2,515
12	BN-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	91	16	63	76	2,573

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	202	1,028	609	170	1,778	35,068	8,640	35
13	BN-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	226	984	588	169	1,721	34,987	8,532	21
13	BN-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	213	989	570	148	1,696	37,279	8,527	26
13	BN-SO-25-IX	Innov-X Systems, Inc. 35kv lamp	202	1,048	587	153	1,694	35,098	8,473	57
13	BN-SO-33-IX	Innov-X Systems, Inc. 35kv lamp	186	982	603	143	1,703	34,919	8,508	32
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-IX	Innov-X Systems, Inc. 35kv lamp	291	1,647	816	167	2,865	40,387	13,952	38
14	BN-SO-19-IX	Innov-X Systems, Inc. 35kv lamp	280	1,762	887	162	2,958	41,278	14,015	45
14	BN-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	287	1,704	894	92	2,839	41,623	13,835	31
14	BN-SO-29-IX	Innov-X Systems, Inc. 35kv lamp	325	1,710	893	153	2,858	40,855	14,108	37
14	BN-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	286	1,737	870	141	2,947	40,845	14,130	18
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-IX	Innov-X Systems, Inc. 35kv lamp	4	26	-20	420	593	42,799	1,044	32
15	CN-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	21	16	-8	382	659	42,705	1,069	14
15	CN-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	13	2	27	331	509	36,299	904	27
15	CN-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	-5	19	-5	402	652	42,870	1,075	37
15	CN-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	22	23	3	395	612	42,694	1,060	35
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-IX	Innov-X Systems, Inc. 35kv lamp	7	34	51	262	111	51,756	1,654	10
16	AS-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	-26	62	37	286	132	54,122	1,589	10
16	AS-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	3	40	4	286	129	50,976	1,636	5
16	AS-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	-1	16	26	285	107	55,314	1,634	8
16	AS-SO-13-IX	Innov-X Systems, Inc. 35kv lamp	-19	48	16	300	132	49,193	1,618	7

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	175	30	93	68	5,290
13	BN-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	167	27	89	82	5,153
13	BN-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	171	26	98	75	5,211
13	BN-SO-25-IX	Innov-X Systems, Inc. 35kv lamp	125	29	115	79	5,233
13	BN-SO-33-IX	Innov-X Systems, Inc. 35kv lamp	197	29	88	80	5,180
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-IX	Innov-X Systems, Inc. 35kv lamp	190	40	122	61	8,302
14	BN-SO-19-IX	Innov-X Systems, Inc. 35kv lamp	233	39	123	72	8,392
14	BN-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	233	46	127	73	8,230
14	BN-SO-29-IX	Innov-X Systems, Inc. 35kv lamp	225	43	133	65	8,451
14	BN-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	211	50	144	73	8,526
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-IX	Innov-X Systems, Inc. 35kv lamp	400	5	1	92	3,226
15	CN-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	370	5	-4	81	3,123
15	CN-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	248	2	-96	88	2,722
15	CN-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	321	4	-9	96	3,228
15	CN-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	305	3	-12	74	3,333
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-IX	Innov-X Systems, Inc. 35kv lamp	74	2	34	84	3,729
16	AS-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	86	2	20	86	3,575
16	AS-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	71	3	23	88	3,690
16	AS-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	71	3	15	85	3,637
16	AS-SO-13-IX	Innov-X Systems, Inc. 35kv lamp	92	2	42	78	3,829

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
17	AS-SO-01-XX	Reference Laboratory	3.8 J-	26	100	420	250	100,000	3,200	1.4
17	AS-SO-04-XX	Reference Laboratory	6.4 UJ	22	110	480	260	110,000	3,300	1.3
17	AS-SO-07-XX	Reference Laboratory	3.6 J-	21	97	380	240	88,000	2,900	1.4
17	AS-SO-09-XX	Reference Laboratory	2.6 UJ	25 J-	100 J-	390 J-	250 J-	94,000 J-	3,200 J-	1.4
17	AS-SO-12-XX	Reference Laboratory	2.6 UJ	29	120	440	270	93,000	3,300	1.4
17	AS-SO-01-IX	Innov-X Systems, Inc. 35kv lamp	-1	144	80	615	266	142,801	3,748	4
17	AS-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	3	129	92	715	275	141,153	3,478	0
17	AS-SO-07-IX	Innov-X Systems, Inc. 35kv lamp	-11	124	95	708	268	141,367	3,458	1
17	AS-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	-3	152	128	687	243	134,152	3,503	0
17	AS-SO-12-IX	Innov-X Systems, Inc. 35kv lamp	-2	170	119	795	285	132,117	3,519	8
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-IX	Innov-X Systems, Inc. 35kv lamp	-25	8	-44	243	46	44,329	17	82
18	SB-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	-1	6	-44	219	43	44,569	19	65
18	SB-SO-14-IX	Innov-X Systems, Inc. 35kv lamp	-8	13	-46	250	44	44,448	12	68
18	SB-SO-38-IX	Innov-X Systems, Inc. 35kv lamp	-8	10	-14	205	50	44,164	16	69
18	SB-SO-41-IX	Innov-X Systems, Inc. 35kv lamp	-28	6	-35	293	48	44,168	15	70
18	SB-SO-47-IX	Innov-X Systems, Inc. 35kv lamp	4	8	-8	304	48	44,061	16	69
18	SB-SO-51-IX	Innov-X Systems, Inc. 35kv lamp	-20	10	1	252	44	43,045	18	73
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-IX	Innov-X Systems, Inc. 35kv lamp	9	9	-8	249	45	41,916	17	351
19	SB-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	-18	9	-34	175	30	41,469	15	361
19	SB-SO-30-IX	Innov-X Systems, Inc. 35kv lamp	-7	8	-35	273	39	41,253	19	333
19	SB-SO-40-IX	Innov-X Systems, Inc. 35kv lamp	-1	9	-43	208	36	42,231	17	316
19	SB-SO-53-IX	Innov-X Systems, Inc. 35kv lamp	-1	10	-20	240	35	41,876	15	320

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
17	AS-SO-01-XX	Reference Laboratory	180	2.6 U	9.3	66	6,900
17	AS-SO-04-XX	Reference Laboratory	200	6.2 U	12	72	7,400
17	AS-SO-07-XX	Reference Laboratory	160	2.7	8.9	63	6,300
17	AS-SO-09-XX	Reference Laboratory	170 J-	2.6 U	9.6 J-	65 J-	6,800 J-
17	AS-SO-12-XX	Reference Laboratory	190	2.6 U	3.2	73	7,500
17	AS-SO-01-IX	Innov-X Systems, Inc. 35kv lamp	114	2	38	140	9,613
17	AS-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	117	6	25	124	9,122
17	AS-SO-07-IX	Innov-X Systems, Inc. 35kv lamp	147	2	10	151	9,039
17	AS-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	158	3	-11	130	8,939
17	AS-SO-12-IX	Innov-X Systems, Inc. 35kv lamp	99	4	67	150	9,454
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-IX	Innov-X Systems, Inc. 35kv lamp	270	2	-4	126	97
18	SB-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	234	3	-17	126	95
18	SB-SO-14-IX	Innov-X Systems, Inc. 35kv lamp	280	3	12	138	99
18	SB-SO-38-IX	Innov-X Systems, Inc. 35kv lamp	247	4	4	126	100
18	SB-SO-41-IX	Innov-X Systems, Inc. 35kv lamp	231	2	-3	111	92
18	SB-SO-47-IX	Innov-X Systems, Inc. 35kv lamp	220	3	-2	110	92
18	SB-SO-51-IX	Innov-X Systems, Inc. 35kv lamp	243	2	-7	114	102
19	SB-SO-05-XX	Reference Laboratory	200	1.3 U	1.3 U	61	80
19	SB-SO-18-XX	Reference Laboratory	210	1.3 U	1.3 U	70	84
19	SB-SO-30-XX	Reference Laboratory	120	1.3 J+	1.3 U	43	50
19	SB-SO-40-XX	Reference Laboratory	180	1.3 U	1.3 U	58	74
19	SB-SO-53-XX	Reference Laboratory	200	1.3 U	1.3 U	64	81
19	SB-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	220	3	11	127	81
19	SB-SO-18-IX	Innov-X Systems, Inc. 35kv lamp	256	3	7	119	79
19	SB-SO-30-IX	Innov-X Systems, Inc. 35kv lamp	236	0	6	100	81
19	SB-SO-40-IX	Innov-X Systems, Inc. 35kv lamp	222	2	2	125	87
19	SB-SO-53-IX	Innov-X Systems, Inc. 35kv lamp	221	1	-3	121	86

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
20	SB-SO-08-XX	Reference Laboratory	5.4 J-	13	0.51 U	120	39	32,000	17	730
20	SB-SO-11-XX	Reference Laboratory	5.7 J-	13	0.51 U	140	46	36,000	20	810
20	SB-SO-21-XX	Reference Laboratory	4.9 J	13	0.51 U	130	43	34,000	18	740
20	SB-SO-39-XX	Reference Laboratory	4.7 J-	13	0.51 U	140	46	34,000	19	790
20	SB-SO-42-XX	Reference Laboratory	4.6 J-	13	0.51 U	140	45	35,000	18	740
20	SB-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	-6	10	-41	169	42	38,689	17	892
20	SB-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	5	13	-29	186	36	37,997	16	918
20	SB-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	13	10	-50	231	45	38,951	23	902
20	SB-SO-39-IX	Innov-X Systems, Inc. 35kv lamp	7	14	-51	191	38	38,581	19	912
20	SB-SO-42-IX	Innov-X Systems, Inc. 35kv lamp	34	12	-32	196	41	38,794	16	838
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-IX	Innov-X Systems, Inc. 35kv lamp	5	14	27	196	37	32,283	29	2,089
21	SB-SO-25-IX	Innov-X Systems, Inc. 35kv lamp	-10	11	-42	151	34	32,354	34	2,070
21	SB-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	22	18	-1	164	37	32,220	27	2,001
21	SB-SO-35-IX	Innov-X Systems, Inc. 35kv lamp	-2	15	-32	160	31	32,429	27	2,097
21	SB-SO-44-IX	Innov-X Systems, Inc. 35kv lamp	8	18	-31	133	40	32,106	23	2,016
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-IX	Innov-X Systems, Inc. 35kv lamp	57	28	-14	24	9	4,832	55	7,278
22	SB-SO-28-IX	Innov-X Systems, Inc. 35kv lamp	34	33	-30	5	8	4,861	42	6,966
22	SB-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	32	27	-58	2	7	4,702	56	7,165
22	SB-SO-43-IX	Innov-X Systems, Inc. 35kv lamp	62	35	-38	35	8	4,988	48	6,919
22	SB-SO-48-IX	Innov-X Systems, Inc. 35kv lamp	32	25	-21	4	11	4,872	51	7,008

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
20	SB-SO-08-XX	Reference Laboratory	180	1.3 U	1.3 U	57	70
20	SB-SO-11-XX	Reference Laboratory	200	1.3 U	1.3 U	66	84
20	SB-SO-21-XX	Reference Laboratory	190	1.3 U	1.3 U	58	75
20	SB-SO-39-XX	Reference Laboratory	200	1.3 U	1.3 U	62	77
20	SB-SO-42-XX	Reference Laboratory	200	1.3 U	1.3 U	65	78
20	SB-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	210	2	2	104	71
20	SB-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	212	3	-34	132	81
20	SB-SO-21-IX	Innov-X Systems, Inc. 35kv lamp	213	3	22	137	74
20	SB-SO-39-IX	Innov-X Systems, Inc. 35kv lamp	215	1	7	126	74
20	SB-SO-42-IX	Innov-X Systems, Inc. 35kv lamp	219	5	-13	138	78
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-IX	Innov-X Systems, Inc. 35kv lamp	198	1	5	145	62
21	SB-SO-25-IX	Innov-X Systems, Inc. 35kv lamp	174	1	-4	131	65
21	SB-SO-27-IX	Innov-X Systems, Inc. 35kv lamp	196	0	0	119	63
21	SB-SO-35-IX	Innov-X Systems, Inc. 35kv lamp	205	1	-22	110	56
21	SB-SO-44-IX	Innov-X Systems, Inc. 35kv lamp	187	1	-15	120	59
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-IX	Innov-X Systems, Inc. 35kv lamp	4	-3	-45	123	-24
22	SB-SO-28-IX	Innov-X Systems, Inc. 35kv lamp	27	2	-55	104	-30
22	SB-SO-32-IX	Innov-X Systems, Inc. 35kv lamp	18	2	-57	126	-20
22	SB-SO-43-IX	Innov-X Systems, Inc. 35kv lamp	14	3	-61	135	-35
22	SB-SO-48-IX	Innov-X Systems, Inc. 35kv lamp	4	3	-59	122	-20

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
23	SB-SO-02-XX	Reference Laboratory	44 J-	23 J-	0.5 U	130	43	35,000	22 J-
23	SB-SO-07-XX	Reference Laboratory	45 J	22	0.5 U	120	38	35,000	23
23	SB-SO-10-XX	Reference Laboratory	62 J	26	0.5 U	140	44	41,000	27
23	SB-SO-26-XX	Reference Laboratory	61 J	30	0.5 U	160	50	46,000	31
23	SB-SO-50-XX	Reference Laboratory	57 J	27	0.5 U	140	46	42,000	28
23	SB-SO-02-IX	Innov-X Systems, Inc. 35kv lamp	73	22	-19	221	47	46,525	22
23	SB-SO-07-IX	Innov-X Systems, Inc. 35kv lamp	75	19	-33	204	40	48,251	29
23	SB-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	78	22	-46	211	44	46,265	23
23	SB-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	93	24	-59	217	40	47,491	27
23	SB-SO-50-IX	Innov-X Systems, Inc. 35kv lamp	55	24	-11	223	45	46,362	21
24	SB-SO-01-XX	Reference Laboratory	180 J	65	0.5 U	140	46	47,000	30
24	SB-SO-16-XX	Reference Laboratory	170 J	64	0.5 U	140	45	47,000	30
24	SB-SO-24-XX	Reference Laboratory	180 J	66	0.5 U	150	49	49,000	32
24	SB-SO-45-XX	Reference Laboratory	180 J	63	0.5 U	140	45	47,000	30
24	SB-SO-52-XX	Reference Laboratory	150 J	62	0.5 U	140	47	46,000	29
24	SB-SO-01-IX	Innov-X Systems, Inc. 35kv lamp	181	55	-17	172	38	50,961	21
24	SB-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	171	53	-15	212	52	51,847	26
24	SB-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	158	52	-25	163	47	51,575	25
24	SB-SO-45-IX	Innov-X Systems, Inc. 35kv lamp	188	48	-16	204	41	51,233	25
24	SB-SO-52-IX	Innov-X Systems, Inc. 35kv lamp	177	53	-33	227	48	50,326	26
25	SB-SO-13-XX	Reference Laboratory	430 J	160	1 U	140	46	61,000	36
25	SB-SO-19-XX	Reference Laboratory	310 J	100	0.5 U	100	32	42,000	25
25	SB-SO-33-XX	Reference Laboratory	350 J	110	0.5 U	100	33	45,000	28
25	SB-SO-37-XX	Reference Laboratory	340 J	130	1 U	120	39	51,000	31
25	SB-SO-55-XX	Reference Laboratory	340 J	120	0.5 U	120	37	49,000	29
25	SB-SO-13-IX	Innov-X Systems, Inc. 35kv lamp	393	98	-53	203	44	60,930	32
25	SB-SO-19-IX	Innov-X Systems, Inc. 35kv lamp	412	93	-21	134	42	60,441	26
25	SB-SO-33-IX	Innov-X Systems, Inc. 35kv lamp	397	105	-14	173	43	62,612	23
25	SB-SO-37-IX	Innov-X Systems, Inc. 35kv lamp	385	100	-28	205	41	62,189	30
25	SB-SO-55-IX	Innov-X Systems, Inc. 35kv lamp	404	100	-41	170	33	61,298	30
26	SB-SO-12-XX	Reference Laboratory	620 J	190	1 U	100	33	55,000	43
26	SB-SO-15-XX	Reference Laboratory	600 J-	170 J-	1 U	91 J-	30 J-	51,000 J-	40 J-
26	SB-SO-17-XX	Reference Laboratory	800 J+	210	1 U	110	37	61,000	48
26	SB-SO-46-XX	Reference Laboratory	740 J+	190	1 U	120	35	57,000	47
26	SB-SO-54-XX	Reference Laboratory	280	31	0.2 U	25	5.8	8,600	5 J-
26	SB-SO-12-IX	Innov-X Systems, Inc. 35kv lamp	610	136	-44	180	34	72,087	41
26	SB-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	600	143	0	132	35	72,870	44
26	SB-SO-17-IX	Innov-X Systems, Inc. 35kv lamp	593	143	-29	132	32	73,568	46
26	SB-SO-46-IX	Innov-X Systems, Inc. 35kv lamp	636	146	-29	141	41	73,974	42
26	SB-SO-54-IX	Innov-X Systems, Inc. 35kv lamp	573	147	-28	185	43	73,872	52

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	203	2	5	123	105
23	SB-SO-07-IX	Innov-X Systems, Inc. 35kv lamp	253	1	-15	128	103
23	SB-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	216	4	-13	102	94
23	SB-SO-26-IX	Innov-X Systems, Inc. 35kv lamp	223	3	12	127	113
23	SB-SO-50-IX	Innov-X Systems, Inc. 35kv lamp	197	3	5	115	100
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-IX	Innov-X Systems, Inc. 35kv lamp	203	3	-11	134	101
24	SB-SO-16-IX	Innov-X Systems, Inc. 35kv lamp	167	3	11	148	91
24	SB-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	198	1	19	150	94
24	SB-SO-45-IX	Innov-X Systems, Inc. 35kv lamp	201	2	0	145	136
24	SB-SO-52-IX	Innov-X Systems, Inc. 35kv lamp	179	2	29	140	88
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-IX	Innov-X Systems, Inc. 35kv lamp	187	1	-8	148	57
25	SB-SO-19-IX	Innov-X Systems, Inc. 35kv lamp	132	4	-10	148	70
25	SB-SO-33-IX	Innov-X Systems, Inc. 35kv lamp	153	3	9	164	72
25	SB-SO-37-IX	Innov-X Systems, Inc. 35kv lamp	144	1	-13	160	75
25	SB-SO-55-IX	Innov-X Systems, Inc. 35kv lamp	163	1	10	134	71
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-IX	Innov-X Systems, Inc. 35kv lamp	121	0	26	154	56
26	SB-SO-15-IX	Innov-X Systems, Inc. 35kv lamp	125	1	12	164	49
26	SB-SO-17-IX	Innov-X Systems, Inc. 35kv lamp	98	2	30	186	62
26	SB-SO-46-IX	Innov-X Systems, Inc. 35kv lamp	73	2	-2	152	59
26	SB-SO-54-IX	Innov-X Systems, Inc. 35kv lamp	77	1	-10	176	51

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	1	9	-37	142	-8	1,292	325	-1
27	KP-SE-11-IX	Innov-X Systems, Inc. 35kv lamp	26	6	-41	135	-9	1,300	329	-4
27	KP-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	7	9	-32	154	-15	1,329	335	-6
27	KP-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	-8	11	-55	147	-14	1,363	340	-4
27	KP-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	-5	9	-60	134	-8	1,262	326	-7
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-IX	Innov-X Systems, Inc. 35kv lamp	-8	6	-24	66	-8	805	336	-4
28	KP-SE-12-IX	Innov-X Systems, Inc. 35kv lamp	8	14	-38	88	-12	884	360	-1
28	KP-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	9	19	-38	92	-11	998	368	-4
28	KP-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	-17	10	-39	83	-11	850	341	-6
28	KP-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	-5	16	-31	87	-10	946	359	-3
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-IX	Innov-X Systems, Inc. 35kv lamp	-8	14	-45	66	2,488	56,927	40	-4
29	TL-SE-10-IX	Innov-X Systems, Inc. 35kv lamp	-20	10	-55	65	1,890	57,667	41	6
29	TL-SE-12-IX	Innov-X Systems, Inc. 35kv lamp	-21	9	-12	61	1,950	56,916	44	3
29	TL-SE-15-IX	Innov-X Systems, Inc. 35kv lamp	-7	9	-31	93	1,962	58,091	42	2
29	TL-SE-20-IX	Innov-X Systems, Inc. 35kv lamp	-16	7	-32	68	1,878	57,333	49	-4
29	TL-SE-24-IX	Innov-X Systems, Inc. 35kv lamp	-17	10	-23	20	1,876	57,456	39	-1
29	TL-SE-26-IX	Innov-X Systems, Inc. 35kv lamp	3	10	-25	72	1,931	57,157	38	-1

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	71	-2	-121	3	5
27	KP-SE-11-IX	Innov-X Systems, Inc. 35kv lamp	57	-1	-129	4	2
27	KP-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	59	-1	-121	6	3
27	KP-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	70	0	-149	3	0
27	KP-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	59	-2	-134	11	2
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-IX	Innov-X Systems, Inc. 35kv lamp	33	-1	-69	5	7
28	KP-SE-12-IX	Innov-X Systems, Inc. 35kv lamp	25	-2	-68	2	9
28	KP-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	46	0	-71	4	5
28	KP-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	22	-3	-79	4	6
28	KP-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	18	-1	-85	3	3
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-IX	Innov-X Systems, Inc. 35kv lamp	51	1	37	154	201
29	TL-SE-10-IX	Innov-X Systems, Inc. 35kv lamp	32	1	11	149	183
29	TL-SE-12-IX	Innov-X Systems, Inc. 35kv lamp	78	1	38	157	209
29	TL-SE-15-IX	Innov-X Systems, Inc. 35kv lamp	38	2	33	160	200
29	TL-SE-20-IX	Innov-X Systems, Inc. 35kv lamp	42	-1	25	153	208
29	TL-SE-24-IX	Innov-X Systems, Inc. 35kv lamp	69	2	29	163	196
29	TL-SE-26-IX	Innov-X Systems, Inc. 35kv lamp	67	-1	21	146	185

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	-23	6	-34	80	1,617	98,977	17	0
30	TL-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	-4	9	-39	58	1,648	101,586	14	4
30	TL-SE-23-IX	Innov-X Systems, Inc. 35kv lamp	-2	8	-37	47	1,653	98,594	15	-2
30	TL-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	-7	7	-36	67	1,657	98,647	17	-3
30	TL-SE-31-IX	Innov-X Systems, Inc. 35kv lamp	6	6	-52	75	1,677	99,485	19	2
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-IX	Innov-X Systems, Inc. 35kv lamp	-20	11	-42	243	1,459	57,132	63	49
31	TL-SE-11-IX	Innov-X Systems, Inc. 35kv lamp	-1	12	-12	252	1,599	57,145	56	38
31	TL-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	-8	7	-30	227	1,583	50,152	55	43
31	TL-SE-18-IX	Innov-X Systems, Inc. 35kv lamp	-7	11	-20	218	1,622	53,292	57	45
31	TL-SE-22-IX	Innov-X Systems, Inc. 35kv lamp	7	11	-11	223	1,575	58,420	57	44
31	TL-SE-27-IX	Innov-X Systems, Inc. 35kv lamp	8	11	-34	205	1,541	59,136	55	56
31	TL-SE-29-IX	Innov-X Systems, Inc. 35kv lamp	-1	11	-37	289	1,552	51,585	64	46
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-IX	Innov-X Systems, Inc. 35kv lamp	-15	29	-9	75	49	33,209	33	36
32	LV-SE-10-IX	Innov-X Systems, Inc. 35kv lamp	-18	28	-42	75	52	32,986	28	44
32	LV-SE-22-IX	Innov-X Systems, Inc. 35kv lamp	-15	23	-46	97	48	31,586	32	40
32	LV-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	-18	23	-22	70	47	31,710	34	45
32	LV-SE-31-IX	Innov-X Systems, Inc. 35kv lamp	1	25	-11	82	46	31,788	38	43
32	LV-SE-35-IX	Innov-X Systems, Inc. 35kv lamp	4	23	-28	68	41	32,656	37	47
32	LV-SE-50-IX	Innov-X Systems, Inc. 35kv lamp	-11	25	-17	85	44	30,877	32	39

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	81	1	12	200	240
30	TL-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	80	2	5	190	227
30	TL-SE-23-IX	Innov-X Systems, Inc. 35kv lamp	117	0	23	223	224
30	TL-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	123	0	18	216	214
30	TL-SE-31-IX	Innov-X Systems, Inc. 35kv lamp	90	1	12	227	225
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-IX	Innov-X Systems, Inc. 35kv lamp	281	6	58	119	181
31	TL-SE-11-IX	Innov-X Systems, Inc. 35kv lamp	312	6	72	115	175
31	TL-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	238	4	75	113	156
31	TL-SE-18-IX	Innov-X Systems, Inc. 35kv lamp	292	4	68	113	181
31	TL-SE-22-IX	Innov-X Systems, Inc. 35kv lamp	273	6	80	131	164
31	TL-SE-27-IX	Innov-X Systems, Inc. 35kv lamp	280	6	52	134	163
31	TL-SE-29-IX	Innov-X Systems, Inc. 35kv lamp	368	7	77	134	187
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-IX	Innov-X Systems, Inc. 35kv lamp	184	9	19	117	93
32	LV-SE-10-IX	Innov-X Systems, Inc. 35kv lamp	168	8	20	94	95
32	LV-SE-22-IX	Innov-X Systems, Inc. 35kv lamp	194	5	15	100	93
32	LV-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	175	7	15	69	95
32	LV-SE-31-IX	Innov-X Systems, Inc. 35kv lamp	166	6	2	98	89
32	LV-SE-35-IX	Innov-X Systems, Inc. 35kv lamp	168	8	26	111	97
32	LV-SE-50-IX	Innov-X Systems, Inc. 35kv lamp	180	8	-7	98	93

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	1	132	-11	29	46	105,540	34	17
33	LV-SE-26-IX	Innov-X Systems, Inc. 35kv lamp	-33	126	-1	-20	42	106,853	36	11
33	LV-SE-33-IX	Innov-X Systems, Inc. 35kv lamp	-20	135	-27	22	33	105,638	27	13
33	LV-SE-39-IX	Innov-X Systems, Inc. 35kv lamp	-23	143	-48	15	38	104,894	33	19
33	LV-SE-42-IX	Innov-X Systems, Inc. 35kv lamp	-26	136	-25	-11	47	107,714	31	24
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-IX	Innov-X Systems, Inc. 35kv lamp	20	300	-49	-44	49	294,077	49	10
34	LV-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	-33	297	-66	-13	48	285,312	54	2
34	LV-SE-27-IX	Innov-X Systems, Inc. 35kv lamp	-24	295	18	-71	55	296,711	58	5
34	LV-SE-36-IX	Innov-X Systems, Inc. 35kv lamp	-8	311	-19	-61	56	289,170	68	14
34	LV-SE-38-IX	Innov-X Systems, Inc. 35kv lamp	-35	306	-19	-58	60	293,049	54	13
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-IX	Innov-X Systems, Inc. 35kv lamp	-53	457	-18	-96	59	431,495	85	3
35	LV-SE-18-IX	Innov-X Systems, Inc. 35kv lamp	-26	482	-22	-119	54	445,631	88	-4
35	LV-SE-23-IX	Innov-X Systems, Inc. 35kv lamp	26	519	-50	-25	70	433,850	74	-1
35	LV-SE-45-IX	Innov-X Systems, Inc. 35kv lamp	-38	471	-42	-97	67	440,217	90	-4
35	LV-SE-48-IX	Innov-X Systems, Inc. 35kv lamp	1	473	-38	-69	69	437,225	87	-5

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	31	4	-6	173	76
33	LV-SE-26-IX	Innov-X Systems, Inc. 35kv lamp	29	2	-2	188	73
33	LV-SE-33-IX	Innov-X Systems, Inc. 35kv lamp	69	5	11	205	77
33	LV-SE-39-IX	Innov-X Systems, Inc. 35kv lamp	0	3	-17	156	83
33	LV-SE-42-IX	Innov-X Systems, Inc. 35kv lamp	42	5	-20	193	89
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-IX	Innov-X Systems, Inc. 35kv lamp	-114	5	-52	238	55
34	LV-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	-91	4	-68	263	52
34	LV-SE-27-IX	Innov-X Systems, Inc. 35kv lamp	-140	5	-70	256	58
34	LV-SE-36-IX	Innov-X Systems, Inc. 35kv lamp	-132	4	-73	261	59
34	LV-SE-38-IX	Innov-X Systems, Inc. 35kv lamp	-203	4	-78	233	60
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-IX	Innov-X Systems, Inc. 35kv lamp	-243	5	-133	384	7
35	LV-SE-18-IX	Innov-X Systems, Inc. 35kv lamp	-156	5	-154	387	1
35	LV-SE-23-IX	Innov-X Systems, Inc. 35kv lamp	-278	7	-135	361	11
35	LV-SE-45-IX	Innov-X Systems, Inc. 35kv lamp	-344	8	-124	419	-1
35	LV-SE-48-IX	Innov-X Systems, Inc. 35kv lamp	-223	7	-144	362	13

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	-28	2	-16	-2	8	611	10	1
36	LV-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	-1	3	-23	8	7	741	17	4
36	LV-SE-21-IX	Innov-X Systems, Inc. 35kv lamp	-14	4	-16	3	-2	624	8	6
36	LV-SE-24-IX	Innov-X Systems, Inc. 35kv lamp	-2	6	-16	-16	11	699	8	3
36	LV-SE-32-IX	Innov-X Systems, Inc. 35kv lamp	-20	5	1	-7	4	740	12	-1
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-IX	Innov-X Systems, Inc. 35kv lamp	-18	29	-40	61	37	33,005	74	43
37	LV-SE-16-IX	Innov-X Systems, Inc. 35kv lamp	0	31	-57	71	25	33,700	79	49
37	LV-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	8	31	-16	68	32	32,473	76	48
37	LV-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	5	31	-21	41	30	33,170	75	48
37	LV-SE-47-IX	Innov-X Systems, Inc. 35kv lamp	4	29	-19	38	40	33,061	73	41
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-IX	Innov-X Systems, Inc. 35kv lamp	-10	108	-6	121	234	53,297	10	9
38	LV-SE-29-IX	Innov-X Systems, Inc. 35kv lamp	-17	105	-8	105	244	53,528	10	14
38	LV-SE-44-IX	Innov-X Systems, Inc. 35kv lamp	0	105	-17	121	250	52,987	14	8
38	LV-SE-46-IX	Innov-X Systems, Inc. 35kv lamp	-8	102	-13	105	235	52,634	13	7
38	LV-SE-52-IX	Innov-X Systems, Inc. 35kv lamp	-2	101	-10	115	250	53,392	10	8

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	45	0	53	0	6
36	LV-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	48	1	53	1	17
36	LV-SE-21-IX	Innov-X Systems, Inc. 35kv lamp	41	2	66	2	10
36	LV-SE-24-IX	Innov-X Systems, Inc. 35kv lamp	52	-1	39	3	11
36	LV-SE-32-IX	Innov-X Systems, Inc. 35kv lamp	41	0	42	-3	11
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-IX	Innov-X Systems, Inc. 35kv lamp	154	8	3	85	88
37	LV-SE-16-IX	Innov-X Systems, Inc. 35kv lamp	169	8	-15	99	94
37	LV-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	122	11	-13	100	99
37	LV-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	133	7	-18	92	93
37	LV-SE-47-IX	Innov-X Systems, Inc. 35kv lamp	113	8	0	85	94
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-IX	Innov-X Systems, Inc. 35kv lamp	1,160	-1	-17	83	203
38	LV-SE-29-IX	Innov-X Systems, Inc. 35kv lamp	1,108	2	-13	88	184
38	LV-SE-44-IX	Innov-X Systems, Inc. 35kv lamp	1,067	0	-16	101	201
38	LV-SE-46-IX	Innov-X Systems, Inc. 35kv lamp	1,075	0	-24	86	194
38	LV-SE-52-IX	Innov-X Systems, Inc. 35kv lamp	1,085	1	-7	69	202

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	-5	9	-20	157	94	25,133	49	55
39	RF-SE-12-IX	Innov-X Systems, Inc. 35kv lamp	-2	11	-37	109	112	25,203	43	56
39	RF-SE-23-IX	Innov-X Systems, Inc. 35kv lamp	7	14	-10	117	99	24,381	43	60
39	RF-SE-36-IX	Innov-X Systems, Inc. 35kv lamp	-22	13	-36	120	103	24,799	42	55
39	RF-SE-42-IX	Innov-X Systems, Inc. 35kv lamp	0	10	-9	145	107	23,810	44	58
39	RF-SE-45-IX	Innov-X Systems, Inc. 35kv lamp	-22	14	-28	88	107	25,669	49	58
39	RF-SE-53-IX	Innov-X Systems, Inc. 35kv lamp	-33	16	-26	119	107	24,176	39	53
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-IX	Innov-X Systems, Inc. 35kv lamp	9	21	-45	104	216	23,696	100	50
40	RF-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	-19	21	-26	111	200	22,647	97	44
40	RF-SE-38-IX	Innov-X Systems, Inc. 35kv lamp	-24	22	7	97	217	23,382	109	41
40	RF-SE-49-IX	Innov-X Systems, Inc. 35kv lamp	-3	23	-36	105	206	22,958	96	33
40	RF-SE-55-IX	Innov-X Systems, Inc. 35kv lamp	-12	28	-24	144	237	24,303	102	44
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-IX	Innov-X Systems, Inc. 35kv lamp	4	52	-4	101	498	25,560	232	44
41	RF-SE-13-IX	Innov-X Systems, Inc. 35kv lamp	2	63	-61	85	477	26,276	236	45
41	RF-SE-27-IX	Innov-X Systems, Inc. 35kv lamp	-14	62	-56	85	504	25,380	225	48
41	RF-SE-31-IX	Innov-X Systems, Inc. 35kv lamp	0	62	-20	76	474	24,701	245	50
41	RF-SE-58-IX	Innov-X Systems, Inc. 35kv lamp	-13	52	-51	95	492	23,903	245	43

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	277	7	25	58	163
39	RF-SE-12-IX	Innov-X Systems, Inc. 35kv lamp	228	6	31	65	172
39	RF-SE-23-IX	Innov-X Systems, Inc. 35kv lamp	280	6	25	57	149
39	RF-SE-36-IX	Innov-X Systems, Inc. 35kv lamp	237	8	4	68	160
39	RF-SE-42-IX	Innov-X Systems, Inc. 35kv lamp	250	7	3	57	163
39	RF-SE-45-IX	Innov-X Systems, Inc. 35kv lamp	237	5	8	71	161
39	RF-SE-53-IX	Innov-X Systems, Inc. 35kv lamp	253	7	14	62	166
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-IX	Innov-X Systems, Inc. 35kv lamp	182	5	14	60	304
40	RF-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	164	5	33	66	329
40	RF-SE-38-IX	Innov-X Systems, Inc. 35kv lamp	196	7	23	67	346
40	RF-SE-49-IX	Innov-X Systems, Inc. 35kv lamp	165	7	9	70	311
40	RF-SE-55-IX	Innov-X Systems, Inc. 35kv lamp	197	4	8	66	341
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-IX	Innov-X Systems, Inc. 35kv lamp	155	4	32	66	807
41	RF-SE-13-IX	Innov-X Systems, Inc. 35kv lamp	169	7	21	69	764
41	RF-SE-27-IX	Innov-X Systems, Inc. 35kv lamp	196	5	8	65	744
41	RF-SE-31-IX	Innov-X Systems, Inc. 35kv lamp	170	5	36	68	718
41	RF-SE-58-IX	Innov-X Systems, Inc. 35kv lamp	173	7	25	69	706

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	-12	73	-37	95	619	26,948	307	42
42	RF-SE-22-IX	Innov-X Systems, Inc. 35kv lamp	-9	79	-32	79	623	26,374	300	55
42	RF-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	14	74	-19	112	636	25,670	314	46
42	RF-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	-13	70	-16	84	652	27,008	297	44
42	RF-SE-57-IX	Innov-X Systems, Inc. 35kv lamp	-18	76	-34	113	623	26,489	286	47
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-IX	Innov-X Systems, Inc. 35kv lamp	12	101	-27	71	853	28,417	399	45
43	RF-SE-24-IX	Innov-X Systems, Inc. 35kv lamp	-3	89	-16	96	860	27,333	444	61
43	RF-SE-32-IX	Innov-X Systems, Inc. 35kv lamp	17	101	-37	78	865	27,823	411	54
43	RF-SE-43-IX	Innov-X Systems, Inc. 35kv lamp	-13	100	-43	77	871	29,418	419	46
43	RF-SE-59-IX	Innov-X Systems, Inc. 35kv lamp	12	99	7	100	877	28,163	412	61
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-IX	Innov-X Systems, Inc. 35kv lamp	4	117	4	84	1,073	29,871	458	52
44	RF-SE-26-IX	Innov-X Systems, Inc. 35kv lamp	-1	119	-18	93	1,101	30,123	460	39
44	RF-SE-39-IX	Innov-X Systems, Inc. 35kv lamp	-19	123	-22	135	1,103	31,404	460	42
44	RF-SE-44-IX	Innov-X Systems, Inc. 35kv lamp	26	119	-6	82	1,138	33,189	453	50
44	RF-SE-56-IX	Innov-X Systems, Inc. 35kv lamp	-3	116	-35	88	1,117	30,125	454	48
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-IX	Innov-X Systems, Inc. 35kv lamp	-45	176	-28	17	1,539	33,362	774	55
45	RF-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	-4	178	10	35	1,501	32,643	746	40
45	RF-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	-4	177	-31	56	1,528	33,601	728	54
45	RF-SE-34-IX	Innov-X Systems, Inc. 35kv lamp	6	166	0	36	1,519	32,379	757	47
45	RF-SE-52-IX	Innov-X Systems, Inc. 35kv lamp	-17	178	-10	59	1,477	32,630	730	56

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	182	8	0	76	997
42	RF-SE-22-IX	Innov-X Systems, Inc. 35kv lamp	195	7	16	69	975
42	RF-SE-25-IX	Innov-X Systems, Inc. 35kv lamp	186	5	36	53	946
42	RF-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	175	8	25	75	923
42	RF-SE-57-IX	Innov-X Systems, Inc. 35kv lamp	192	4	2	71	890
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-IX	Innov-X Systems, Inc. 35kv lamp	190	4	27	62	1,253
43	RF-SE-24-IX	Innov-X Systems, Inc. 35kv lamp	198	7	33	72	1,245
43	RF-SE-32-IX	Innov-X Systems, Inc. 35kv lamp	198	8	12	74	1,247
43	RF-SE-43-IX	Innov-X Systems, Inc. 35kv lamp	201	8	28	76	1,309
43	RF-SE-59-IX	Innov-X Systems, Inc. 35kv lamp	206	4	24	66	1,325
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-IX	Innov-X Systems, Inc. 35kv lamp	175	7	50	79	1,604
44	RF-SE-26-IX	Innov-X Systems, Inc. 35kv lamp	172	7	22	73	1,646
44	RF-SE-39-IX	Innov-X Systems, Inc. 35kv lamp	179	7	30	71	1,683
44	RF-SE-44-IX	Innov-X Systems, Inc. 35kv lamp	169	7	30	74	1,736
44	RF-SE-56-IX	Innov-X Systems, Inc. 35kv lamp	140	6	42	68	1,672
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-IX	Innov-X Systems, Inc. 35kv lamp	174	6	51	78	2,291
45	RF-SE-14-IX	Innov-X Systems, Inc. 35kv lamp	137	5	50	82	2,166
45	RF-SE-19-IX	Innov-X Systems, Inc. 35kv lamp	165	6	40	68	2,322
45	RF-SE-34-IX	Innov-X Systems, Inc. 35kv lamp	145	7	31	71	2,238
45	RF-SE-52-IX	Innov-X Systems, Inc. 35kv lamp	167	5	19	74	2,184

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	94	2,195	764	1,181	134	29,897	64	11
46	BN-SO-14-IX	Innov-X Systems, Inc. 35kv lamp	66	2,244	789	1,122	137	30,207	62	23
46	BN-SO-23-IX	Innov-X Systems, Inc. 35kv lamp	91	2,302	777	1,201	156	29,969	67	12
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-IX	Innov-X Systems, Inc. 35kv lamp	968	139	2,881	3,426	110	29,088	4,951	1
47	BN-SO-12-IX	Innov-X Systems, Inc. 35kv lamp	908	144	2,759	3,176	139	29,592	4,627	0
47	BN-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	948	123	2,808	3,378	103	28,805	4,675	2
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-IX	Innov-X Systems, Inc. 35kv lamp	-17	6	-39	235	127	43,424	16	47
48	SB-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	-21	8	-43	255	122	43,503	13	44
48	SB-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	-8	8	-43	295	144	44,648	19	45
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-IX	Innov-X Systems, Inc. 35kv lamp	-16	3	-20	207	143	44,283	24	51
49	SB-SO-36-IX	Innov-X Systems, Inc. 35kv lamp	-27	7	6	237	129	43,734	20	42
49	SB-SO-56-IX	Innov-X Systems, Inc. 35kv lamp	7	9	-16	242	138	43,275	16	56
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-IX	Innov-X Systems, Inc. 35kv lamp	1,078	11	2,895	3,545	90	40,647	18	56
50	SB-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	1,093	9	3,030	3,698	122	41,793	24	60
50	SB-SO-49-IX	Innov-X Systems, Inc. 35kv lamp	1,078	10	2,917	3,647	109	40,655	16	54

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	4,166	137	129	112	4,135
46	BN-SO-14-IX	Innov-X Systems, Inc. 35kv lamp	4,168	138	152	122	4,235
46	BN-SO-23-IX	Innov-X Systems, Inc. 35kv lamp	4,545	145	137	113	4,415
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-IX	Innov-X Systems, Inc. 35kv lamp	2,223	316	308	154	84
47	BN-SO-12-IX	Innov-X Systems, Inc. 35kv lamp	2,035	310	305	156	100
47	BN-SO-24-IX	Innov-X Systems, Inc. 35kv lamp	2,083	303	333	150	96
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-IX	Innov-X Systems, Inc. 35kv lamp	4,214	28	300	148	3,730
48	SB-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	4,257	31	289	131	3,685
48	SB-SO-31-IX	Innov-X Systems, Inc. 35kv lamp	4,376	30	329	142	3,838
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-IX	Innov-X Systems, Inc. 35kv lamp	264	158	0	210	3,992
49	SB-SO-36-IX	Innov-X Systems, Inc. 35kv lamp	268	151	-8	222	3,870
49	SB-SO-56-IX	Innov-X Systems, Inc. 35kv lamp	211	155	-6	199	3,863
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-IX	Innov-X Systems, Inc. 35kv lamp	4,302	382	-11	96	86
50	SB-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	4,338	380	5	104	87
50	SB-SO-49-IX	Innov-X Systems, Inc. 35kv lamp	4,211	375	-35	116	89

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb
51	WS-SO-07-XX	Reference Laboratory	3.8	53	1.9	640	4,400	25,000	1,700
51	WS-SO-11-XX	Reference Laboratory	1.2 U	46	1.4	570	3,900	19,000	1,500
51	WS-SO-25-XX	Reference Laboratory	1.2 U	59	3.1	730	4,900	24,000	1,900
51	WS-SO-07-IX	Innov-X Systems, Inc. 35kv lamp	-50	94	-27	1,066	5,713	33,164	2,086
51	WS-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	-14	67	-38	947	5,294	32,705	2,041
51	WS-SO-25-IX	Innov-X Systems, Inc. 35kv lamp	19	73	-15	1,037	5,437	32,934	2,050
52	WS-SO-10-XX	Reference Laboratory	1.3 U	83	1.8	67	76	19,000	1,900
52	WS-SO-20-XX	Reference Laboratory	1.3 U	100	1.9	81	90	23,000	2,300
52	WS-SO-23-XX	Reference Laboratory	1.3 U	110	2.1	82	96	23,000	2,500
52	WS-SO-10-IX	Innov-X Systems, Inc. 35kv lamp	-17	109	-14	128	93	32,196	2,696
52	WS-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	-3	156	-27	131	99	32,323	2,657
52	WS-SO-23-IX	Innov-X Systems, Inc. 35kv lamp	6	174	-16	126	111	32,448	2,732
53	AS-SO-03-XX	Reference Laboratory	1.2 U	14	1,300	33	6,200	15,000	160
53	AS-SO-05-XX	Reference Laboratory	1.2 U	9	900	23	4,500	11,000	110
53	AS-SO-08-XX	Reference Laboratory	1.2 U	10	930	24	4,600	11,000	120
53	AS-SO-03-IX	Innov-X Systems, Inc. 35kv lamp	109	10	1,239	85	5,582	19,183	162
53	AS-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	108	10	1,209	73	5,690	18,848	165
53	AS-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	117	9	1,196	101	5,549	19,037	163
54	LV-SO-03-XX	Reference Laboratory	1.6	42	590	600	130	24,000	94
54	LV-SO-40-XX	Reference Laboratory	2.7	42	580	590	130	24,000	92
54	LV-SO-49-XX	Reference Laboratory	7.4	43	600	610	130	25,000	98
54	LV-SO-03-IX	Innov-X Systems, Inc. 35kv lamp	48	42	643	909	162	41,422	126
54	LV-SO-40-IX	Innov-X Systems, Inc. 35kv lamp	65	39	617	936	146	40,768	118
54	LV-SO-49-IX	Innov-X Systems, Inc. 35kv lamp	63	37	627	879	163	41,686	130
55	LV-SO-04-XX	Reference Laboratory	860	120	2,400	2,300	98	22,000	4,000
55	LV-SO-34-XX	Reference Laboratory	870 J-	110 J-	2,300 J-	2,200 J-	87	20,000 J-	3,700 J-
55	LV-SO-37-XX	Reference Laboratory	590	84	1,700	1,600	66	16,000	2,800
55	LV-SO-04-IX	Innov-X Systems, Inc. 35kv lamp	922	228	2,790	3,341	151	39,948	4,891
55	LV-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	908	210	2,842	3,445	146	41,401	5,051
55	LV-SO-37-IX	Innov-X Systems, Inc. 35kv lamp	937	217	2,873	3,585	144	42,028	5,158
56	CN-SO-03-XX	Reference Laboratory	22	87	63	17	72	15,000	130
56	CN-SO-06-XX	Reference Laboratory	20	91	64	18	74	16,000	130
56	CN-SO-07-XX	Reference Laboratory	20	90	63	19	72	17,000	130
56	CN-SO-03-IX	Innov-X Systems, Inc. 35kv lamp	23	87	46	23	98	30,109	180
56	CN-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	22	81	38	22	92	26,227	183
56	CN-SO-07-IX	Innov-X Systems, Inc. 35kv lamp	8	90	84	33	86	24,128	186

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube 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-IX	Innov-X Systems, Inc. 35kv lamp	476	-3	338	73	255
51	WS-SO-11-IX	Innov-X Systems, Inc. 35kv lamp	395	-2	357	82	246
51	WS-SO-25-IX	Innov-X Systems, Inc. 35kv lamp	417	-2	348	82	248
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-IX	Innov-X Systems, Inc. 35kv lamp	529	364	-46	172	2,842
52	WS-SO-20-IX	Innov-X Systems, Inc. 35kv lamp	559	370	-48	182	2,808
52	WS-SO-23-IX	Innov-X Systems, Inc. 35kv lamp	548	387	-35	166	2,985
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-IX	Innov-X Systems, Inc. 35kv lamp	601	182	395	63	358
53	AS-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	619	177	384	54	367
53	AS-SO-08-IX	Innov-X Systems, Inc. 35kv lamp	655	174	379	52	365
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-IX	Innov-X Systems, Inc. 35kv lamp	3,123	138	178	152	4,432
54	LV-SO-40-IX	Innov-X Systems, Inc. 35kv lamp	3,099	135	181	138	4,358
54	LV-SO-49-IX	Innov-X Systems, Inc. 35kv lamp	3,001	135	204	137	4,382
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-IX	Innov-X Systems, Inc. 35kv lamp	3,325	284	-3	166	95
55	LV-SO-34-IX	Innov-X Systems, Inc. 35kv lamp	3,469	279	11	181	101
55	LV-SO-37-IX	Innov-X Systems, Inc. 35kv lamp	3,505	293	12	194	104
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-IX	Innov-X Systems, Inc. 35kv lamp	98	43	127	74	76
56	CN-SO-06-IX	Innov-X Systems, Inc. 35kv lamp	121	43	116	81	75
56	CN-SO-07-IX	Innov-X Systems, Inc. 35kv lamp	131	41	115	68	92

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	194	2	592	39	165	29,795	45	302
57	CN-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	203	4	616	46	175	29,956	43	303
57	CN-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	182	1	588	61	168	29,026	47	286
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-IX	Innov-X Systems, Inc. 35kv lamp	39	59	188	771	57	32,523	2,048	721
58	LV-SE-13-IX	Innov-X Systems, Inc. 35kv lamp	73	63	171	769	54	30,820	2,004	693
58	LV-SE-41-IX	Innov-X Systems, Inc. 35kv lamp	33	76	145	794	56	31,992	2,043	706
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-IX	Innov-X Systems, Inc. 35kv lamp	204	28	627	1,514	63	32,230	27	40
59	LV-SE-20-IX	Innov-X Systems, Inc. 35kv lamp	211	23	609	1,499	71	32,417	26	52
59	LV-SE-43-IX	Innov-X Systems, Inc. 35kv lamp	195	24	617	1,524	65	32,294	30	41
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-IX	Innov-X Systems, Inc. 35kv lamp	357	23	1,153	102	2,061	31,486	22	567
60	LV-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	332	21	1,227	79	2,074	31,410	33	559
60	LV-SE-51-IX	Innov-X Systems, Inc. 35kv lamp	358	23	1,185	109	2,091	31,566	26	585
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-IX	Innov-X Systems, Inc. 35kv lamp	146	9	-77	42	3,922	36,354	1,200	888
61	TL-SE-09-IX	Innov-X Systems, Inc. 35kv lamp	185	40	-31	40	4,021	36,251	1,151	836
61	TL-SE-13-IX	Innov-X Systems, Inc. 35kv lamp	137	40	-43	51	4,150	37,444	1,177	872
62	TL-SE-06-XX	Reference Laboratory	1.2 U	86	350	34	2000	22,000	1,700	2.2
62	TL-SE-17-XX	Reference Laboratory	1.2 U	85	340	33	2100	21,000	1,700	2.6
62	TL-SE-28-XX	Reference Laboratory	1.2 U	89	360	34	2100	22,000	1,700	2.8
62	TL-SE-06-IX	Innov-X Systems, Inc. 35kv lamp	25	87	372	47	2,065	35,446	1,916	10
62	TL-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	65	111	410	18	2,156	35,884	1,908	-2
62	TL-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	38	111	391	53	2,178	35,357	1,881	-1

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	509	186	109	110	2,294
57	CN-SO-05-IX	Innov-X Systems, Inc. 35kv lamp	497	175	97	116	2,282
57	CN-SO-09-IX	Innov-X Systems, Inc. 35kv lamp	520	178	92	102	2,218
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-IX	Innov-X Systems, Inc. 35kv lamp	599	197	135	233	98
58	LV-SE-13-IX	Innov-X Systems, Inc. 35kv lamp	586	193	133	243	93
58	LV-SE-41-IX	Innov-X Systems, Inc. 35kv lamp	576	197	131	245	87
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-IX	Innov-X Systems, Inc. 35kv lamp	809	459	74	214	2,457
59	LV-SE-20-IX	Innov-X Systems, Inc. 35kv lamp	759	441	92	221	2,416
59	LV-SE-43-IX	Innov-X Systems, Inc. 35kv lamp	745	449	90	230	2,473
60	LV-SE-15-XX	Reference Laboratory	500	92	300 J-	180	62
60	LV-SE-17-XX	Reference Laboratory	490	89	200 J-	170	58
60	LV-SE-51-XX	Reference Laboratory	470	76	250 J-	160	54
60	LV-SE-15-IX	Innov-X Systems, Inc. 35kv lamp	567	81	381	120	96
60	LV-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	559	80	401	132	109
60	LV-SE-51-IX	Innov-X Systems, Inc. 35kv lamp	585	85	416	115	114
61	TL-SE-05-XX	Reference Laboratory	980	130	180 J-	66	100
61	TL-SE-09-XX	Reference Laboratory	820	130	170 J-	63	100
61	TL-SE-13-XX	Reference Laboratory	990	120	160 J	59 J+	96
61	TL-SE-05-IX	Innov-X Systems, Inc. 35kv lamp	888	123	167	101	119
61	TL-SE-09-IX	Innov-X Systems, Inc. 35kv lamp	836	126	177	94	121
61	TL-SE-13-IX	Innov-X Systems, Inc. 35kv lamp	872	124	185	85	119
62	TL-SE-06-XX	Reference Laboratory	2.2	45	56	78	83
62	TL-SE-17-XX	Reference Laboratory	2.6	44	56	78	81
62	TL-SE-28-XX	Reference Laboratory	2.8	45	57	81	83
62	TL-SE-06-IX	Innov-X Systems, Inc. 35kv lamp	10	47	107	92	93
62	TL-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	-2	47	90	122	93
62	TL-SE-28-IX	Innov-X Systems, Inc. 35kv lamp	-1	54	92	103	103

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
63	TL-SE-07-XX	Reference Laboratory	30	11	48	66	2200	37,000	13	40
63	TL-SE-21-XX	Reference Laboratory	33	13	51	73	2300	44,000	15	120
63	TL-SE-30-XX	Reference Laboratory	31	11	47	64	2200	36,000	14	100
63	TL-SE-07-IX	Innov-X Systems, Inc. 35kv lamp	50	13	39	46	2,387	87,577	18	46
63	TL-SE-21-IX	Innov-X Systems, Inc. 35kv lamp	77	8	26	78	2,321	87,729	21	52
63	TL-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	57	10	40	47	2,342	86,195	16	53
64	TL-SE-02-XX	Reference Laboratory	77	15	160	64	3,100	32,000	12	400
64	TL-SE-08-XX	Reference Laboratory	66	10	180	74	3,200	45,000	11	350
64	TL-SE-16-XX	Reference Laboratory	73	15	170	69	3,100	38,000	13	420
64	TL-SE-02-IX	Innov-X Systems, Inc. 35kv lamp	174	12	155	97	3,278	90,769	20	185
64	TL-SE-08-IX	Innov-X Systems, Inc. 35kv lamp	184	12	179	47	3,232	89,540	18	188
64	TL-SE-16-IX	Innov-X Systems, Inc. 35kv lamp	172	13	162	36	3,126	91,438	17	173
65	RF-SE-01-XX	Reference Laboratory	12	230	40	280	63	14,000	22	47
65	RF-SE-09-XX	Reference Laboratory	10	260	45	310	71	16,000	26	45
65	RF-SE-11-XX	Reference Laboratory	11	240	43	300	72	15,000	25	52
65	RF-SE-17-XX	Reference Laboratory	11	250	43	300	67	15,000	26	20
65	RF-SE-29-XX	Reference Laboratory	13	280	49	330	75	17,000	26	20
65	RF-SE-37-XX	Reference Laboratory	11	260	45	320	72	16,000	27	22
65	RF-SE-50-XX	Reference Laboratory	8.9	230	40	280	65	14,000	23	19
65	RF-SE-01-IX	Innov-X Systems, Inc. 35kv lamp	1	198	10	391	94	19,471	45	74
65	RF-SE-09-IX	Innov-X Systems, Inc. 35kv lamp	13	238	30	529	101	22,066	36	97
65	RF-SE-11-IX	Innov-X Systems, Inc. 35kv lamp	16	216	-5	441	89	19,913	38	79
65	RF-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	-6	207	13	420	88	18,816	32	89
65	RF-SE-29-IX	Innov-X Systems, Inc. 35kv lamp	23	209	22	470	89	20,366	84	97
65	RF-SE-37-IX	Innov-X Systems, Inc. 35kv lamp	35	215	15	466	97	20,376	39	95
65	RF-SE-50-IX	Innov-X Systems, Inc. 35kv lamp	22	208	43	390	87	19,867	36	77
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-IX	Innov-X Systems, Inc. 35kv lamp	17	390	65	821	2,047	23,349	648	127
66	RF-SE-10-IX	Innov-X Systems, Inc. 35kv lamp	7	387	60	724	1,984	23,327	622	120
66	RF-SE-33-IX	Innov-X Systems, Inc. 35kv lamp	10	401	78	787	1,963	23,512	602	118
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-IX	Innov-X Systems, Inc. 35kv lamp	113	67	325	1,220	92	21,429	41	365
67	RF-SE-41-IX	Innov-X Systems, Inc. 35kv lamp	122	68	341	1,247	98	21,873	59	347
67	RF-SE-48-IX	Innov-X Systems, Inc. 35kv lamp	130	67	312	1,266	108	21,889	39	365

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Hg	Ni	Se	Ag	V	Zn
63	TL-SE-07-XX	Reference Laboratory	40	40	120	63	110	160
63	TL-SE-21-XX	Reference Laboratory	120	120	140	67	120	170
63	TL-SE-30-XX	Reference Laboratory	100	100	120	62	100	160
63	TL-SE-07-IX	Innov-X Systems, Inc. 35kv lamp	46	46	163	100	218	192
63	TL-SE-21-IX	Innov-X Systems, Inc. 35kv lamp	52	52	157	116	225	201
63	TL-SE-30-IX	Innov-X Systems, Inc. 35kv lamp	53	53	157	100	197	191
64	TL-SE-02-XX	Reference Laboratory	400	400	44	120	110	160
64	TL-SE-08-XX	Reference Laboratory	350	350	39	130	120	170
64	TL-SE-16-XX	Reference Laboratory	420	420	44	120	110	160
64	TL-SE-02-IX	Innov-X Systems, Inc. 35kv lamp	185	185	60	158	199	192
64	TL-SE-08-IX	Innov-X Systems, Inc. 35kv lamp	188	188	60	176	196	201
64	TL-SE-16-IX	Innov-X Systems, Inc. 35kv lamp	173	173	59	181	196	199
65	RF-SE-01-XX	Reference Laboratory	47	47	21	37	29	1,700
65	RF-SE-09-XX	Reference Laboratory	45	45	23	42	32	1,900
65	RF-SE-11-XX	Reference Laboratory	52	52	20	40	29	1,800
65	RF-SE-17-XX	Reference Laboratory	20	20	22	40	30	1,800
65	RF-SE-29-XX	Reference Laboratory	20	20	26	44	35	2,100
65	RF-SE-37-XX	Reference Laboratory	22	22	23	44	32	1,900
65	RF-SE-50-XX	Reference Laboratory	19	19	20	38	29	1,700
65	RF-SE-01-IX	Innov-X Systems, Inc. 35kv lamp	74	74	28	57	57	1,993
65	RF-SE-09-IX	Innov-X Systems, Inc. 35kv lamp	97	97	31	51	60	2,211
65	RF-SE-11-IX	Innov-X Systems, Inc. 35kv lamp	79	79	31	69	56	2,083
65	RF-SE-17-IX	Innov-X Systems, Inc. 35kv lamp	89	89	29	47	53	1,961
65	RF-SE-29-IX	Innov-X Systems, Inc. 35kv lamp	97	97	32	59	48	2,136
65	RF-SE-37-IX	Innov-X Systems, Inc. 35kv lamp	95	95	26	61	54	2,050
65	RF-SE-50-IX	Innov-X Systems, Inc. 35kv lamp	77	77	30	60	61	1,999
66	RF-SE-08-XX	Reference Laboratory	29	250	42	0.39 U	120	120
66	RF-SE-10-XX	Reference Laboratory	27	220	39	0.34 U	100	110
66	RF-SE-33-XX	Reference Laboratory	28	240	41	0.33 U	120	130
66	RF-SE-08-IX	Innov-X Systems, Inc. 35kv lamp	127	368	52	27	99	176
66	RF-SE-10-IX	Innov-X Systems, Inc. 35kv lamp	120	362	50	25	88	169
66	RF-SE-33-IX	Innov-X Systems, Inc. 35kv lamp	118	363	53	8	104	180
67	RF-SE-16-XX	Reference Laboratory	260	1,700 J-	1.2 U	130 J-	32 J-	760 J-
67	RF-SE-41-XX	Reference Laboratory	230	1,900	1.2 U	140	39	830
67	RF-SE-48-XX	Reference Laboratory	250	2,000	2.2	150	40	880
67	RF-SE-16-IX	Innov-X Systems, Inc. 35kv lamp	365	2,667	8	135	71	846
67	RF-SE-41-IX	Innov-X Systems, Inc. 35kv lamp	347	2,624	5	144	56	836
67	RF-SE-48-IX	Innov-X Systems, Inc. 35kv lamp	365	2,656	8	155	64	843

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Sb	As	Cd	Cr	Cu	Fe	Pb	Hg
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-IX	Innov-X Systems, Inc. 35kv lamp	344	684	859	1,235	97	20,581	943	963
68	RF-SE-35-IX	Innov-X Systems, Inc. 35kv lamp	335	661	777	1,164	77	20,285	885	877
68	RF-SE-54-IX	Innov-X Systems, Inc. 35kv lamp	309	669	809	1,256	94	20,835	930	924
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-IX	Innov-X Systems, Inc. 35kv lamp	378	922	483	91	99	22,874	32	65
69	RF-SE-46-IX	Innov-X Systems, Inc. 35kv lamp	366	924	498	102	93	23,067	33	70
69	RF-SE-51-IX	Innov-X Systems, Inc. 35kv lamp	374	916	486	114	88	22,615	36	57
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-IX	Innov-X Systems, Inc. 35kv lamp	211	110	1,981	123	1,201	24,101	2,614	507
70	RF-SE-40-IX	Innov-X Systems, Inc. 35kv lamp	192	102	2,055	113	1,257	24,141	2,670	517
70	RF-SE-47-IX	Innov-X Systems, Inc. 35kv lamp	215	93	2,012	111	1,216	23,711	2,582	477

Appendix D-1. Analytical Data Summary Innov-X XT400 with 35kV X-ray Tube and Reference Laboratory (Continued)

Blend No.	Sample ID	Source of Data	Ni	Se	Ag	V	Zn
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-IX	Innov-X Systems, Inc. 35kv lamp	538	172	215	149	162
68	RF-SE-35-IX	Innov-X Systems, Inc. 35kv lamp	535	161	197	164	154
68	RF-SE-54-IX	Innov-X Systems, Inc. 35kv lamp	566	172	208	166	170
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-IX	Innov-X Systems, Inc. 35kv lamp	1,838	367	51	51	1,302
69	RF-SE-46-IX	Innov-X Systems, Inc. 35kv lamp	1,893	364	52	54	1,311
69	RF-SE-51-IX	Innov-X Systems, Inc. 35kv lamp	1,819	356	68	62	1,267
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-IX	Innov-X Systems, Inc. 35kv lamp	312	543	332	98	149
70	RF-SE-40-IX	Innov-X Systems, Inc. 35kv lamp	351	557	331	100	161
70	RF-SE-47-IX	Innov-X Systems, Inc. 35kv lamp	301	537	306	88	153

Notes:

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

J Estimated concentration

J+ Concentration is considered estimated and biased high

J- Concentrations considered estimated and biased low

U Analyte is not detected; the associated concentration value is the sample reporting limit

APPENDIX E

STATISTICAL DATA SUMMARIES

Figure E-1: Linear Correlation Plot for Antimony

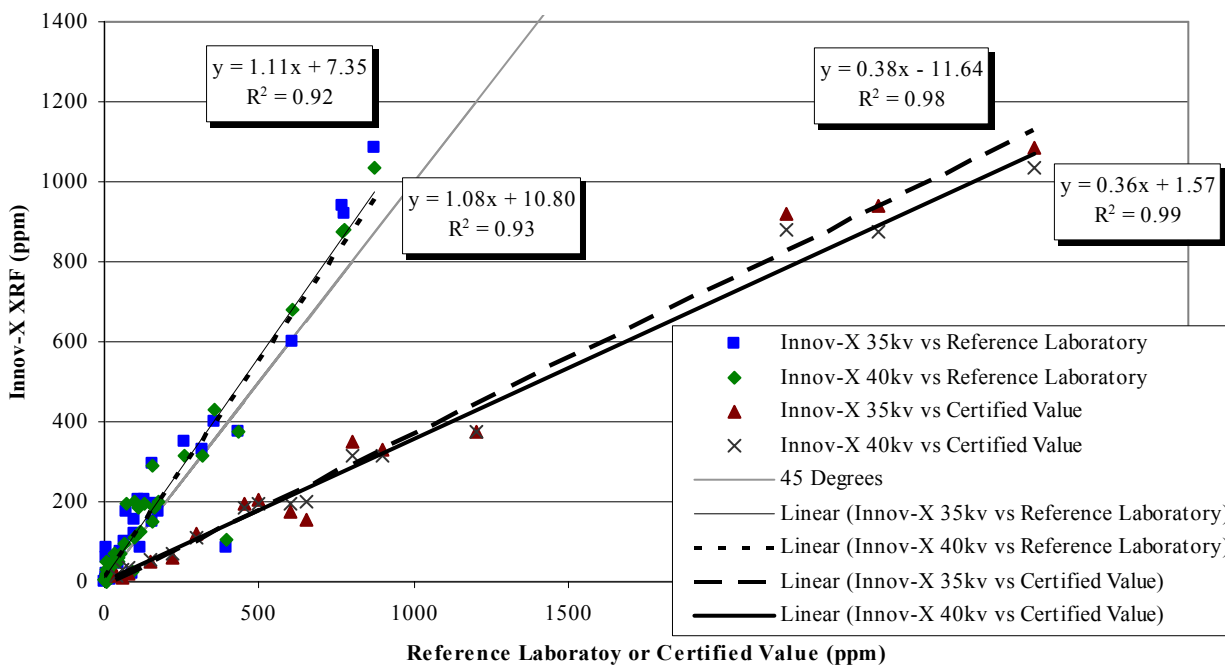


Figure E-2: Linear Correlation Plot for Arsenic

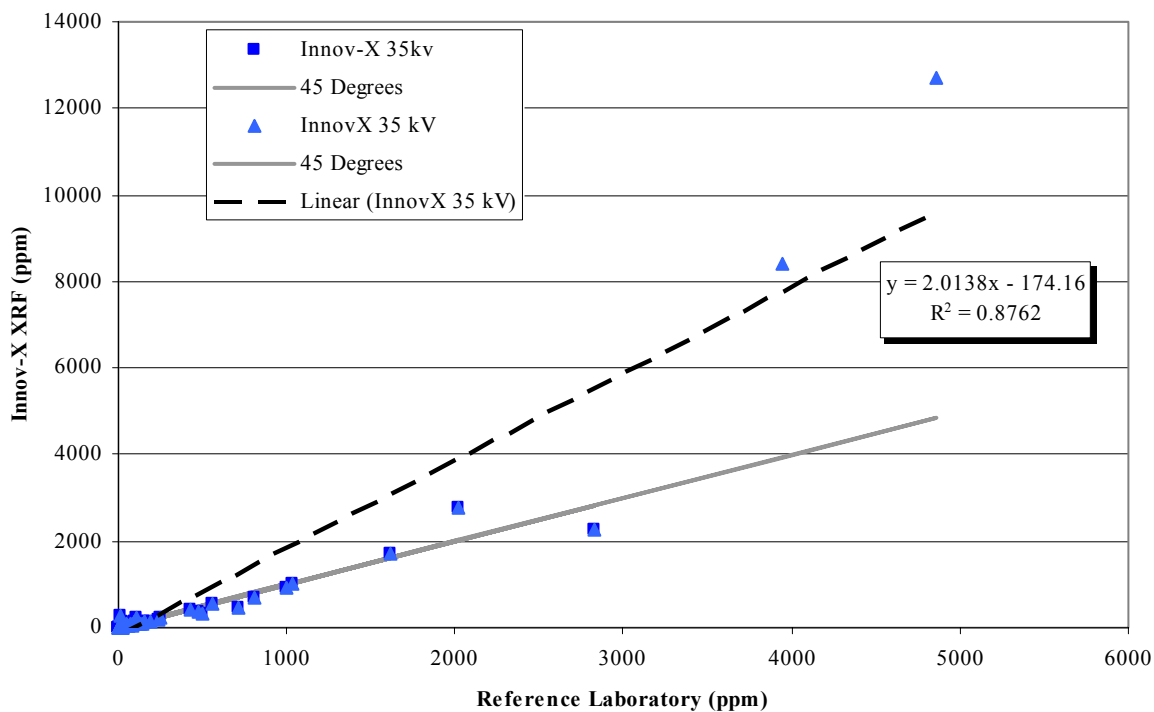


Figure E-3: Linear Correlation Plot for Cadmium

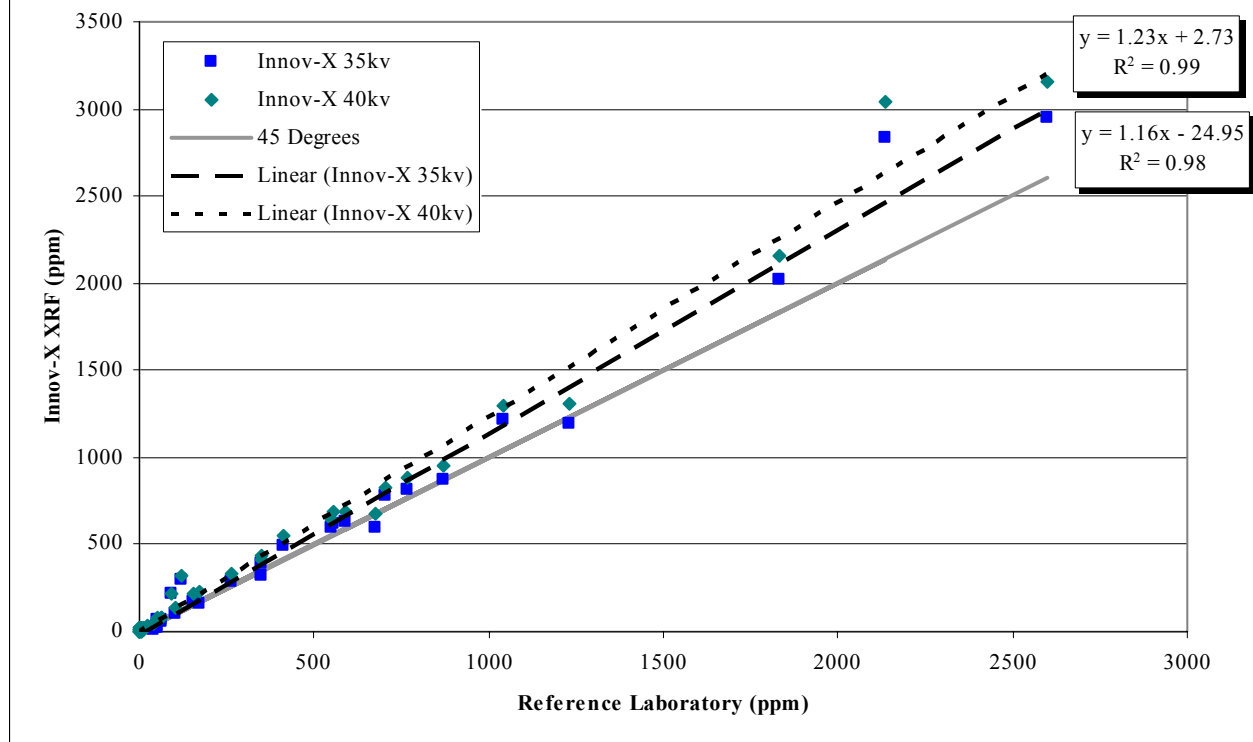


Figure E-4: Linear Correlation Plot for Chromium

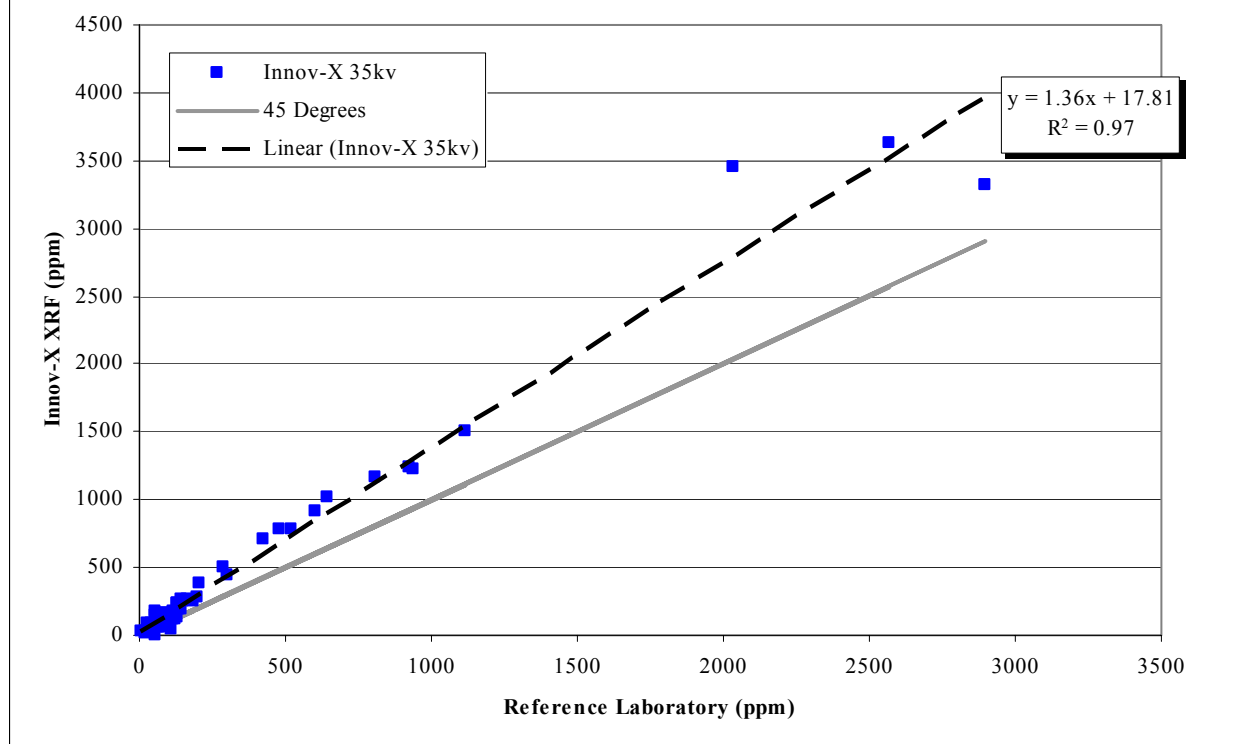


Figure E-5: Linear Correlation Plot for Copper

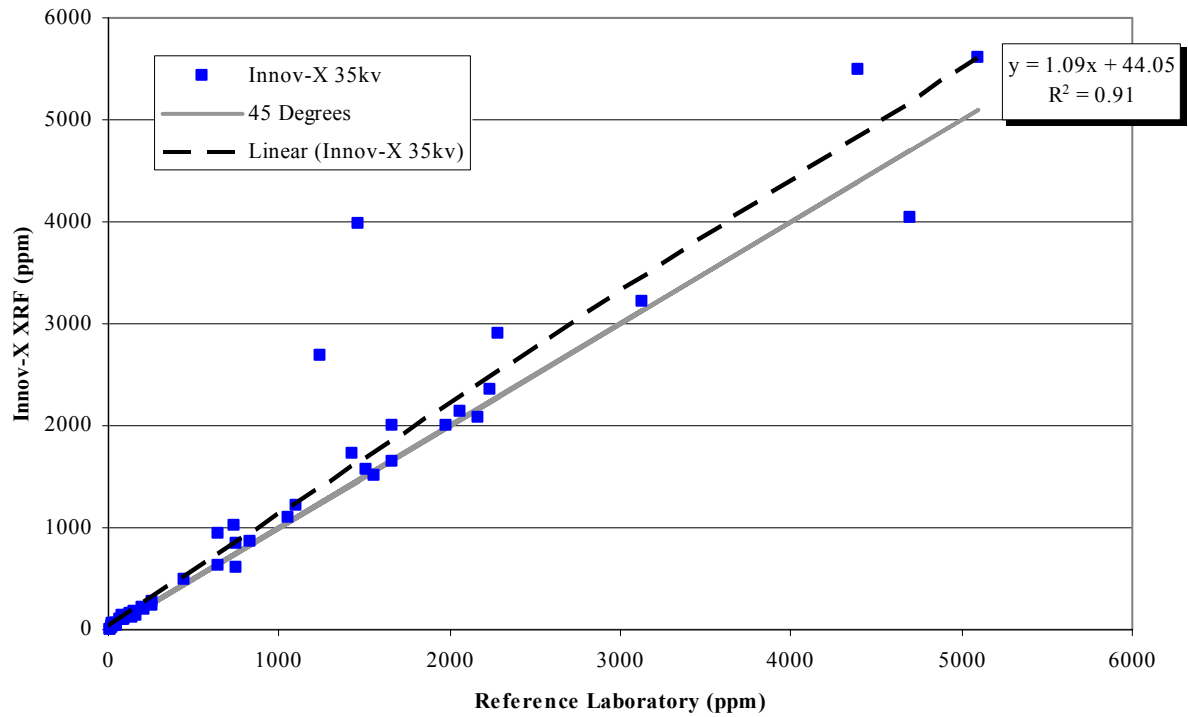


Figure E-6: Linear Correlation Plot for Iron

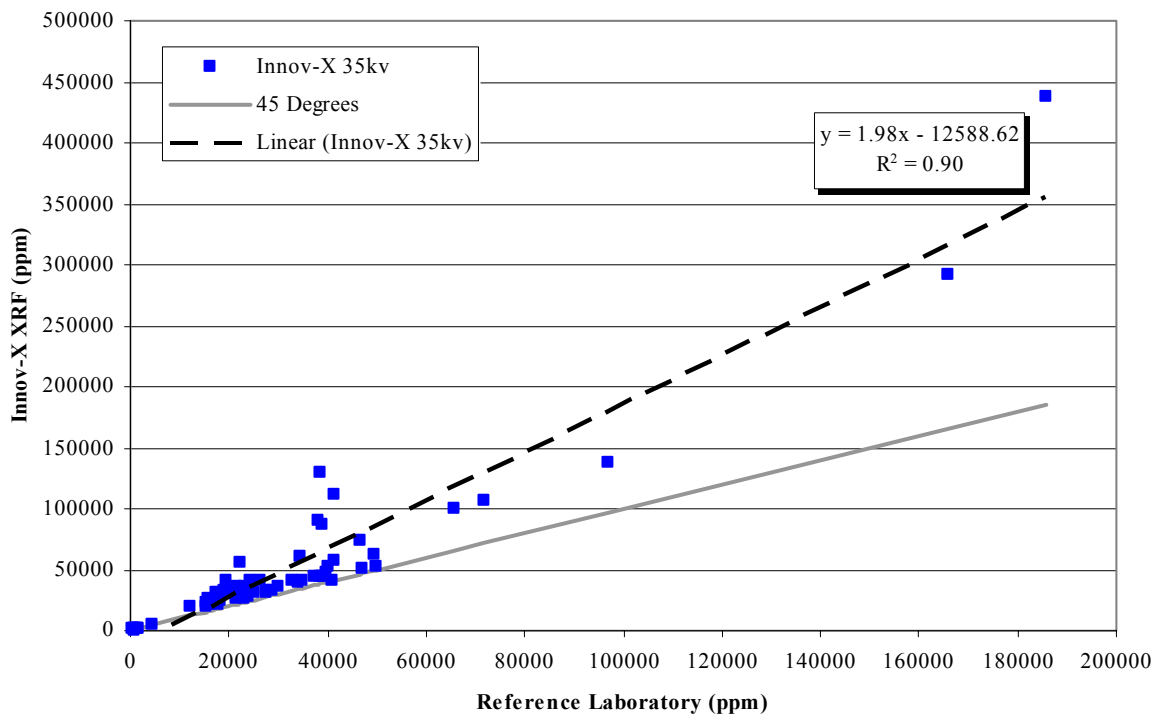


Figure E-7: Linear Correlation Plot for Lead

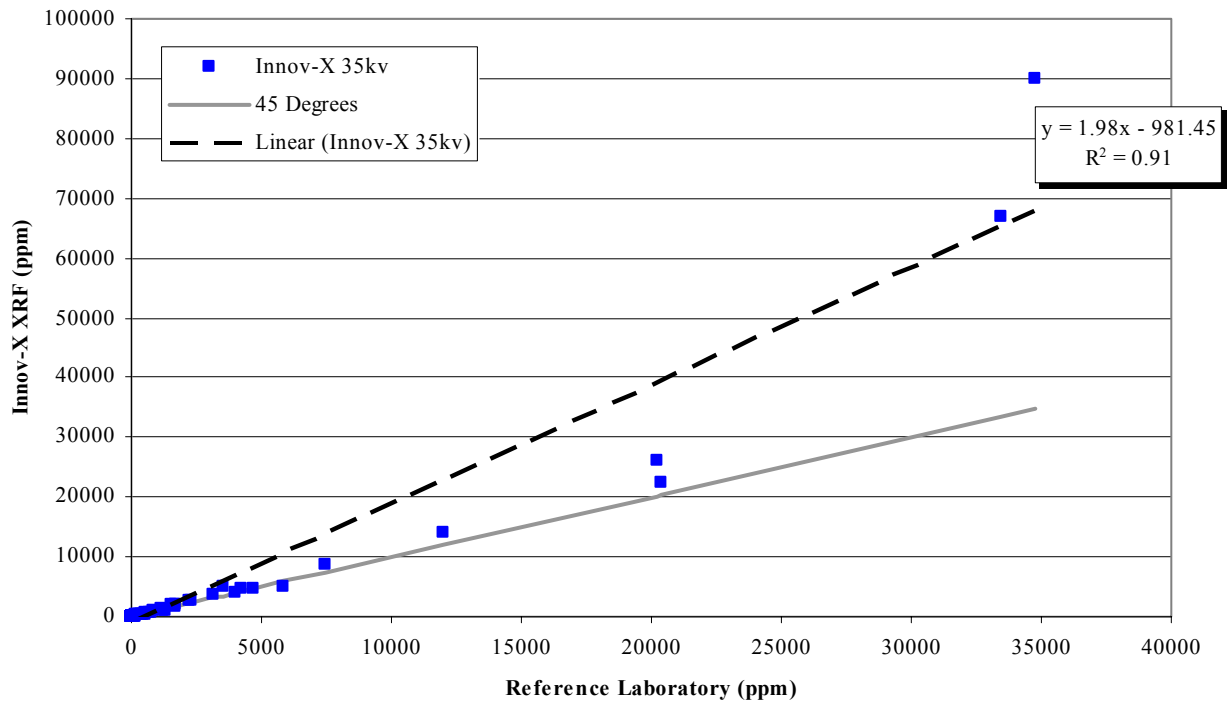


Figure E-8: Linear Correlation Plot for Mercury

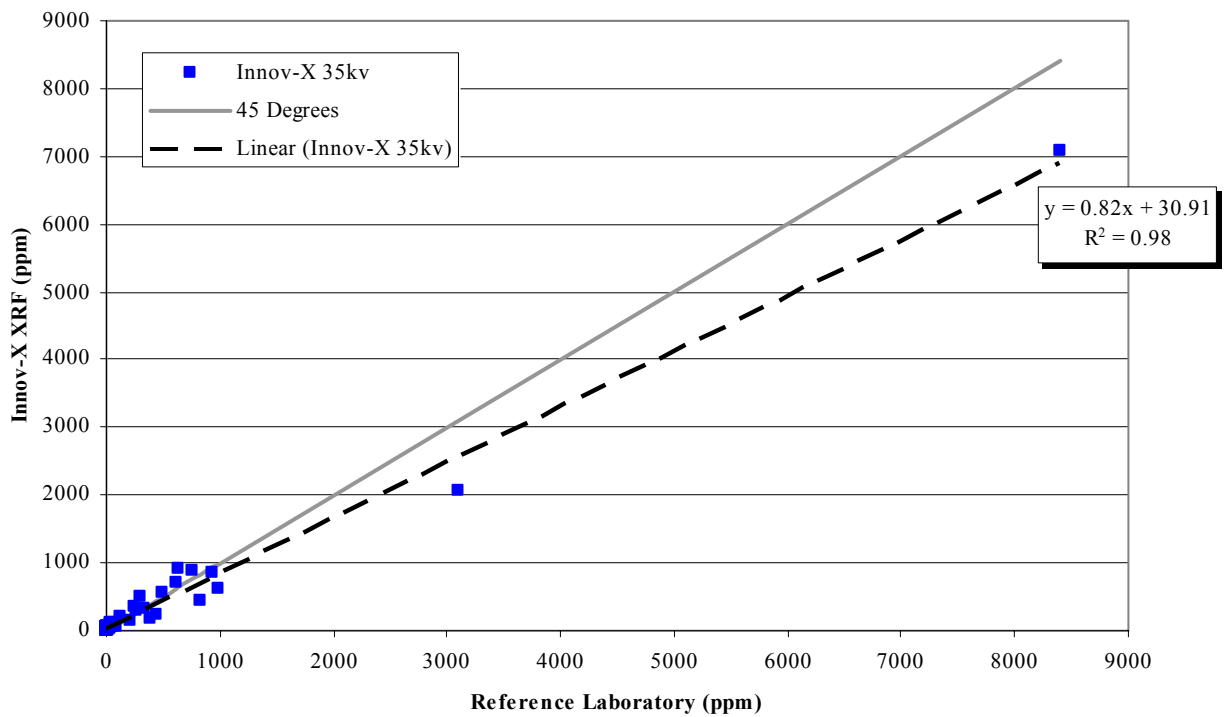


Figure E-9: Linear Correlation Plot for Nickel

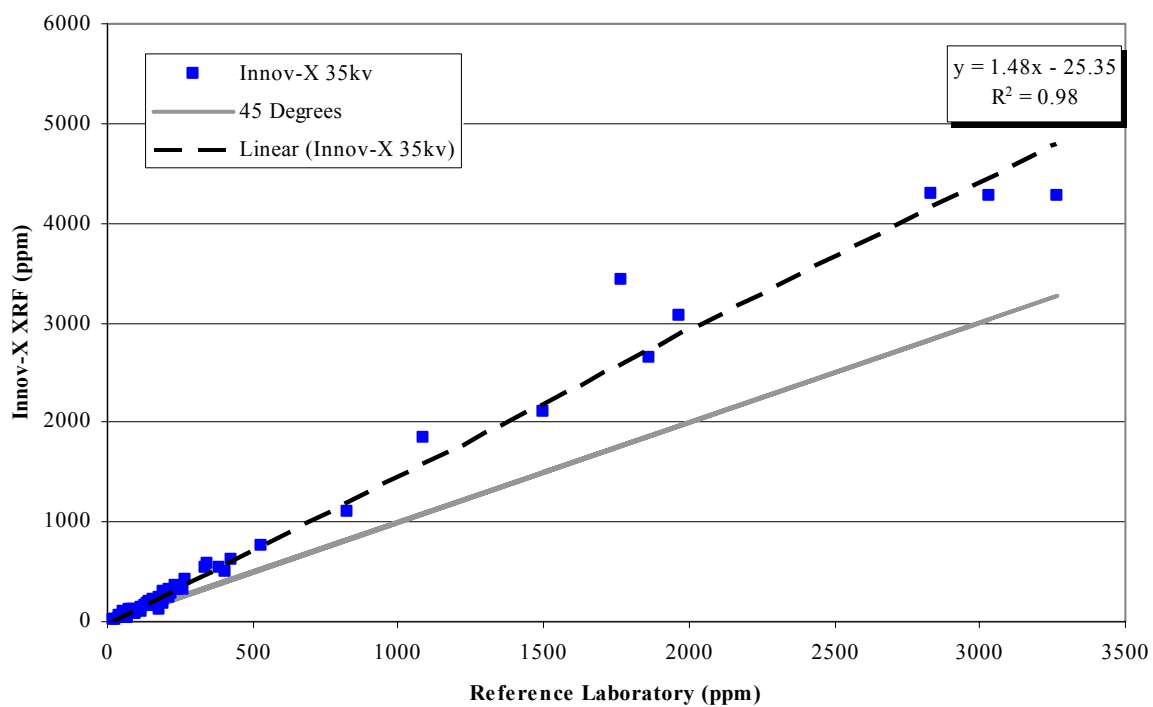


Figure E-10: Linear Correlation Plot for Selenium

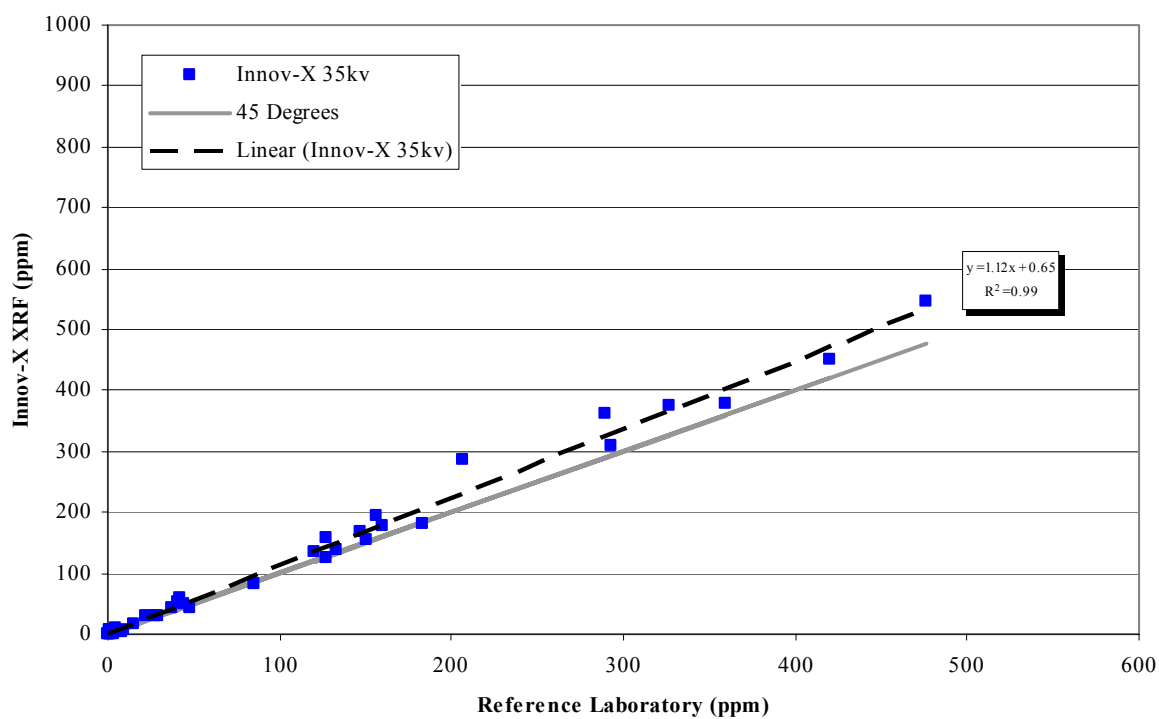


Figure E-11: Linear Correlation Plot for Silver

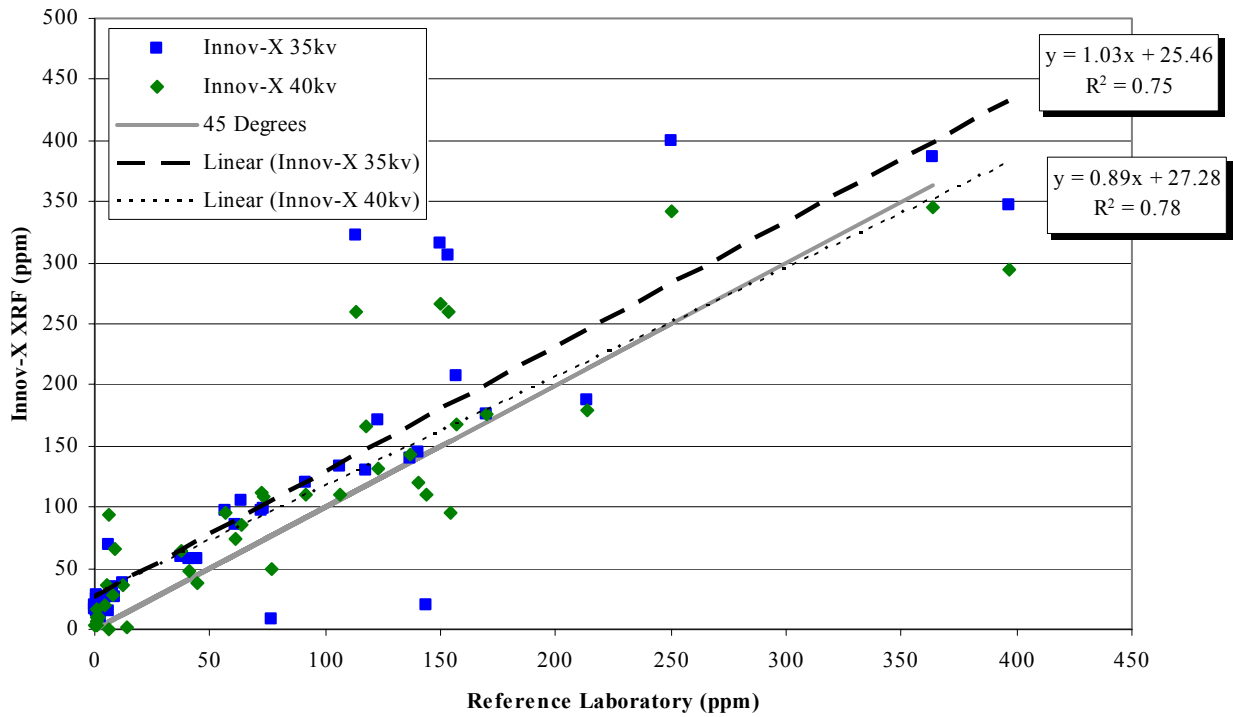


Figure E-12: Linear Correlation Plot for Vanadium

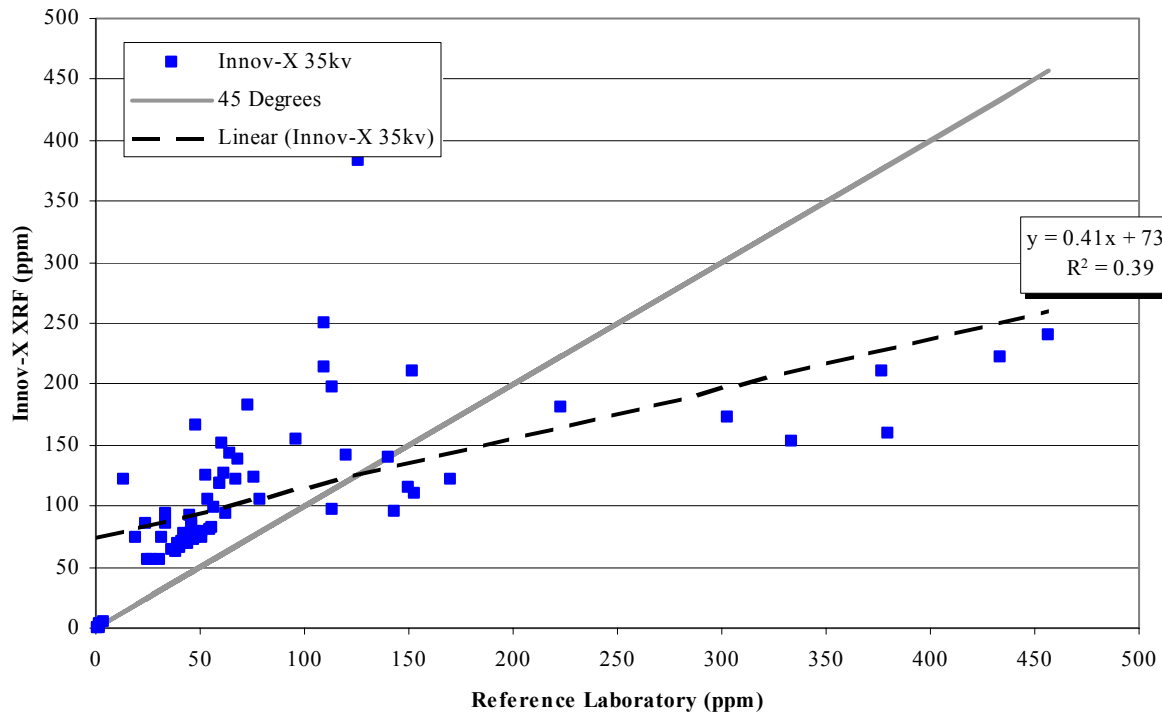
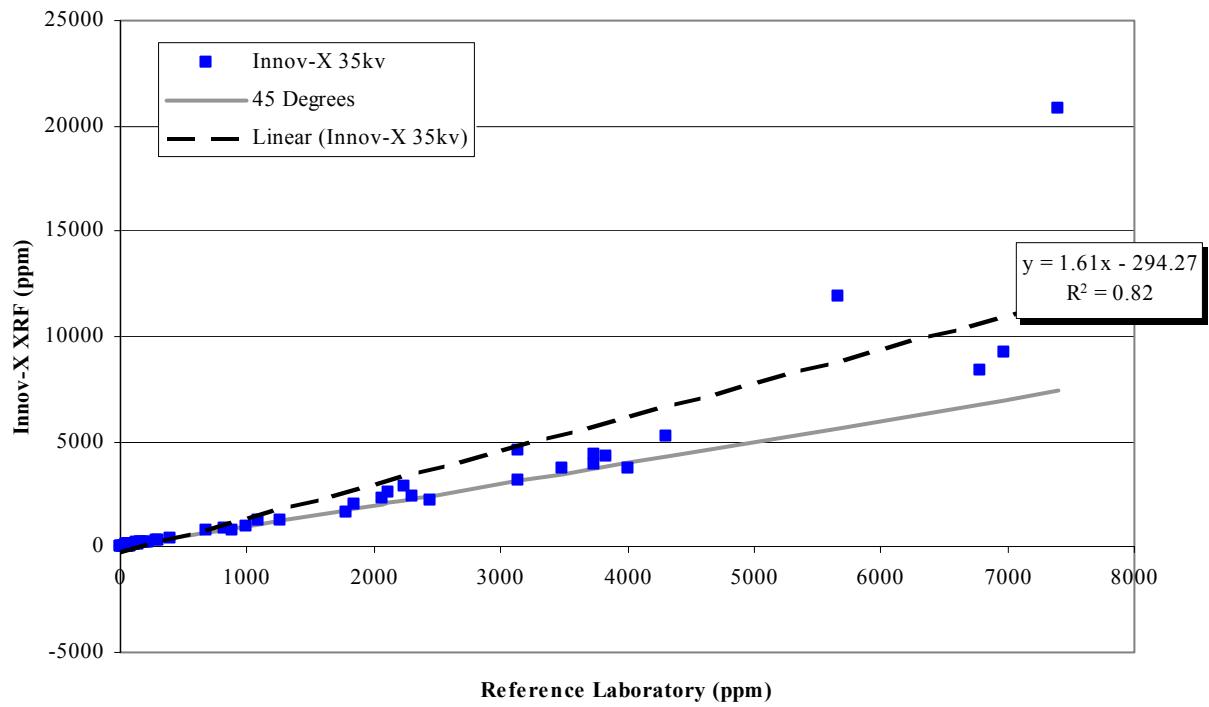
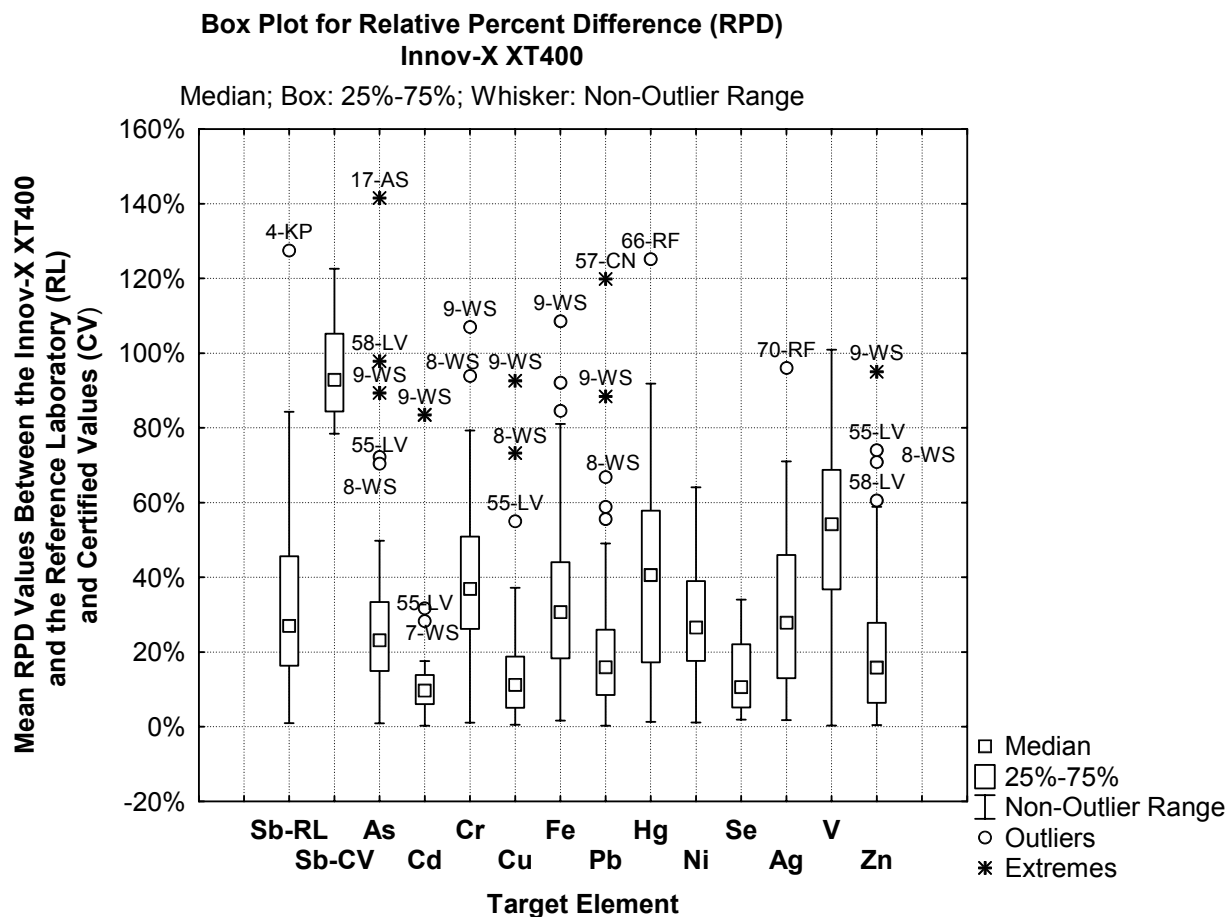


Figure E-13: Linear Correlation Plot for Zinc





Notes:

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

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

Figure E-14. Box and Whisker Plot for Mean RPD Values Showing Outliers and Extremes for Target Elements

Table E-1. Evaluation of Sensitivity - Method Detection Limits Calculated for the Innov-X with a 40 kV Lamp

Matrix	Blend No.	Antimony			Cadmium			Silver		
		MDL ²	Innov-X ³	Ref. Lab ⁴	MDL ²	Innov-X ³	Ref. Lab ⁴	MDL ²	Innov-X ³	Ref. Lab ⁴
Soil	2	15	9	17	34	3	ND	18	-26	ND
Soil	5	24	-1	ND	35	7	1.9	39	-68	0.93
Soil	6	20	8	8	31	15	12	33	-36	14
Soil	8	48	125	118	102	223	91	52	110	144
Soil	10	19	-3	ND	30	4	0.96	31	4	ND
Soil	12	19	97	62	43	328	263	47	65	38
Soil	18	19	6	ND	30	0	ND	22	-24	ND
Sediment	29	13	-5	ND	47	-1	ND	20	10	ND
Sediment	31	20	-2	ND	51	9	ND	111	94	6.2
Sediment	32	12	2	ND	50	4	ND	45	-7	ND
Sediment	39	11	-5	ND	40	-1	ND	52	-7	ND
Sediment	65	27	15	11	32	62	44	22	47	41
Mean		21			44			41		

Notes:

1. Bolded cells show calculated MDLs.
2. Detection limits and concentrations are milligrams per kilogram (mg/kg) and parts per million (ppm).
3. This column reports the mean concentration reported for this blend by the XT400.
4. This column reports the mean concentration reported for this blend by the Reference Laboratory.
5. To Increase the number of calculated MDLs for this element, this blend was included despite the fact that detections were reported by the vendor for only six of the seven replicates. This mean concentration and the corresponding MDL were calculated using the six replicated detected concentrations.

kV Kilovolt

MDL Method detection limit

NC MDL not calculated due to reference laboratory concentrations greater than 250 ppm or insufficient number of detected concentrations.

ND One or more results for this blend were reported as "Not Detected". Blends with one or more ND result as reported by the XRF were not used for MDL calculation for this element.

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Innov-X XT400 (35kV X-Ray Tube)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel
			Ref Lab	ERA Spike								
Soil	Level 1	Number	6	--	15	5	28	16	5	15	6	19
		Minimum	4.2%	--	3.9%	2.3%	1.1%	1.5%	2.9%	3.0%	22.8%	1.1%
		Maximum	127.5%	--	141.6%	83.5%	107.0%	54.9%	27.1%	119.8%	77.9%	60.1%
		Mean	44.1%	--	34.0%	42.3%	46.6%	14.9%	13.8%	24.9%	48.2%	23.0%
		Median	34.1%	--	23.0%	31.6%	50.2%	12.7%	13.0%	20.1%	45.5%	23.7%
	Level 2	Number	5	1	4	7	4	8	13	4	7	5
		Minimum	1.8%	79.9%	3.1%	0.3%	36.6%	12.7%	14.8%	3.3%	1.3%	21.9%
		Maximum	62.4%	79.9%	31.0%	28.3%	51.9%	92.6%	72.1%	23.1%	62.8%	46.8%
		Mean	30.7%	79.9%	11.0%	11.3%	43.4%	38.9%	37.0%	15.4%	33.0%	35.1%
		Median	18.6%	79.9%	5.0%	9.9%	42.6%	27.7%	36.9%	17.5%	38.8%	38.5%
	Level 3	Number	4	3	4	2	2	2	13	8	2	6
		Minimum	1.0%	81.8%	23.1%	3.0%	13.7%	9.5%	10.2%	1.1%	17.2%	26.9%
		Maximum	21.4%	93.9%	89.4%	12.5%	34.3%	21.9%	108.6%	35.9%	40.6%	64.1%
		Mean	15.0%	88.8%	54.1%	7.8%	24.0%	15.7%	27.4%	13.3%	28.9%	40.7%
		Median	18.8%	90.6%	51.9%	7.8%	24.0%	15.7%	16.5%	12.1%	28.9%	37.6%
	Level 4	Number	--	--	--	--	--	--	7	5	--	--
		Minimum	--	--	--	--	--	--	1.6%	9.2%	--	--
		Maximum	--	--	--	--	--	--	92.1%	88.4%	--	--
		Mean	--	--	--	--	--	--	29.7%	41.2%	--	--
		Median	--	--	--	--	--	--	21.4%	26.0%	--	--
	All Soil	Number	15	4	23	14	34	26	38	32	15	30
		Minimum	1.0%	79.9%	3.1%	0.3%	1.1%	1.5%	1.6%	1.1%	1.3%	1.1%
		Maximum	127.5%	93.9%	141.6%	83.5%	107.0%	92.6%	108.6%	119.8%	77.9%	64.1%
		Mean	31.9%	86.6%	33.5%	21.9%	44.9%	22.4%	29.3%	23.3%	38.5%	28.6%
		Median	20.0%	86.2%	23.1%	11.3%	44.9%	15.6%	22.4%	18.9%	40.6%	26.6%
Sediment	Level 1	Number	2	2	16	3	13	8	3	16	3	12
		Minimum	45.6%	102.7%	0.9%	3.0%	2.8%	3.8%	32.7%	0.3%	52.5%	1.2%
		Maximum	64.5%	113.0%	97.9%	7.2%	56.2%	28.3%	55.3%	58.8%	125.2%	39.5%
		Mean	55.0%	107.8%	26.4%	5.7%	23.4%	14.9%	41.6%	24.8%	89.9%	19.7%
		Median	55.0%	107.8%	25.5%	6.9%	18.2%	12.0%	36.8%	19.4%	91.8%	17.6%
	Level 2	Number	4	4	4	4	3	4	19	4	4	6
		Minimum	24.7%	84.4%	9.8%	7.1%	37.7%	1.8%	20.6%	0.7%	15.8%	24.2%
		Maximum	84.3%	122.6%	49.8%	17.6%	47.3%	10.7%	84.5%	12.0%	72.8%	51.4%
		Mean	49.4%	100.1%	28.7%	11.5%	41.6%	5.2%	36.7%	5.2%	43.4%	36.9%
		Median	44.4%	96.7%	27.5%	10.7%	39.8%	4.2%	31.1%	4.1%	42.5%	37.2%

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Innov-X XT400 (35kV X-Ray Tube) (Continued)

Matrix	Conc Range	Statistic	Selenium	Silver	Vanadium	Zinc
Soil	Level 1	Number	4	2	13	20
		Minimum	2.2%	28.1%	36.8%	0.6%
		Maximum	13.5%	30.0%	85.9%	74.0%
		Mean	7.9%	29.0%	60.9%	17.5%
		Median	7.9%	29.0%	66.0%	11.1%
	Level 2	Number	5	3	4	6
		Minimum	1.9%	1.8%	0.3%	0.4%
		Maximum	12.2%	26.4%	33.4%	37.0%
		Mean	6.5%	12.5%	19.2%	16.1%
		Median	4.7%	9.4%	21.5%	14.1%
	Level 3	Number	4	5	4	9
		Minimum	5.1%	6.1%	21.4%	4.6%
		Maximum	31.9%	71.0%	74.1%	95.0%
		Mean	14.0%	34.0%	51.7%	30.2%
		Median	9.4%	13.3%	55.6%	19.2%
	Level 4	Number	--	--	--	--
		Minimum	--	--	--	--
		Maximum	--	--	--	--
		Mean	--	--	--	--
		Median	--	--	--	--
	All Soil	Number	13	10	21	35
		Minimum	1.9%	1.8%	0.3%	0.4%
		Maximum	31.9%	71.0%	85.9%	95.0%
		Mean	9.2%	26.5%	51.2%	20.5%
		Median	5.7%	19.8%	56.6%	12.2%
Sediment	Level 1	Number	5	5	6	19
		Minimum	4.4%	25.0%	28.9%	2.2%
		Maximum	34.0%	52.6%	86.3%	60.5%
		Mean	20.1%	38.7%	50.5%	24.4%
		Median	23.9%	34.5%	47.2%	20.8%
	Level 2	Number	4	4	8	5
		Minimum	2.0%	3.3%	15.4%	1.2%
		Maximum	22.7%	32.6%	100.9%	17.7%
		Mean	15.1%	15.4%	52.2%	7.0%
		Median	17.9%	12.8%	47.2%	4.3%

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Innov-X XT400 (35kV X-Ray Tube) (Continued)

Matrix	Conc Range	Statistic	Antimony		Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel
			Ref Lab	ERA Spike								
	Level 3	Number	3	3	2	3	3	10	4	3	3	4
		Minimum	4.9%	78.4%	7.9%	3.7%	26.1%	0.5%	18.3%	11.2%	7.2%	28.3%
		Maximum	29.3%	105.2%	18.7%	9.5%	30.4%	18.1%	81.1%	25.8%	36.1%	52.3%
		Mean	16.4%	92.2%	13.3%	6.3%	28.7%	5.6%	49.0%	16.7%	18.8%	38.1%
		Median	15.1%	92.9%	13.3%	5.7%	29.6%	3.1%	48.4%	13.1%	13.1%	35.9%
	Level 4	Number	--	--	--	--	--	--	6	--	--	--
		Minimum	--	--	--	--	--	--	28.3%	--	--	--
		Maximum	--	--	--	--	--	--	80.7%	--	--	--
		Mean	--	--	--	--	--	--	45.9%	--	--	--
		Median	--	--	--	--	--	--	39.5%	--	--	--
	All Sediment	Number	9	9	22	10	19	22	32	23	10	22
		Minimum	4.9%	78.4%	0.9%	3.0%	2.8%	0.5%	18.3%	0.3%	7.2%	1.2%
		Maximum	84.3%	122.6%	97.9%	17.6%	56.2%	28.3%	84.5%	58.8%	125.2%	52.3%
		Mean	39.7%	99.2%	25.6%	8.2%	27.1%	8.9%	40.4%	20.4%	50.0%	27.7%
		Median	43.4%	102.7%	22.4%	7.2%	29.6%	5.1%	33.6%	13.1%	42.5%	25.5%
All Samples	XT400 (35 kv)	Number	24	13	45	24	53	48	70	55	25	52
		Minimum	1.0%	78.4%	0.9%	0.3%	1.1%	0.5%	1.6%	0.3%	1.3%	1.1%
		Maximum	127.5%	122.6%	141.6%	83.5%	107.0%	92.6%	108.6%	119.8%	125.2%	64.1%
		Mean	34.8%	95.3%	29.6%	16.2%	38.5%	16.2%	34.4%	22.1%	43.1%	28.2%
		Median	27.0%	92.9%	23.1%	9.7%	36.9%	11.2%	30.7%	16.0%	40.6%	26.5%
All Samples	All Instruments	Number	206	110	320	209	338	363	558	392	192	403
		Minimum	0.1%	0.1%	0.2%	0.1%	0.1%	0.2%	0.0%	0.1%	0.0%	0.3%
		Maximum	181.5%	162.0%	182.8%	168.1%	151.7%	111.1%	190.1%	135.2%	158.1%	146.5%
		Mean	80.6%	62.7%	36.6%	29.6%	30.8%	24.6%	35.4%	30.9%	62.5%	31.0%
		Median	84.3%	70.6%	26.2%	16.7%	26.0%	16.2%	26.0%	21.5%	58.6%	25.4%

Table E-2. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Innov-X XT400 (35kV X-Ray Tube) (Continued)

Matrix	Conc Range	Statistic	Selenium	Silver	Vanadium	Zinc
	Level 3	Number	3	3	3	4
		Minimum	6.8%	27.5%	62.0%	6.3%
		Maximum	22.1%	96.1%	81.7%	11.2%
		Mean	14.2%	56.5%	69.4%	8.1%
		Median	13.5%	46.0%	64.7%	7.5%
	Level 4	Number	--	--	--	--
		Minimum	--	--	--	--
		Maximum	--	--	--	--
		Mean	--	--	--	--
		Median	--	--	--	--
	All Sediment	Number	12	12	17	28
		Minimum	2.0%	3.3%	15.4%	1.2%
		Maximum	34.0%	96.1%	100.9%	60.5%
		Mean	17.0%	35.4%	54.6%	18.9%
		Median	17.9%	32.6%	53.8%	17.2%
All Samples	XT400 (35 kv)	Number	25	22	38	63
		Minimum	1.9%	1.8%	0.3%	0.4%
		Maximum	34.0%	96.1%	100.9%	95.0%
		Mean	12.9%	31.4%	52.7%	19.8%
		Median	10.6%	27.8%	54.2%	15.8%
All Samples	All Instruments	Number	195	177	218	471
		Minimum	0.0%	0.0%	0.1%	0.0%
		Maximum	127.1%	129.7%	129.5%	138.0%
		Mean	32.0%	36.0%	42.2%	26.3%
		Median	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.

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

ERA

kV

Number

RPD

Environmental Resource Associates, Inc.

Kilovolt.

Number of demonstration samples evaluated.

Relative percent difference

Table E-3. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Innov-X XT400 (40 kV X-ray Tube)

Matrix	Conc Range	Statistic	Antimony		Cadmium	Silver
			Ref Lab	ERA Spike		
Soil	Level 1	Number	8	--	7	2
		Minimum	2.9%	--	20.6%	38.0%
		Maximum	116.6%	--	89.3%	42.4%
		Mean	36.8%	--	44.7%	40.2%
		Median	29.0%	--	27.8%	40.2%
	Level 2	Number	5	1	7	3
		Minimum	14.1%	83.8%	0.4%	4.6%
		Maximum	60.7%	83.8%	34.9%	33.9%
		Mean	31.9%	83.8%	16.2%	18.8%
		Median	19.6%	83.8%	15.7%	17.9%
	Level 3	Number	4	3	2	7
		Minimum	11.1%	85.7%	8.9%	5.2%
		Maximum	17.2%	97.2%	19.1%	55.8%
		Mean	13.5%	93.1%	14.0%	33.4%
		Median	12.8%	96.3%	14.0%	29.5%
	Level 4	Number	--	--	--	--
		Minimum	--	--	--	--
		Maximum	--	--	--	--
		Mean	--	--	--	--
		Median	--	--	--	--
	All Soil	Number	17	4	16	12
		Minimum	2.9%	83.8%	0.4%	4.6%
		Maximum	116.6%	97.2%	89.3%	55.8%
		Mean	29.9%	90.8%	28.4%	30.9%
		Median	17.2%	91.0%	21.1%	31.7%
Sediment	Level 1	Number	3	3	3	3
		Minimum	55.7%	64.6%	26.3%	19.1%
		Maximum	81.0%	101.5%	32.9%	50.7%
		Mean	71.8%	86.9%	30.2%	32.9%
		Median	78.6%	94.4%	31.2%	28.7%

Table E-3. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Innov-X XT400 (40 kV X-ray Tube) (Continued)

Matrix	Conc Range	Statistic	Antimony		Cadmium	Silver
			Ref Lab	ERA Spike		
	Level 2	Number	4	4	4	4
		Minimum	15.0%	86.8%	15.4%	3.3%
		Maximum	91.8%	106.2%	29.2%	15.6%
		Mean	53.8%	96.9%	21.8%	7.2%
		Median	54.2%	97.3%	21.3%	5.0%
	Level 3	Number	3	3	3	3
		Minimum	0.6%	87.5%	5.8%	6.9%
		Maximum	18.6%	104.3%	16.3%	78.7%
		Mean	11.0%	96.0%	12.1%	38.9%
		Median	13.8%	96.2%	14.1%	31.1%
	Level 4	Number	--	--	--	--
		Minimum	--	--	--	--
		Maximum	--	--	--	--
		Mean	--	--	--	--
		Median	--	--	--	--
	All Sediment	Number	10	10	10	10
		Minimum	0.6%	64.6%	5.8%	3.3%
		Maximum	91.8%	106.2%	32.9%	78.7%
		Mean	46.3%	93.6%	21.4%	24.4%
		Median	48.2%	95.3%	21.3%	17.4%

Table E-3. Evaluation of Accuracy - Relative Percent Differences Versus Reference Laboratory Data Calculated for the Innov-X XT400 (40 kV X-ray Tube) (Continued)

Matrix	Conc Range	Statistic	Antimony		Cadmium	Silver
			Ref Lab	ERA Spike		
All Samples	XT400	Number	27	14	26	22
		Minimum	0.6%	64.6%	0.4%	3.3%
		Maximum	116.6%	106.2%	89.3%	78.7%
		Mean	36.0%	92.8%	25.7%	27.9%
		Median	19.6%	95.3%	21.1%	28.0%
All Samples	All Instruments	Number	206	110	209	177
		Minimum	0.1%	0.1%	0.1%	0.0%
		Maximum	181.5%	162.0%	168.1%	129.7%
		Mean	80.6%	62.7%	29.6%	36.0%
		Median	84.3%	70.6%	16.7%	28.7%

Notes:

All RPDs presented in this table are absolute values.

-- No samples reported by the reference laboratory in this concentration range.

Conc Concentration.

ERA Environmental Resource Associates, Inc.

kV Kilovolt.

Number Number of demonstration samples evaluated.

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

RPD Relative percent difference.

Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated for the Innov-X XT400 (35 kV X-ray Tube)

Matrix	Concentration Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
Soil	Low	Number	6	15	5	28	16	5	15	6	19	4
		Minimum	13.9%	3.1%	2.0%	1.9%	2.4%	2.1%	1.0%	4.0%	1.6%	1.8%
		Maximum	40.2%	23.2%	21.4%	35.2%	16.2%	5.3%	13.9%	10.0%	29.8%	10.2%
		Mean	25.7%	8.6%	11.3%	11.9%	7.8%	3.6%	6.5%	6.8%	11.2%	5.5%
		Median	22.1%	6.8%	8.2%	11.8%	7.3%	3.9%	4.7%	7.0%	9.7%	5.0%
	Medium	Number	5	4	7	4	8	13	4	7	5	5
		Minimum	2.6%	2.5%	1.5%	3.2%	1.4%	0.4%	1.2%	2.6%	2.3%	1.2%
		Maximum	7.2%	10.9%	3.8%	6.1%	14.1%	11.3%	7.0%	5.7%	17.9%	3.1%
		Mean	5.5%	4.8%	2.3%	4.1%	4.6%	2.1%	2.7%	4.0%	7.4%	2.4%
		Median	6.1%	2.9%	2.0%	3.5%	2.3%	1.4%	1.4%	4.4%	4.4%	2.3%
	High	Number	4	4	2	2	2	13	8	2	6	4
		Minimum	0.8%	2.1%	2.2%	2.1%	1.3%	0.4%	0.7%	2.1%	1.5%	1.0%
		Maximum	3.8%	3.1%	2.5%	4.0%	3.9%	2.1%	4.3%	2.1%	5.1%	3.1%
		Mean	2.4%	2.5%	2.3%	3.1%	2.6%	1.3%	2.3%	2.1%	3.0%	2.2%
		Median	2.4%	2.4%	2.3%	3.1%	2.6%	1.3%	2.0%	2.1%	2.4%	2.3%
	Very High	Number	--	--	--	--	--	7	5	--	--	--
		Minimum	--	--	--	--	--	1.1%	0.9%	--	--	--
		Maximum	--	--	--	--	--	7.0%	3.7%	--	--	--
		Mean	--	--	--	--	--	2.9%	2.1%	--	--	--
		Median	--	--	--	--	--	1.7%	2.1%	--	--	--
	All Soil	Number	15	23	14	34	26	38	32	15	30	13
		Minimum	0.8%	2.1%	1.5%	1.9%	1.3%	0.4%	0.7%	2.1%	1.5%	1.0%
		Maximum	40.2%	23.2%	21.4%	35.2%	16.2%	11.3%	13.9%	10.0%	29.8%	10.2%
		Mean	12.8%	6.8%	5.5%	10.5%	6.4%	2.2%	4.3%	4.9%	8.9%	3.3%
		Median	6.3%	4.4%	2.5%	8.9%	5.8%	1.6%	3.3%	4.4%	7.5%	2.4%

**Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated for the Innov-X XT400 (35 kV X-ray Tube)
(Continued)**

Matrix	Concentration Range	Statistic	Silver	Vanadium	Zinc
Soil	Low	Number	2	13	20
		Minimum	8.8%	4.4%	1.3%
		Maximum	11.2%	10.9%	19.1%
		Mean	10.0%	8.4%	6.5%
		Median	10.0%	8.5%	4.8%
	Medium	Number	3	4	6
		Minimum	5.7%	4.8%	1.4%
		Maximum	8.6%	6.4%	7.6%
		Mean	7.1%	5.8%	3.4%
		Median	7.1%	6.0%	2.9%
	High	Number	5	4	9
		Minimum	2.1%	2.1%	0.9%
		Maximum	7.6%	7.8%	3.3%
		Mean	4.8%	4.9%	2.0%
		Median	4.9%	4.9%	1.9%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Soil	Number	10	21	35
		Minimum	2.1%	2.1%	0.9%
		Maximum	11.2%	10.9%	19.1%
		Mean	6.6%	7.2%	4.8%
		Median	7.0%	7.8%	3.3%

**Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated for the Innov-X XT400 (35 kV X-ray Tube)
(Continued)**

Matrix	Concentration Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
Sediment	Low	Number	2	16	3	13	8	3	16	3	12	5
		Minimum	22.8%	1.3%	7.5%	5.5%	2.6%	2.9%	0.8%	3.9%	3.0%	0.7%
		Maximum	45.5%	13.3%	14.1%	22.7%	12.2%	9.1%	41.0%	11.7%	20.8%	7.6%
		Mean	34.1%	6.9%	11.5%	13.1%	6.3%	6.9%	8.7%	7.8%	10.5%	4.2%
		Median	34.1%	5.0%	12.9%	12.6%	6.0%	8.5%	5.0%	7.8%	9.0%	3.2%
	Medium	Number	4	4	4	3	4	19	4	4	6	4
		Minimum	3.6%	1.9%	1.5%	1.8%	1.1%	0.2%	2.1%	2.4%	1.0%	1.2%
		Maximum	16.5%	5.8%	4.9%	11.1%	2.4%	6.4%	3.7%	4.2%	8.7%	3.7%
		Mean	7.8%	3.7%	3.1%	6.4%	1.9%	2.1%	2.9%	3.4%	4.0%	2.1%
		Median	5.5%	3.6%	3.1%	6.3%	2.2%	1.8%	2.9%	3.6%	2.6%	1.7%
	High	Number	3	2	3	3	10	4	3	3	4	3
		Minimum	1.6%	0.4%	1.8%	0.8%	0.7%	1.0%	1.0%	1.9%	0.8%	1.6%
		Maximum	5.6%	1.8%	5.1%	3.9%	11.0%	4.5%	1.7%	4.7%	4.4%	2.0%
		Mean	3.8%	1.1%	3.4%	2.2%	3.0%	2.0%	1.3%	3.2%	2.7%	1.8%
		Median	4.2%	1.1%	3.1%	1.9%	2.3%	1.3%	1.2%	3.1%	2.7%	1.8%
	Very High	Number	--	--	--	--	--	6	--	--	--	--
		Minimum	--	--	--	--	--	0.7%	--	--	--	--
		Maximum	--	--	--	--	--	1.5%	--	--	--	--
		Mean	--	--	--	--	--	1.1%	--	--	--	--
		Median	--	--	--	--	--	1.2%	--	--	--	--
	All Sediment	Number	9	22	10	19	22	32	23	10	22	12
		Minimum	1.6%	0.4%	1.5%	0.8%	0.7%	0.2%	0.8%	1.9%	0.8%	0.7%
		Maximum	45.5%	13.3%	14.1%	22.7%	12.2%	9.1%	41.0%	11.7%	20.8%	7.6%
		Mean	12.3%	5.8%	5.7%	10.3%	4.0%	2.4%	6.7%	4.7%	7.3%	2.9%
		Median	5.6%	4.7%	4.7%	11.1%	2.7%	1.4%	3.7%	4.1%	7.0%	1.9%

**Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated for the Innov-X XT400 (35 kV X-ray Tube)
(Continued)**

Matrix	Concentration Range	Statistic	Silver	Vanadium	Zinc
Sediment	Low	Number	5	6	19
		Minimum	8.8%	3.8%	0.7%
		Maximum	16.6%	15.5%	8.9%
		Mean	11.8%	10.1%	4.7%
		Median	11.6%	9.6%	4.3%
	Medium	Number	4	8	5
		Minimum	1.4%	0.9%	0.6%
		Maximum	7.1%	8.5%	5.3%
		Mean	5.0%	6.1%	3.0%
		Median	5.8%	6.7%	3.0%
	High	Number	3	3	4
		Minimum	4.2%	2.6%	1.2%
		Maximum	4.6%	5.9%	4.3%
		Mean	4.4%	4.0%	2.9%
		Median	4.4%	3.5%	3.0%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Sediment	Number	12	17	28
		Minimum	1.4%	0.9%	0.6%
		Maximum	16.6%	15.5%	8.9%
		Mean	7.7%	7.2%	4.1%
		Median	6.9%	7.0%	4.2%

Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated for the Innov-X XT400 (35 kV X-ray Tube)
(Continued)

Matrix	Concentration Range	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium
All Samples	XT400	Number	24	45	24	53	48	70	55	25	52	25
		Minimum	0.8%	0.4%	1.5%	0.8%	0.7%	0.2%	0.7%	1.9%	0.8%	0.7%
		Maximum	45.5%	23.2%	21.4%	35.2%	16.2%	11.3%	41.0%	11.7%	29.8%	10.2%
		Mean	12.6%	6.3%	5.6%	10.4%	5.3%	2.3%	5.3%	4.8%	8.3%	3.1%
		Median	6.2%	4.5%	2.8%	9.3%	3.6%	1.5%	3.5%	4.2%	7.4%	2.3%
All Samples	All Instruments	Number	206	320	209	338	363	558	392	192	403	195
		Minimum	0.5%	0.2%	0.4%	0.6%	0.1%	0.1%	0.2%	1.0%	0.3%	0.1%
		Maximum	97.7%	71.7%	92.8%	116.3%	58.3%	101.8%	115.6%	137.1%	164.2%	98.8%
		Mean	8.9%	11.2%	8.2%	15.9%	7.5%	5.2%	9.3%	14.3%	10.8%	7.2%
		Median	6.1%	8.2%	3.6%	12.1%	5.1%	2.2%	4.9%	6.8%	7.0%	4.5%

**Table E-4. Evaluation of Precision - Relative Standard Deviations Calculated for the Innov-X XT400 (35 kV X-ray Tube)
(Continued)**

Matrix	Concentration Range	Statistic	Silver	Vanadium	Zinc
All Samples	XT400	Number	22	38	63
		Minimum	1.4%	0.9%	0.6%
		Maximum	16.6%	15.5%	19.1%
		Mean	7.2%	7.2%	4.5%
		Median	7.0%	7.1%	3.7%
All Samples	All Instruments	Number	177	218	471
		Minimum	0.6%	0.4%	0.1%
		Maximum	125.3%	86.1%	192.9%
		Mean	10.3%	12.5%	8.0%
		Median	5.2%	8.5%	5.3%

Notes:

-- No samples reported by the reference laboratory in this concentration range.
kV Kilovolt.
Number Number of demonstration samples evaluated.
RSD Relative standard deviation.

**Table E-5. Evaluation of Precision - Relative Standard Deviations
Calculated for the Innov-X XT400 (40 kV X-ray Tube)**

Matrix	Concentration Range	Statistic	Antimony	Cadmium	Silver
Soil	Low	Number	8	7	2
		Minimum	6.2%	3.3%	5.2%
		Maximum	19.4%	30.2%	13.1%
		Mean	12.3%	12.1%	9.1%
		Median	10.9%	9.4%	9.1%
	Medium	Number	5	7	3
		Minimum	1.2%	0.6%	6.5%
		Maximum	4.4%	3.4%	13.3%
		Mean	3.1%	1.5%	9.6%
		Median	3.8%	1.4%	9.0%
	High	Number	4	2	7
		Minimum	0.6%	0.6%	3.6%
		Maximum	2.1%	1.1%	36.8%
		Mean	1.3%	0.9%	10.6%
		Median	1.2%	0.9%	4.7%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Soil	Number	17	16	12
		Minimum	0.6%	0.6%	3.6%
		Maximum	19.4%	30.2%	36.8%
		Mean	7.0%	6.0%	10.1%
		Median	4.4%	2.6%	5.9%
Sediment	Low	Number	3	3	3
		Minimum	4.8%	1.2%	7.1%
		Maximum	6.0%	11.3%	23.5%
		Mean	5.5%	7.4%	13.8%
		Median	5.8%	9.7%	10.7%
	Medium	Number	4	4	4
		Minimum	3.7%	0.6%	3.3%
		Maximum	5.0%	5.8%	12.2%
		Mean	4.3%	2.4%	6.2%
		Median	4.2%	1.6%	4.6%
	High	Number	3	3	3
		Minimum	1.0%	1.2%	3.7%
		Maximum	3.2%	6.2%	11.2%
		Mean	1.9%	3.0%	6.3%
		Median	1.5%	1.5%	3.8%
	Very High	Number	--	--	--
		Minimum	--	--	--
		Maximum	--	--	--
		Mean	--	--	--
		Median	--	--	--
	All Sediment	Number	10	10	10
		Minimum	1.0%	0.6%	3.3%
		Maximum	6.0%	11.3%	23.5%
		Mean	3.9%	4.1%	8.5%
		Median	4.2%	1.8%	5.9%

Table E-5. Evaluation of Precision - Relative Standard Deviations Calculated for the Innov-X XT400 (40 kV X-ray Tube) (Continued)

Matrix	Concentration Range	Statistic	Antimony	Cadmium	Silver
All Samples	XT400	Number	27	26	22
		Minimum	0.6%	0.6%	3.3%
		Maximum	19.4%	30.2%	36.8%
		Mean	5.8%	5.3%	9.4%
		Median	4.4%	2.0%	5.9%
All Samples	All Instruments	Number	206	209	195
		Minimum	0.5%	0.4%	0.1%
		Maximum	97.7%	92.8%	98.8%
		Mean	8.9%	8.2%	7.2%
		Median	6.1%	3.6%	4.5%

Notes:

-- No samples reported by the reference laboratory in this concentration range.
kV Kilovolt.
Number Number of demonstration samples evaluated.
RSD Relative standard deviation.

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

Matrix	Statistic	Antimony	Arsenic	Cadmium	Chromium	Copper	Iron	Lead	Mercury	Nickel	Selenium	Silver
All Soil	Number	17	23	15	34	26	38	33	16	35	13	13
	Minimum	3.6%	1.4%	0.9%	1.4%	0.0%	1.6%	0.0%	0.0%	0.0%	0.0%	2.3%
	Maximum	38.0%	45.8%	21.4%	137.0%	21.0%	46.2%	150.0%	50.7%	44.9%	22.7%	37.1%
	Mean	14.3%	11.7%	11.1%	14.3%	10.1%	10.2%	17.6%	13.8%	11.4%	8.9%	12.4%
	Median	9.8%	12.4%	9.0%	10.6%	9.1%	8.7%	13.2%	6.6%	10.0%	7.1%	7.5%
All Sediment	Number	7	24	10	26	21	31	22	10	27	12	10
	Minimum	2.9%	2.4%	2.9%	4.6%	1.8%	2.7%	0.0%	2.8%	0.6%	1.3%	1.0%
	Maximum	33.6%	36.7%	37.5%	35.5%	38.8%	37.5%	41.1%	48.0%	35.8%	37.3%	21.3%
	Mean	14.4%	10.7%	11.4%	9.8%	9.7%	9.9%	11.6%	14.3%	9.4%	10.0%	9.4%
	Median	9.1%	9.2%	8.2%	7.5%	8.9%	8.1%	7.4%	6.9%	7.3%	7.6%	6.6%
All	Number	24	47	25	60	47	69	55	26	62	25	23
	Minimum	2.9%	1.4%	0.9%	1.4%	0.0%	1.6%	0.0%	0.0%	0.0%	0.0%	1.0%
	Maximum	38.0%	45.8%	37.5%	137.0%	38.8%	46.2%	150.0%	50.7%	44.9%	37.3%	37.1%
	Mean	14.3%	11.2%	11.2%	12.4%	9.9%	10.1%	15.2%	14.0%	10.6%	9.4%	11.1%
	Median	9.5%	9.5%	9.0%	8.4%	8.9%	8.5%	8.6%	6.6%	8.2%	7.4%	7.1%

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

Matrix	Statistic	Vanadium	Zinc
All Soil	Number	21	35
	Minimum	0.0%	1.0%
	Maximum	18.1%	46.5%
	Mean	8.4%	10.4%
	Median	6.6%	9.1%
All Sediment	Number	17	27
	Minimum	2.2%	1.4%
	Maximum	21.9%	35.8%
	Mean	8.4%	8.9%
	Median	8.1%	6.9%
All	Number	38	62
	Minimum	0.0%	1.0%
	Maximum	21.9%	46.5%
	Mean	8.4%	9.8%
	Median	7.2%	7.4%

Table E-7. 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	9	39	5	8	40	1	8
RPD of Target Element ²	Minimum	-35.6%	-89.4%	-141.6%	-64.1%	-42.5%	-60.1%	-92.6%	-13.4%	-54.9%
	Maximum	49.8%	31.0%	23.1%	34.3%	16.6%	26.7%	21.1%	-13.4%	1.5%
	Mean	18.3%	-15.1%	-50.4%	-25.2%	-13.7%	-27.1%	-10.2%	-13.4%	-20.8%
	Median	20.2%	3.1%	-39.9%	-26.4%	-12.2%	-28.1%	-5.1%	-13.4%	-17.7%
RPD of Target Element ² (Absolute Value)	Minimum	0.9%	3.1%	16.9%	1.1%	6.7%	17.6%	0.5%	13.4%	1.5%
	Maximum	49.8%	89.4%	141.6%	64.1%	42.5%	60.1%	92.6%	13.4%	54.9%
	Mean	20.9%	32.7%	55.5%	28.1%	20.4%	33.8%	15.2%	13.4%	21.2%
	Median	20.7%	23.0%	39.9%	26.9%	16.6%	28.8%	10.5%	13.4%	17.7%
Interferent Concentration Range	Minimum	11	667	1902	11	848	1512	38	546	1850
	Maximum	919	89940	26229	1225	1998	5607	1099	546	4293
	Mean	167	26942	5651	163	1276	3040	220	546	3247
	Median	50	8536	2695	99	1106	2515	167	546	3254
Target Element Concentration Range	Minimum	7	92	66	34	98	73	30	89	93
	Maximum	2247	12711	2778	4293	364	625	5607	89	157
	Mean	231	3543	419	890	224	257	1367	89	124
	Median	86	1006	135	246	198	193	903	89	124

Table E-7. 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	-73.3%	-92.6%	-28.3%	-95.0%	-30.5%	-58.9%
	Maximum	21.1%	10.8%	10.8%	12.2%	-8.2%	-15.8%
	Mean	-10.1%	-40.9%	-13.0%	-16.0%	-22.9%	-26.7%
	Median	-6.3%	-40.9%	-14.3%	-10.2%	-29.9%	-21.9%
RPD of Target Element ² (Absolute Value)	Minimum	0.5%	10.8%	1.5%	0.4%	8.2%	15.8%
	Maximum	73.3%	92.6%	28.3%	95.0%	30.5%	58.9%
	Mean	14.5%	51.7%	15.2%	18.3%	22.9%	26.7%
	Median	10.5%	51.7%	14.3%	10.7%	29.9%	21.9%
Interferent Concentration Range	Minimum	41	784	842	30	1022	1561
	Maximum	11862	20797	9233	3979	1650	5607
	Mean	1335	10790	3507	441	1299	3045
	Median	197	10790	3692	120	1225	2242
Target Element Concentration Range	Minimum	46	144	92	41	138	96
	Maximum	5607	3979	267	20797	226	363
	Mean	1432	2061	137	2164	173	187
	Median	1022	2061	131	570	154	185

Notes:

- Concentrations are reported in units of milligrams per kilogram (mg/kg), or parts per million (ppm).
Table presents data only for the instrument equipped with 35 kilovolt x-ray tube.
 - Table presents statistics for raw (unmodified) RPDs as well as absolute value RPDs
- < Less than.
> Greater than.
RPD Relative percent difference

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube)

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	2	2
			Minimum	--	--	--	--	-141.6%	141.6%	-15.2%	2.3%
			Maximum	--	--	--	--	-141.6%	141.6%	2.3%	15.2%
			Mean	--	--	--	--	-141.6%	141.6%	-6.4%	8.8%
			Median	--	--	--	--	-141.6%	141.6%	-6.4%	8.8%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	1	1	5	5	5	5
			Minimum	-62.4%	20.0%	90.6%	90.6%	-35.6%	3.1%	-10.4%	0.3%
			Maximum	-20.0%	62.4%	90.6%	90.6%	23.1%	35.6%	-0.3%	10.4%
			Mean	-46.7%	46.7%	90.6%	90.6%	-2.1%	14.4%	-6.1%	6.1%
			Median	-52.2%	52.2%	90.6%	90.6%	3.1%	5.5%	-6.9%	6.9%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	1	1	1	1	1	1
			Minimum	-18.6%	18.6%	79.9%	79.9%	3.9%	3.9%	12.2%	12.2%
			Maximum	-18.6%	18.6%	79.9%	79.9%	3.9%	3.9%	12.2%	12.2%
			Mean	-18.6%	18.6%	79.9%	79.9%	3.9%	3.9%	12.2%	12.2%
			Median	-18.6%	18.6%	79.9%	79.9%	3.9%	3.9%	12.2%	12.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	1	1	--	--	--	--	--	--
			Minimum	127.5%	127.5%	--	--	--	--	--	--
			Maximum	127.5%	127.5%	--	--	--	--	--	--
			Mean	127.5%	127.5%	--	--	--	--	--	--
			Median	127.5%	127.5%	--	--	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	4	4	11	11	5	5
			Minimum	-45.6%	17.6%	78.4%	78.4%	-97.9%	0.9%	-28.3%	3.7%
			Maximum	-17.6%	45.6%	102.7%	102.7%	49.8%	97.9%	3.7%	28.3%
			Mean	-34.0%	34.0%	86.8%	86.8%	3.5%	34.5%	-9.6%	11.1%
			Median	-36.4%	36.4%	83.1%	83.1%	20.2%	30.7%	-6.9%	6.9%
Sediment	RF	Silty fine sand (tailings)	Number	3	3	3	3	12	12	5	5
			Minimum	-24.7%	4.9%	84.5%	84.5%	-39.9%	7.9%	-17.6%	5.7%
			Maximum	15.1%	24.7%	105.2%	105.2%	28.8%	39.9%	7.1%	17.6%
			Mean	-4.8%	14.9%	94.2%	94.2%	13.8%	20.5%	-6.6%	9.4%
			Median	-4.9%	15.1%	92.9%	92.9%	18.1%	19.0%	-7.2%	7.2%

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (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	-50.1%	36.6%	-9.5%	5.1%	-42.7%	4.8%	-22.6%	3.3%
			Maximum	-36.6%	50.1%	10.8%	10.8%	-4.8%	42.7%	3.3%	22.6%
			Mean	-43.3%	43.3%	-1.3%	8.5%	-27.5%	27.5%	-10.0%	12.2%
			Median	-43.3%	43.3%	-5.1%	9.5%	-35.1%	35.1%	-10.7%	10.7%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	7	7	6	6	7	7	7	7
			Minimum	-55.6%	13.7%	-23.7%	7.8%	-44.0%	14.8%	-19.5%	1.1%
			Maximum	-13.7%	55.6%	7.8%	23.7%	-14.8%	44.0%	10.9%	19.5%
			Mean	-34.4%	34.4%	-13.5%	16.1%	-28.4%	28.4%	-6.0%	10.3%
			Median	-36.0%	36.0%	-17.0%	17.0%	-28.0%	28.0%	-7.7%	10.9%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	3	3	3	3	3	3
			Minimum	-59.9%	59.9%	-23.3%	14.2%	-50.5%	1.6%	-33.8%	23.1%
			Maximum	79.3%	79.3%	21.1%	23.3%	-1.6%	50.5%	119.8%	119.8%
			Mean	9.7%	69.6%	-5.5%	19.6%	-28.2%	28.2%	36.4%	58.9%
			Median	9.7%	69.6%	-14.2%	21.1%	-32.4%	32.4%	23.1%	33.8%
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	-52.8%	29.4%	-31.7%	11.6%	-55.3%	2.9%	-9.2%	8.5%
			Maximum	-29.4%	52.8%	11.6%	31.7%	-2.9%	55.3%	22.9%	22.9%
			Mean	-42.4%	42.4%	-10.1%	21.7%	-25.6%	25.6%	8.1%	14.0%
			Median	-43.7%	43.7%	-10.1%	21.7%	-22.7%	22.7%	13.2%	13.2%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	7	7	4	4	12	12	6	6
			Minimum	-51.9%	2.8%	-54.9%	3.8%	-80.7%	26.6%	-38.4%	23.5%
			Maximum	2.8%	51.9%	4.3%	54.9%	32.7%	80.7%	-23.5%	38.4%
			Mean	-26.6%	27.4%	-16.4%	20.5%	-42.2%	47.6%	-30.0%	30.0%
			Median	-30.4%	30.4%	-7.6%	11.6%	-46.6%	46.6%	-28.1%	28.1%
Sediment	RF	Silty fine sand (tailings)	Number	12	12	13	13	13	13	13	13
			Minimum	-47.3%	7.1%	-28.3%	1.8%	-39.6%	18.3%	-58.8%	0.3%
			Maximum	-7.1%	47.3%	3.1%	28.3%	-18.3%	39.6%	0.3%	58.8%
			Mean	-26.4%	26.4%	-11.4%	12.1%	-26.5%	26.5%	-21.8%	21.8%
			Median	-27.9%	27.9%	-10.5%	10.5%	-27.1%	27.1%	-13.1%	13.1%

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (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	--	--	3	3	1	1	1	1
			Minimum	--	--	-38.5%	21.7%	-10.6%	10.6%	-6.1%	6.1%
			Maximum	--	--	34.3%	38.5%	-10.6%	10.6%	-6.1%	6.1%
			Mean	--	--	5.8%	31.5%	-10.6%	10.6%	-6.1%	6.1%
			Median	--	--	21.7%	34.3%	-10.6%	10.6%	-6.1%	6.1%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	--	--	5	5	4	4	4	4
			Minimum	--	--	-41.0%	6.7%	-5.4%	2.2%	-71.0%	1.8%
			Maximum	--	--	-6.7%	41.0%	10.0%	10.0%	-1.8%	71.0%
			Mean	--	--	-27.5%	27.5%	0.5%	5.6%	-27.6%	27.6%
			Median	--	--	-30.9%	30.9%	-1.3%	5.1%	-18.8%	18.8%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	3	3	2	2	2	2
			Minimum	-35.2%	9.5%	-43.4%	21.9%	-13.5%	1.9%	-30.0%	26.4%
			Maximum	-9.5%	35.2%	-21.9%	43.4%	1.9%	13.5%	-26.4%	30.0%
			Mean	-22.3%	22.3%	-29.2%	29.2%	-5.8%	7.7%	-28.2%	28.2%
			Median	-22.3%	22.3%	-22.2%	22.2%	-5.8%	7.7%	-28.2%	28.2%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	3	3	--	--	--	--
			Minimum	--	--	-41.0%	23.9%	--	--	--	--
			Maximum	--	--	-23.9%	41.0%	--	--	--	--
			Mean	--	--	-33.1%	33.1%	--	--	--	--
			Median	--	--	-34.5%	34.5%	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	8	8	5	5	4	4
			Minimum	-77.9%	13.1%	-64.1%	1.2%	-31.9%	4.4%	-46.0%	13.0%
			Maximum	-13.1%	77.9%	-1.2%	64.1%	4.4%	31.9%	13.0%	46.0%
			Mean	-39.3%	39.3%	-33.5%	33.5%	-13.7%	15.5%	-21.9%	28.4%
			Median	-33.0%	33.0%	-32.7%	32.7%	-12.2%	12.2%	-27.4%	27.4%
Sediment	RF	Silty fine sand (tailings)	Number	5	5	13	13	5	5	5	5
			Minimum	-125.2%	36.1%	-52.3%	12.2%	-28.6%	13.5%	-96.1%	3.3%
			Maximum	-36.1%	125.2%	-12.2%	52.3%	-13.5%	28.6%	-3.3%	96.1%
			Mean	-67.6%	67.6%	-28.3%	28.3%	-20.4%	20.4%	-37.3%	37.3%
			Median	-48.0%	48.0%	-24.2%	24.2%	-22.1%	22.1%	-27.5%	27.5%

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (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%	-27.8%	5.9%
			Maximum	-68.7%	68.7%	-5.9%	27.8%
			Mean	-68.7%	68.7%	-19.1%	19.1%
			Median	-68.7%	68.7%	-23.6%	23.6%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	7	7
			Minimum	-42.2%	26.1%	-21.1%	0.6%
			Maximum	74.1%	74.1%	0.6%	21.1%
			Mean	5.3%	44.8%	-12.8%	12.9%
			Median	-5.3%	39.5%	-17.1%	17.1%
Soil	CN	Sandy loam (burn pit residue)	Number	1	1	3	3
			Minimum	33.4%	33.4%	-32.5%	0.4%
			Maximum	33.4%	33.4%	0.4%	32.5%
			Mean	33.4%	33.4%	-13.7%	14.0%
			Median	33.4%	33.4%	-9.1%	9.1%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	--	--	2	2
			Minimum	--	--	-29.9%	9.5%
			Maximum	--	--	9.5%	29.9%
			Mean	--	--	-10.2%	19.7%
			Median	--	--	-10.2%	19.7%
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	9	9	10	10
			Minimum	-100.9%	16.8%	-74.0%	2.2%
			Maximum	64.7%	100.9%	-2.2%	74.0%
			Mean	-17.2%	57.4%	-31.2%	31.2%
			Median	-16.8%	62.0%	-25.8%	25.8%
Sediment	RF	Silty fine sand (tailings)	Number	3	3	13	13
			Minimum	15.4%	15.4%	-37.3%	1.2%
			Maximum	81.7%	81.7%	8.5%	37.3%
			Mean	45.8%	45.8%	-11.3%	14.3%
			Median	40.4%	40.4%	-9.8%	9.8%

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (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	5	5	1	1
			Minimum	-32.8%	1.0%	93.9%	93.9%	10.3%	10.3%	-12.5%	12.5%
			Maximum	1.0%	32.8%	93.9%	93.9%	22.2%	22.2%	-12.5%	12.5%
			Mean	-13.4%	13.8%	93.9%	93.9%	17.9%	17.9%	-12.5%	12.5%
			Median	-11.8%	11.8%	93.9%	93.9%	20.7%	20.7%	-12.5%	12.5%
Sediment			Number	3	3	3	3	1	1	2	2

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (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	6	6
			Minimum	-56.3%	30.6%	-13.7%	1.5%	-44.7%	7.5%	-32.0%	5.5%
			Maximum	-30.6%	56.3%	7.5%	13.7%	-7.5%	44.7%	19.5%	32.0%
			Mean	-44.2%	44.2%	-3.8%	8.2%	-16.8%	16.8%	-7.3%	18.1%
			Median	-43.6%	43.6%	-4.4%	8.8%	-14.1%	14.1%	-7.3%	19.8%
Sediment	TL	Silt and clay (slag- enriched)	Number	1	1	7	7	7	7	4	4
			Minimum	-56.2%	56.2%	-5.1%	0.5%	-84.5%	32.3%	-23.2%	0.8%
			Maximum	-56.2%	56.2%	15.3%	15.3%	-32.3%	84.5%	-0.8%	23.2%
			Mean	-56.2%	56.2%	0.2%	4.3%	-58.5%	58.5%	-12.3%	12.3%
			Median	-56.2%	56.2%	-2.5%	3.1%	-48.6%	48.6%	-12.7%	12.7%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	7	7	6	6	7	7	7	7
			Minimum	-107.0%	1.1%	-92.6%	10.8%	-108.6%	10.2%	-88.4%	1.9%
			Maximum	1.1%	107.0%	10.8%	92.6%	-10.2%	108.6%	3.0%	88.4%
			Mean	-50.3%	50.6%	-38.1%	41.7%	-51.4%	51.4%	-30.6%	32.0%
			Median	-50.6%	50.6%	-29.5%	29.5%	-39.5%	39.5%	-19.1%	19.1%
	All		Number	53	53	48	48	70	70	55	55
			Minimum	-107.0%	1.1%	-92.6%	0.5%	-108.6%	1.6%	-88.4%	0.3%
			Maximum	79.3%	107.0%	21.1%	92.6%	32.7%	108.6%	119.8%	119.8%
			Mean	-35.4%	38.5%	-12.0%	16.2%	-33.5%	34.4%	-12.5%	22.1%
			Median	-36.6%	36.9%	-10.2%	11.2%	-30.1%	30.7%	-13.1%	16.0%

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (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	1	1
			Minimum	-61.9%	1.3%	-34.2%	1.1%	-5.7%	3.0%	-66.4%	66.4%
			Maximum	62.8%	62.8%	1.1%	34.2%	-3.0%	5.7%	-66.4%	66.4%
			Mean	10.8%	36.8%	-15.9%	16.1%	-4.6%	4.6%	-66.4%	66.4%
			Median	17.2%	40.6%	-14.6%	14.6%	-5.1%	5.1%	-66.4%	66.4%
Sediment	TL	Silt and clay (slag-enriched)	Number	3	3	3	3	4	4	4	4
			Minimum	7.2%	7.2%	-39.5%	16.6%	-34.0%	2.0%	-52.6%	3.5%
			Maximum	72.8%	72.8%	26.7%	39.5%	2.0%	34.0%	-3.5%	52.6%
			Mean	44.2%	44.2%	1.3%	27.6%	-16.1%	17.1%	-34.4%	34.4%
			Median	52.5%	52.5%	16.6%	26.7%	-16.3%	16.3%	-40.7%	40.7%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	--	--	3	3	1	1	1	1
			Minimum	--	--	-60.1%	46.3%	-13.4%	13.4%	13.3%	13.3%
			Maximum	--	--	-46.3%	60.1%	-13.4%	13.4%	13.3%	13.3%
			Mean	--	--	-51.1%	51.1%	-13.4%	13.4%	13.3%	13.3%
			Median	--	--	-46.8%	46.8%	-13.4%	13.4%	13.3%	13.3%
	All		Number	25	25	52	52	25	25	22	22
			Minimum	-125.2%	1.3%	-64.1%	1.1%	-34.0%	1.9%	-96.1%	1.8%
			Maximum	72.8%	125.2%	34.3%	64.1%	10.0%	34.0%	13.3%	96.1%
			Mean	-11.5%	43.1%	-24.4%	28.2%	-11.3%	12.9%	-29.0%	31.4%
			Median	-15.8%	40.6%	-24.8%	26.5%	-10.6%	10.6%	-27.8%	27.8%

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (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	-85.9%	0.3%	-49.2%	1.6%
			Maximum	56.6%	85.9%	6.4%	49.2%
			Mean	-51.1%	62.4%	-9.2%	11.4%
			Median	-66.4%	66.4%	-7.4%	7.4%
Sediment	TL	Silt and clay (slag- enriched)	Number	7	7	7	7
			Minimum	-63.9%	28.9%	-22.8%	8.2%
			Maximum	-28.9%	63.9%	-8.2%	22.8%
			Mean	-44.7%	44.7%	-17.6%	17.6%
			Median	-47.0%	47.0%	-18.9%	18.9%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	3	7	7
			Minimum	-38.2%	37.8%	-95.0%	1.2%
			Maximum	54.6%	54.6%	12.2%	95.0%
			Mean	-7.2%	43.5%	-35.3%	39.1%
			Median	-37.8%	38.2%	-32.4%	32.4%
	All		Number	38	38	63	63
			Minimum	-100.9%	0.3%	-95.0%	0.4%
			Maximum	81.7%	100.9%	12.2%	95.0%
			Mean	-23.1%	52.7%	-18.1%	19.8%
			Median	-38.8%	54.2%	-15.8%	15.8%

Table E-8. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (35 kV X-Ray Tube) (Continued)

Site Abbreviations:

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

Other Notes:

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

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (40 kV X-ray Tube)

Matrix	Site	Matrix Description	Statistic	Antimony				Cadmium		Silver	
				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	--	--	--	--	3	3	1	1
			Minimum	--	--	--	--	-27.8%	20.6%	5.2%	5.2%
			Maximum	--	--	--	--	-20.6%	27.8%	5.2%	5.2%
			Mean	--	--	--	--	-23.3%	23.3%	5.2%	5.2%
			Median	--	--	--	--	-21.5%	21.5%	5.2%	5.2%
Soil	BN	Sandy loam, low organic (ore residuals)	Number	4	4	1	1	5	5	4	4
			Minimum	-60.7%	12.8%	96.3%	96.3%	-22.1%	8.9%	-55.8%	4.6%
			Maximum	-12.8%	60.7%	96.3%	96.3%	-8.9%	22.1%	-4.6%	55.8%
			Mean	-41.6%	41.6%	96.3%	96.3%	-14.3%	14.3%	-34.2%	34.2%
			Median	-46.4%	46.4%	96.3%	96.3%	-15.4%	15.4%	-38.2%	38.2%
Soil	CN	Sandy loam (burn pit residue)	Number	2	2	1	1	2	2	2	2
			Minimum	-50.7%	14.1%	83.8%	83.8%	-27.7%	0.4%	-38.0%	17.9%
			Maximum	-14.1%	50.7%	83.8%	83.8%	-0.4%	27.7%	-17.9%	38.0%
			Mean	-32.4%	32.4%	83.8%	83.8%	-14.0%	14.0%	-27.9%	27.9%
			Median	-32.4%	32.4%	83.8%	83.8%	-14.0%	14.0%	-27.9%	27.9%
Soil & Sediment	KP	Soil: Fine to medium quartz sand. Sed.: Sandy loam, high organic. (Gun and skeet ranges)	Number	1	1	--	--	--	--	--	--
			Minimum	116.6%	116.6%	--	--	--	--	--	--
			Maximum	116.6%	116.6%	--	--	--	--	--	--
			Mean	116.6%	116.6%	--	--	--	--	--	--
			Median	116.6%	116.6%	--	--	--	--	--	--
Sediment	LV	Clay/clay loam, salt crust (iron and other precipitate)	Number	4	4	4	4	5	5	4	4
			Minimum	-55.7%	12.9%	85.7%	85.7%	-34.9%	5.8%	-31.1%	3.3%
			Maximum	-12.9%	55.7%	94.4%	94.4%	-5.8%	34.9%	17.5%	31.1%
			Mean	-32.0%	32.0%	88.6%	88.6%	-21.9%	21.9%	-9.0%	17.8%
			Median	-29.6%	29.6%	87.1%	87.1%	-20.4%	20.4%	-11.2%	18.3%
Sediment	RF	Silty fine sand (tailings)	Number	4	4	4	4	5	5	3	3
			Minimum	-81.0%	0.6%	64.6%	64.6%	-29.2%	14.1%	-78.7%	6.9%
			Maximum	13.8%	81.0%	104.3%	104.3%	-14.1%	29.2%	15.6%	78.7%
			Mean	-20.7%	27.6%	89.4%	89.4%	-20.3%	20.3%	-23.3%	33.7%
			Median	-7.8%	14.4%	94.3%	94.3%	-16.3%	16.3%	-6.9%	15.6%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (40 kV X-ray Tube) (Continued)

Matrix	Site	Matrix Description	Statistic	Antimony				Cadmium		Silver	
				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	6	6	1	1	1	1	1	1
			Minimum	-30.2%	11.1%	97.2%	97.2%	-19.1%	19.1%	-51.6%	51.6%
			Maximum	-11.1%	30.2%	97.2%	97.2%	-19.1%	19.1%	-51.6%	51.6%
			Mean	-20.3%	20.3%	97.2%	97.2%	-19.1%	19.1%	-51.6%	51.6%
			Median	-18.4%	18.4%	97.2%	97.2%	-19.1%	19.1%	-51.6%	51.6%
Sediment	TL	Silt and clay (slag- enriched)	Number	3	3	3	3	2	2	4	4
			Minimum	-91.8%	67.8%	101.5%	101.5%	-31.2%	22.3%	-50.7%	3.9%
			Maximum	-67.8%	91.8%	106.2%	106.2%	-22.3%	31.2%	-3.9%	50.7%
			Mean	-79.4%	79.4%	103.3%	103.3%	-26.8%	26.8%	-22.3%	22.3%
			Median	-78.6%	78.6%	102.2%	102.2%	-26.8%	26.8%	-17.4%	17.4%
Soil	WS	Coarse sand and gravel (roaster slag)	Number	3	3	--	--	3	3	3	3
			Minimum	-16.7%	2.9%	--	--	-89.3%	41.6%	27.3%	27.3%
			Maximum	2.9%	16.7%	--	--	-41.6%	89.3%	46.7%	46.7%
			Mean	-6.6%	8.5%	--	--	-71.5%	71.5%	34.5%	34.5%
			Median	-5.8%	5.8%	--	--	-83.7%	83.7%	29.5%	29.5%
	All		Number	27	27	14	14	26	26	22	22
			Minimum	-91.8%	0.6%	64.6%	64.6%	-89.3%	0.4%	-78.7%	3.3%
			Maximum	116.6%	116.6%	106.2%	106.2%	-0.4%	89.3%	46.7%	78.7%
			Mean	-26.1%	36.0%	92.8%	92.8%	-25.7%	25.7%	-15.0%	27.9%
			Median	-18.6%	19.6%	95.3%	95.3%	-21.1%	21.1%	-12.4%	28.0%

Table E-9. Evaluation of the Effects of Soil Type on RPDs (Accuracy) of Target Elements for the Innov-X XT400 (40 kV X-ray Tube) (Continued)

Site Abbreviations:

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

Other Notes:

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

Notes Regarding the Performance of the XT400 with the 40kV X-ray Tube versus the 35kV X-ray Tube

Samples were also analyzed on a second XT400 with an experimental 40 kV x-ray tube in the same manner and technique as for the original instrument with the 35kV x-ray tube. Only the analysis and results for three elements were of interest for the second instrument.

A second set of summary statistics was calculated for antimony, cadmium, and silver using the data set from the experimental XT400 instrument with the 40-kV x-ray tube. A comparison of the results obtained using the 40-kV x-ray tube and those obtained using the 35-kV x-ray tube revealed the following:

- When compared with the mean MDL for the 35-kV instrument, the mean MDL obtained for antimony using the 40-kV XRF analyzer decreased by more than one-half, from 45 to 21 ppm. The MDLs for cadmium and silver were equivalent in the data sets for the 35-kV and 40-kV analyzers at approximately 40 ppm.
- When compared with results obtained from the 35-kV instrument, the 40-kV analyzer reduced the overall median RPD for antimony from 27.0 percent to 19.6 percent and the overall median RPD for silver from 27.8 percent to 21.1 percent, improving the accuracy from the “fair” range into the “good” range for these elements. However, the 40-kV instrument increased the overall median RPD for cadmium from 9.7 percent to 21.1 percent. In summary, it cannot be concluded that the 40-kV instrument provided any overall improvement in accuracy in comparison to the 35-kV instrument because there was no consistent trend and differences in the median RPDs were relatively small.
- There were no significant differences in precision for antimony, cadmium, and silver using the 40-kV versus the 35-kV s-ray tube.