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Experimental Evaluation of Selected Field Portable Instrumentation for the Quantitative Determination of Contaminant Levels in Soil and Water at Rocky Mountain Arsenal

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FINAL REPORT

October 1989

R. A. Jenkins, F. F. Dyer,
R. L. Moody, C. K. Bayne, and C. V. Thompson

Army No. MIPR0389

Supported by
U.S. Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground, Maryland 21010-5401

Project Officer: Douglas Scarborough
U.S. Army Toxic and Hazardous Materials Agency
(USATHAMA)
CETHA-TS-C, Building E-4460
Beal Road
Aberdeen Proving Ground, Maryland 21010-5401

The findings in this report are not to be construed as an official Department of the Army position unless otherwise so designated by other authorized documents.

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MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
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MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
Under Contract No. DE-AC05-84OR21400

EXECUTIVE SUMMARY

The

The purpose of the work described in this report was to evaluate two instrumental systems for use under field conditions at the Army's Rocky Mountain Arsenal. The two systems were a portable gas chromatograph (GC), for the determination of volatile organic constituents in water and soil, and a portable x-ray fluorescence (XRF) unit, for the determination of selected elemental contamination in water and soil. The particular instruments chosen for evaluation were selected following an assessment of the most appropriate commercial instrumentation available at the time of the project. The two instruments were to be evaluated first under laboratory conditions, and then taken to the field. Under the latter set of conditions, USATHAMA Class 1 Certification runs and EPA Equivalency Testing would be used as tools for evaluating the utility of the instrumental systems chosen for field work. evaluate

Laboratory evaluation studies indicated that the particular portable GC chosen for use was insufficiently reliable to be used in a field setting, and so the remainder of the effort was placed on the evaluation of the portable XRF system. For the XRF system, the nature of the matrix being examined can affect the apparent quantity of the target element present, and it is critical to use calibration standards that are prepared from a material that simulates as closely as possible the chemical and physical properties of the environmental samples being analyzed. For the purposes of this study, clean RMA soil, and groundwater obtained from a well at Oak Ridge National Laboratory, were used as the standard matrices. Standards were prepared by spiking a known quantity of soil or water with a solution of the elements in question. For internal instrumental quantitation, soil and water samples are spiked at randomly chosen concentrations with solutions of the target elements, and a multivariate regression calibration model is developed. The term "internal" is used here to refer to the multivariate regression calibration that is developed and used by software that is internal (ROM-based) to the XRF system. A procedure such as this is required because the presence of one element may affect the apparent quantity of a second element. A sequential series of soil and water samples spiked according to the USATHAMA quality assurance guidelines were used for external calibration.

Ruggedness testing was performed to determine the effects of temperature, atmospheric pressure, and soil moisture and iron content. Temperature was shown to have essentially no effect, as long as the unit was operated above its designed lower temperature limit of 0°C. The ruggedness test for pressure determined a 2.2% difference in response to a copper single element standard run at an elevation of 5200 feet, and the same sample run at an elevation of less than 1000 feet. This difference was not significant from a practical standpoint. The ruggedness test for iron content of the soil, which was expected to affect primarily the copper intensity, yielded data showing a 12% difference in the mean copper intensity for 0% iron versus 2.2% iron. This is a statistically significant difference; however, soil iron content in soil samples collected at RMA varied only between 1 and 2%. Soil moisture content was shown to have a significant effect. However, once a soil

sample was moistened, the degree of response variation as a function of moisture content, up to the point of saturation was determined to be about ± 12%. Instrumental detection limits, taken to be 3 times the square root of the background count rate, were determined in a laboratory setting. For wet soil, these were 25, 12, 29, and 60 ppm, for Cu, As, Hg, and Pb, respectively, and 9, 4, 6, and 42 ppm for water.

Class 1 Certification of the analytical methods was performed in both the laboratory and under field conditions according to USATHAMA guidelines. Field analyses of actual environmental samples were conducted with four large surface soil and one sump water collected at RMA. Separate duplicate aliquots of each sample were removed from the larger samples daily for each of a minimum of ten days. This approach was taken to determine the variability of the analytical results with time. For soil, Certified Reporting Limits (CRL's) were determined to be 112, 187, and 192 ppm, for arsenic, mercury, and lead in RMA soil, respectively. We could not obtain a sufficiently high quality calibration curve for copper in soil in the 100 ppm range, probably due to the overlapping nature of the copper and iron photopeaks, and the high iron content of the soil. CRL's for copper, mercury, and lead in water were 38, 39, and 176 ppm, respectively.

For environmental samples collected at RMA with contaminant levels above the CRL's, the agreement between levels of target element contamination calculated using a multivariate regression calibration routine and those using a single variate routine was good. The exception to this was arsenic in water, which failed certification. The multivariate (manufacturer's) and the single variate (USATHAMA) regression calibrations both have their advantages and disadvantages. The multivariate calibration takes into account a wider range of potential concentrations of the target elements varying independently, but is somewhat more complicated to perform due to the fact that it is necessary to prepare and use at least 18 standards when four analytes are to be measured.

Equivalency testing, in which results using the XRF system were compared with those obtained using laboratory methods, was conducted both on surrogate samples in a laboratory setting and real samples in the field. For all cases in which contaminant levels were greater than the CRL, the XRF system was shown not to be equivalent to the laboratory based procedure. However, in nearly all of the element/sample comparisons, the XRF system was able to accurately determine whether the contamination level was above or below the CRL, and the approximate level of contamination if it was above the CRL. Thus, experience at RMA indicated that the XRF system can be used under field portable conditions and achieve reasonably quantitative results for wet soil and water contaminated in the 100-3000 ppm range. However, it was not equivalent to conventional laboratory based methods in terms of accuracy and precision. Field equivalency tests;

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I. INTRODUCTION AND PURPOSE

Interest in field analysis of contaminants, either for screening or quantitative purposes, has increased dramatically in recent years. There have been at least two driving forces behind this increase. First, there is a need to more easily identify those areas where contamination exists and avoid sampling and analyzing samples from areas where contamination is below some action level. This is in order to avoid the cost associated with the detailed laboratory analysis. Secondly, field analysis can provide much more rapid turnaround times, which are critical when restoration operations are under way. The purpose of the work described in this report is the evaluation of two instrumental systems for use under field conditions at the Army's Rocky Mountain Arsenal. The two systems were a portable gas chromatograph (GC), for the determination of volatile organic constituents in water and soil, and a portable x-ray fluorescence (XRF) unit, for the determination of selected elemental contamination in water and soil. The particular instruments selected for evaluation were chosen following an assessment of the most appropriate commercial instrumentation available at the time of the project

II. EXPERIMENTAL DESIGN

A detailed experimental plan was developed for the laboratory and field phases of this study in close cooperation with USATHAMA project management. The purpose of the plan was to provide a clear agreement between the sponsor and ORNL concerning the details of the experimental aspects of the undertaking. The work was divided into two phases. First, both the laboratory reference methods and the candidate field methods were established. This effort had three tasks. In the first task, reference analytical methods were to be established in a laboratory setting and subsequently certified at a Class 2 level, according to USATHAMA guidelines. In the second task, candidate field methods were to be established in a laboratory setting and evaluated as to their potential efficacy for field analysis. The final task of this phase was the certification at a Class 1 level and the determination of the degree of equivalency between the reference and the candidate field method in a laboratory setting. In this case, EPA guidelines for method equivalency were used.

The field phase of the project was to be comprised of several tasks. In the first task, a Class 1 certification of the field methods was to be conducted under field conditions out-of-doors at RMA. Next, field analysis would be performed on a suite of samples, and splits would be returned to the laboratory for analysis using the reference methods, as an initial Equivalency Test. Experience gained during the field analysis would be used also in the development and conduct of a ruggedness test for the field method, to further refine the analytical protocol and prepare for the final field Equivalency Test. Finally, we would return to RMA to perform the Equivalency Testing.

In practice, there were a number of changes in the conduct of the experimental plan, both in the laboratory and field phases, resulting from both experimental findings and budget requirements. These findings and changes are described below.

III. EVALUATION OF FIELD DETERMINATION OF ORGANICS USING A PORTABLE GAS CHROMATOGRAPH

III.A. Establishment of Laboratory Methods

An important aspect of the laboratory phase of the study was to establish USATHAMA standard analytical methods for the target organic compounds and certify their efficacy at the USATHAMA Class 2 level. For the target organic species, benzene, trichloroethylene, tetrachloroethylene, and dicyclopentadiene, three analytical methods were to be used. DCPD is determined by extraction of the soil or water sample with methylene chloride, and subjecting an aliquot to gas chromatographic analysis on a fused silica DB-5 coated capillary column with flame ionization detection (Methods Z-8 and ZZ-9). Benzene in soil and water is quantitated using purge and trap methodology, followed by packed column (1% SP-1000 on Carbopack B) GC analysis using photoionization detection (PID) (Method W-8). TRCLE and TCLEE are determined using essentially the same procedure as for the benzene, except that a Hall electrolytic conductivity detector is used (Methods Y-8 and YY-9).

Class 2 Certified Reporting Limits (CRL's) were obtained for the laboratory methods for benzene and DCPD in water and soil. For benzene, the levels were 0.9 μg/L and 0.9 μg/kg, respectively. For DCPD, the levels were 55 μg/L and 5 μg/g, respectively. The determination of the chlorinated hydrocarbons calls for the use of a Hall electrolytic conductivity detector. Repeated attempts were made to get either one of two such systems available to us to function reproducibly. About the time that a decision was made to switch the analytical determination to an electron capture detector, an overall project decision was made not to proceed with the analysis of the organics. Thus, CRL's were not obtained for the chlorinated hydrocarbons.

III.B. Laboratory Evaluation of the Portable GC

III.B.1. Instrument Description and Operation. The Scentograph portable GC (manufactured by Sentex Sensing Technology, Inc.) is a self-contained briefcase shaped instrument, which is placed on its side (bottom) when in use. The system, shown in Figure 1, is designed for continuous or intermittent monitoring of airborne volatile organics. The GC is comprised of five major components. The first consists of two gas cylinders and related plumbing, which are located in the back of the instrument. These cylinders contain carrier gas and calibration gas. The plumbing provides for filling of the cylinders, with connections in the back of the instrument, and for supplying the gases to the analytical module. The second component consists of four lead-acid, 6-volt, 6 amp hour batteries connected so as to provide 12-volt, 12 amp hour power for the instrument.

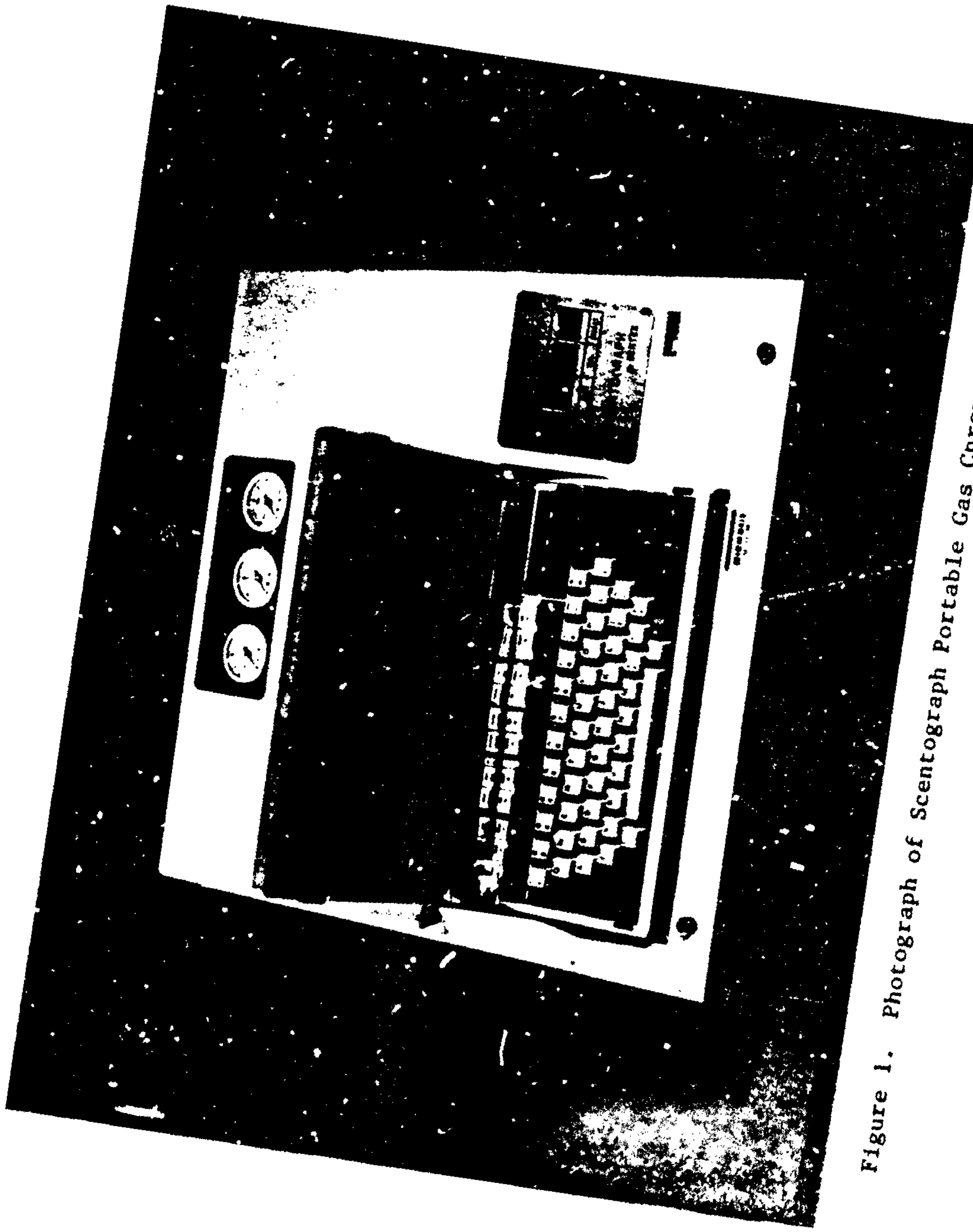

Figure 1. Photograph of Scentograph Portable Gas Chromatograph

The batteries are in the "bottom" of the instrument. The electronic components of the instrument are located primarily on a single board positioned over the batteries. The fourth component is the analytical module, which contains sample valves and plumbing, a Tenax tube on which the sample volatiles are preconcentrated, a column and column oven, a detector and detector oven, and associated electronic components. The analytical module is self-contained and is completely replaced in order to use a different detector. The fifth component consists of a Toshiba 1100 Plus personal computer, which controls operation of the instrument. The computer is located on top of the instrument. In addition, a built-in pump draws air through the Tenax cartridge.

After an analytical cycle is initiated, the air sampling pump is activated, and valving is set to pull either an air sample or calibration gas through the preconcentrator tube for a predetermined period of time ranging from 1 to 300 seconds. The Tenax preconcentrator tube is then flushed with carrier gas for a predetermined period of time ranging from 0.1 to 4 seconds. The chromatographic separation is initiated by the desorption of the preconcentrated samples from the Tenax tube by heating the filament wire, which is wrapped around the Tenax tube, for a predetermined period of time ranging from 0.1 to 4 seconds. The valving is then set to allow carrier gas to flush the sample from the Tenax tube, in the opposite direction from sample collection, into the packed GC column. Chromatography occurs isothermally at a predetermined temperature ranging from 30 to 140 degrees Centigrade, and detection occurs immediately after sample elution from the packed column.

The efficacy of two detector types for this work was investigated. One was an argon ionization/electron capture detector. This type of detector system, used in the electron capture mode, would be very sensitive to chlorinated hydrocarbons, but would not be sensitive to all the compounds of interest in this study. Using the detector in the argon ionization mode would make it a more universal detector. However, it appeared to be insufficiently sensitive to be able to quantitatively determine the target constituents as well as the photoionization detector (see below).

The Scentograph portable gas chromatograph was designed to be operated by an attached Toshiba 1100 Plus personal computer (laptop). Operating parameters could be established, sample or standard analysis initiated, chromatograms saved to diskette, recalled from diskette for display, or overlayed for comparison, and sample components' concentrations calculated.

III.B.2. Interface with Portable Purge Unit. The Scentograph was designed to be an air sampling system. Because it contains an air sampling pump and a preconcentrator tube, it was thought to be readily adaptable to purge and trap sampling of water and soil samples. The purge system which was fabricated consists of a canister filled with activated charcoal, which would trap volatiles from the ambient air and prevent contamination of the sample, and a 40 mL sample bottle used as the purge vessel. The charcoal canister is connected to the purge vessel via a tube that runs through the septum cap and stops at a depth of 1 cm from the bottom of the vessel. This is below the level of sample in the vessel. The purge vessel is connected to the air sampling pump of the instrument via a tube which extends just through the septum cap into the purge vessel well above the sample level. When sampling is initiated, the air sampling pump pulls a partial vacuum inside the purge vessel, the force of which pulls ambient air through the charcoal canister and through the tube into the purge vessel, bubbling up through the sample. The air which is thus bubbled through the sample carries the volatile components of the sample with it through the Tenax tube, where it is trapped. After the sample purge is complete, the air sampling pump is shut off, and the contents trapped on the Tenax tube are desorbed into the packed column. Standard operation of the Scentograph does not allow for evaporating any moisture from the Tenax trap prior to desorption of the sample. However, this could be accomplished by removing the sample vial from the purge stream a few seconds prior to the end of the purge cycle.

While three of the four target organic species are relatively volatile, DCPD is typically considered a semivolatile. Thus, standard analytical methods rely on isolating the DCPD from an environmental matrix by extraction with organic solvent. However, if DCPD could be isolated using purge and trap techniques along with the other target species, it would reduce the number of analytical procedures required in the field. Thus, purging efficiency studies for DCPD in water samples were conducted. Mean purge efficiency, as determined by comparison with standards of DCPD directly injected into the GC, was 67.9% ± 10.7% for 8 determinations. Since this is higher than the 60% specified in the experimental plan, the data indicated that all of the target organics could be isolated with a single purge and trap process in the field.

III.B.3. Instrumental Difficulties. Both the Scentograph, and it's noncomputer operated counterpart, the Scentor, were evaluated for this study. (The Scentor was available because it was no longer being used for the study for which it was originally purchased). At one point, it was believed that two GC's would be required in the field, and thus using instruments which were essentially identical seemed most appropriate. With both of these instruments, a substantial number of mechanical and electrical problems were encountered. These included:

1. Loss of AID/ECD detector response of the Scentor, as well as a blown power transistor in the circuitry controlling the desorption heating cycle. Factory repair was required.

2. Air sampling pump of the Scentor failed, and a detector signal diminished. This required factory repair.

3. Failure of Scentor column oven to maintain 130°C set point, even with constant battery charging. To overcome this problem, we constructed a large battery pack to provide full 8-hour operation at maximum temperature set point.

4. Scentor was returned from factory with electrometer problem. Returned to factory for additional repair.

5. Scentograph preconcentrator tube cracked on first use. Repaired in-house.

6. Loss of communication between computer and Scentograph electronics module. Electronics board replaced, problem eventually traced to faulty in-line fuse holder. Repaired in-house.

7. Reversed power polarity during trouble shooting of Scentograph required factory replacement of PID system.

8. Blown battery charge rectifier replaced in-house.

9. Scentograph/PID developed very noisy signal. Required factory cleaning of PID cell window.

In general, both GC's had a number of instrument breakdowns, plus expected coating of photoionization detector windows, all which required very time consuming inhouse or factory repairs. The lack of reliability, combined with difficulty of field repair, caused us considerable delay and concern for the efficiency of these units in the fields. It was primarily because of these continued difficulties and the resources required to overcome them that a joint decision between ORNL and USATHAMA was made to terminate the organics part of the project.

III.B.4. Interference Studies. A detailed review of the Ebasco and ESE site survey data suggested that a number of organic compounds would likely be found in substantial concentrations with the target organics in many of the potential sampling sites at RMA. These compounds were: chlorobenzene, chloroform, ethylbenzene, toluene, aldrin, dieldrin, and endrin. Thus, it was necessary to be able to separate these compounds chromatographically from the target compounds in order to be able to quantitate the latter. To accomplish this, a number of chromatographic columns were evaluated. These are listed below.

1. 0.2% Carbowax on 60/80 Carbopack C.
2. 3% to 20% SP1000 on 100/120 Supelcoport.
3. 3% SP2250 on 100/120 Supelcoport.
4. 10% SP2340 on 100/120 Chromosorb WAW.
5. 3% Carbowax on 100/120 Supelcoport.
6. 10% Carbowax 20M/0.1% KOH on Supelcoport.
7. 20% SP2100/0.1% Carbowax 1500 on 100/120 Supelcoport.

We discovered that we are unable to elute all of the target compounds from any carbon-based packing, thus eliminating column 1 from further consideration. We were unable to separate toluene, an interferent, from tetrachloroethylene (TCLEE), a target compound, with columns 2-6. Finally, we were able to achieve adequate toluene/TCLEE separation, and adequate separation of all the other detectable (PID) interferents and target compounds with column 7. A sample chromatogram of these separations is portrayed in Figure 2.

Under these conditions, initial instrumental limits of detection (ILOD) were determined. The ILOD's were taken as five times the level of background noise. For benzene, trichloroethylene, tetrachloroethylene, and dicyclopentadiene, the ILOD's were 4.7, 4.5, 5.7, and 97 ng/mL, respectively, using the photoionization detector.

III.B.5. <u>Summary Evaluation</u>. The Scentograph portable GC has several advantages. It is computer controlled, facilitating data acquisition and retrieval. It has the ability to preconcentrate samples, which enhances sensitivity to airborne species. The unit is easily portable. However, for field purge and trap sampling of volatile organics in soil and water, it appeared to have some serious shortcomings. The built-in battery pack can only power the GC for approximately 3 hours at 130°C oven temperature, necessitating the use of an external battery pack for extended field use at higher temperatures. If cleaning

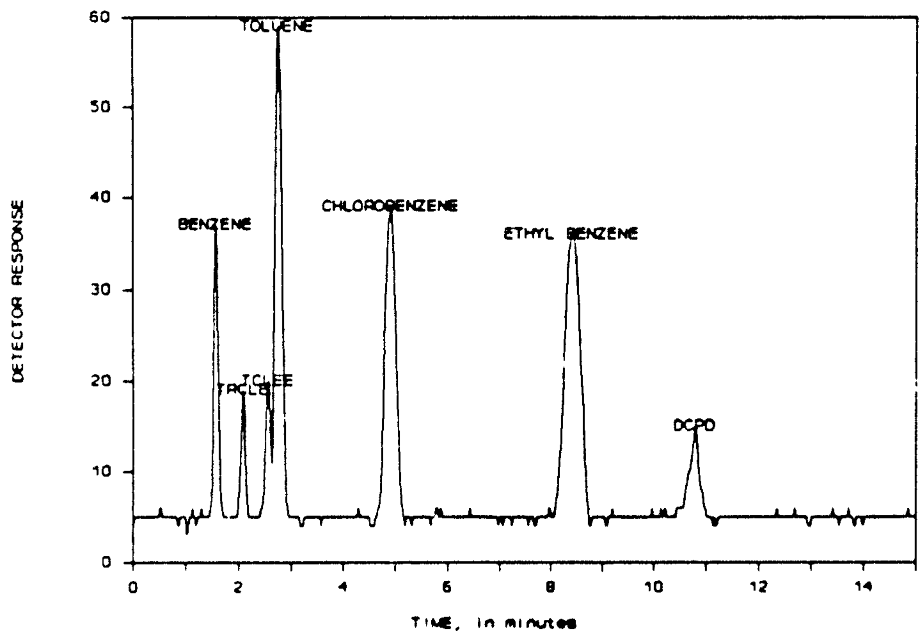

Figure 2. Detector Trace from Scentograph Portable GC. [TRCLE: trichloroethylene; TCLEE: tetrachloroethylene; DCPD: dicyclopentadiene.]

or repairs are necessary in the field, many of the common repairs to be expected would be impossible to accomplish without a return of the instrument to the factory. In particular, cleaning of a dirty PID window or repair of a broken Tenax preconcentrator tube is virtually impossible in the field. The Sentex portable GC's do not appear to have been engineered with field repair in mind. Even though the instruments are fairly easy to disassemble, many of the electronic components are not commonly stocked items, and many items can only be replaced as larger modules. While we received exceptionally good service from the manufacturer, our experience in the laboratory was that too-frequent cleaning of the PID was required, and that the number of electronic and mechanical breakdowns which we experienced attempting to use the system for purge and trap analysis of volatile organics precluded its use in a field setting for this particular application.

The use of a PID as a primary detector system was chosen based on its historic high sensitivity and trouble free operation. However, such did not prove to be the case with this system. A new generation AID/ECD, which would be an even more universal detector when used in the argon ionization mode, has been developed by the manufacturer. It is reported to have sensitivity comparable to that of the PID. However, this new unit was not available at the time of purchase of the instrument. We speculate that with an improvement in reliability and if the new generation AID/ECD detector proved sufficiently sensitive and stable, the Scentograph could be used in the field for purge and trap analysis of volatiles in water and soil samples, using a 6 foot 20% SP2100/0.1% Carbowax 1500 on 100/120 Supelcoport column, with satisfactory results.

IV. EVALUATION OF FIELD DETERMINATION OF INORGANIC SPECIES USING PORTABLE X-RAY FLUORESCENCE

IV.A. Experimental

IV.A.1. Establishment of Laboratory Methods. As with the determination of the target organic species, laboratory based methods for the determination of arsenic, copper, mercury, and lead were established and certified at the USATHAMA Class 2 level. Three analytical methods were used. Arsenic in soil and water were determined using graphite furnace atomic absorption spectroscopy (USATHAMA Methods A8 and T9). Copper and lead were determined by inductively coupled plasma spectroscopy (USATHAMA Methods D9A and B8). Mercury was determined by cold vapor atomic absorption (USATHAMA Methods V9 and L8). All of these methods have very high sensitivities. However, because of the much higher absolute limits of detection of the XRF system, it was deemed unnecessary to certify the laboratory methods several orders of magnitude below the expected sensitivity of the XRF system. For this reason, all of the analytical methods for water were certified at 350 ng/mL (0.35 ppm), and the soil methods were certified at 35 μg/g.

IV.A.2. Operation and Calibration of Portable XRF Unit. The X-Met 840 is a portable x-ray fluorescence system manufactured by Outokompu in Finland and sold in the United States by Columbia Scientific Instruments. The instrument consists of a microprocessor-based multichannel analyzer with 256 channels and a probe that holds a gas proportional radiation detector and a radioisotope source that emits either x-rays or low-energy gamma rays to excite characteristic x-rays in samples. The front panel of the analyzer has a two-line liquid crystal display, a membrane covered alpha-numeric keyboard, a connection for the probe, and an RS232 interface for communication with a computer. Results of analyses, commands, and information generated by commands are printed on the LCD screen. The analyzer is controlled either by special keys, e.g., a start key that starts acquisition of an x-ray spectrum, or three-character commands, e.g., STD which causes the analyzer to print on the screen the standard deviations of assays when elemental analyses are made. The RS232 computer interface allows a computer, by means of communication software, to receive and send information to the system microprocessor. Nearly all information sent to the LCD screen is transmitted to the RS232 interface. All of the commands that can be issued from the analyzer's keyboard can also be sent from a computer. This feature permits a considerable amount of automation of the analyzer by computer programs. In this work, information acquired with the X-Met and its control was accomplished with keyboard macros that operated in the communication environment of the Lotus SYMPHONY program run in a Toshiba T1000 laptop computer. Additional information about this mode of operation will be given below.

The X-Met 840, shown in Figure 3, weighs about 29 pounds, including the weight of the probe, and is operated either with a battery supply or an AC operated power supply. A 12 volt DC supply is needed. The lead/acid gel-cell battery pack supplied with the system is specified to operate the instrument about 10 hours, but the one supplied with the instrument used in this work, when fully charged, would power the system for only about 3-4 hours. A much larger battery pack using similar cells and enclosed in an attache case was fabricated. It weighed about 28 lbs. and could power the X-Met and the computer for continuous periods of at least 24 hours.

The radioisotope source in the probe is located between the detector window and the sample. Radiation from the source hits the sample, and fluorescent radiation shines back from the sample around the source to the detector window. Sources of ^{109}Cd, ^{242}Pu, and ^{241}Am are commercially available; each source excites a different set of elements, depending on the energy of the radiation emitted by the source. An ^{55}Fe source is also available for exciting elements of low atomic number. The source used in this study, ^{244}Cm, emits a 14.2 KeV x-ray that will excite K x-rays of elements from titanium (atomic number 22) to selenium (atomic number 34) and L x-rays from lanthanum (atomic number 57) to lead (atomic number 82).

Both a laboratory probe and a contact probe are available. The contact probe is normally operated by placing it against a specimen for measurement; a trigger is pulled which withdraws a shutter (a shield) and allows radiation from the source to impinge on the external sample. In the case of the laboratory probe, samples are loaded in plastic cups,

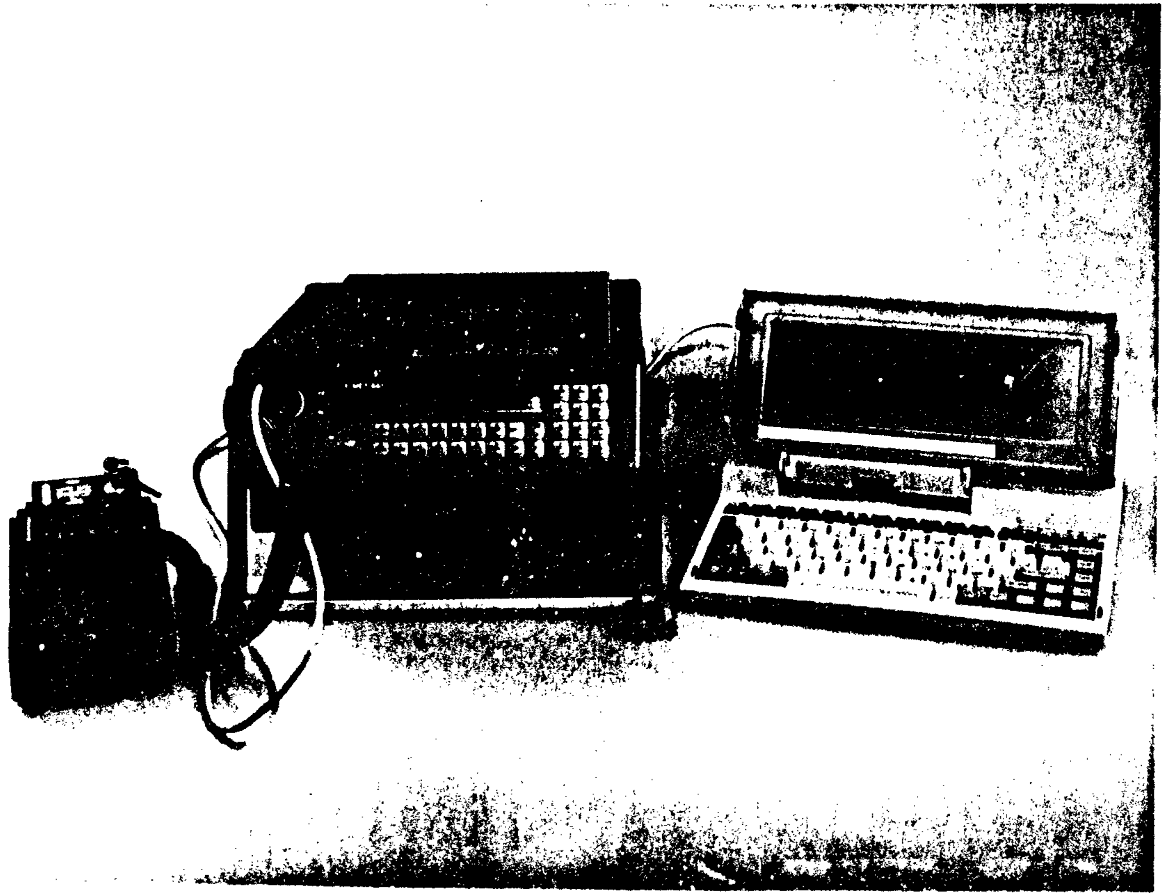

Figure 3. Photograph of CSI X Met Model 840 Portable X-Ray Fluorescence System

which are then placed in a chamber that is moved over the radioactive source when the lid of the probe is closed. Samples in the cups are covered with 0.06-0.1 mm of mylar or polypropylene film to serve as a window that is transparent to radiation from the source and x-rays generated in samples. Because the surface probe was received from the manufacturer immediately prior to the trip to RMA, it was not evaluated in the field. Thus, the laboratory probe was used for all of the work reported here.

The X-Met will function either as a system to identify alloys or to quantitatively measure the concentration of chemical elements in a sample matrix. This discussion will be limited to those features pertaining to elemental analysis.

Calibration

Calibration of the X-Met is described in the users manual (see references) and will only briefly be reviewed here. Calibration of the instrument is divided into a phase that pertains to instrumental factors and a phase that, in the case of chemical assay, pertains to factors related to the samples, i.e., those factors that relate x-rays intensities with element concentrations in samples. The instrumental stage, involves an initialization of the probe, automatic gain compensation, and measurements of single element standards to establish channel regions, "windows" that correspond to the energies of the full energy peaks for the fluorescent x-rays of the elements. The sample calibration stage for chemical assays involves measurements on a set of assay calibration standards containing known concentrations of the elements of interest to permit a multivariate regression model to be derived that gives the best fit of x-ray intensities and element concentrations.

The shutter on both probes has a pure element copper standard attached to the side facing the source. When the instrument is on and the shutter is closed, the copper standard is positioned over the source, and automatic gain control operates periodically to compensate for spectral shifts caused by temperature changes. The gain control operates by causing a brief count to be taken of the copper standard on the shutter. The gain control operates immediately when the instrument is turned on, and the shutter should be in the closed position (laboratory probe open) until the gain control parameters have been determined. Probe initialization is required when a probe is first placed into operation, and serves to establish initial values of the gain control parameters for that particular probe. Subsequent probe initializations are seldom required.

Instrument calibration is completed by measuring spectra of single pure elements that may be in the samples that are to be analyzed. These measurements permit the microprocessor to determine the channel locations of the full energy peaks of the elements as well as the channel of the source radiation that is coherently scattered (without loss of energy) off the sample. One of the "pure element standards" is a backscatter sample that, when measured, allows the analyzer to establish an equivalency of channel 255 and the coherent backscatter peak of the source and equate channel 0 to zero energy. In the present study, the backscatter standard was an aluminum foil. For other pure element standards, the channel locations of full energy peaks are determined by the linear relation that exists between energy and channel number. Peak overlap and background correction factors are stored at the time pure single element standards are counted. A number of single pure element standards are provided with the instrument. The pure single elements used in the calibration should include not only those that are to be measured but also those that might cause spectral interferences as well as those that might interfere by matrix effects. Those measured in this study were copper, arsenic, mercury, and lead as well as aluminum for a backscatter standard, and iron because of its somewhat large concentration in the soil matrix obtained from Rocky Mountain Arsenal and the fact that iron is known to cause a matrix effect interference for the measurement of copper. It should be noted, however, that matrix effects should be minor even with iron concetrations as high as those in the RMA soil.

Calibration for chemical assays is carried out by measuring a set of standards that contain the elements to be determined at a range of concentrations that spans the range that are expected to be encountered in "unknown" samples. The assay standards are prepared in a matrix that simulates that of samples to be measured as closely as possible. Elements whose assay is not sought but which cause interferences through matrix effects also need to be present and to span the concentration range that will be encountered in the matrix to be analyzed. One type of matrix effect arises when an element is present at relatively large concentrations that will absorb the radiation from the source and prevent it from reaching and exciting the atoms of the elements of interest that are buried deeply within the sample. For example, the iron present in many soils will absorb the 14.2 KeV x-ray of the ^{244}Cm source used in this study and decrease the intensity of the x-rays in the sample. Thus it is possible to have a set of soil samples that all contain the same concentrations of copper but varying levels of iron and have the observed copper exhibit varying concentrations.

Another type of matrix effect is also exemplified by the measurement of copper in soil that contains iron. The energies of the K x-rays of copper range from 8.0 to 8.9 KeV, and are sufficiently energetic to remove K electrons from iron and produce K x-ray fluorescence in iron. In this case, iron would represent a potential interference to assays of copper by absorbing the copper x-rays and prevent them from escaping from the sample. The same type of matrix effect from iron also affects measurements of arsenic, mercury and lead since the K x-rays of arsenic and the L x-rays of mercury and lead have sufficient energy to excite the K x-rays of iron. Because the energies of copper x-rays are closer to the so called absorption edge of iron than the x-rays of arsenic, mercury, and lead, the absorption cross sections are larger for copper and the resulting matrix effect would thus be larger for copper than for the other elements studied. Again a constant concentration of copper could be observed to vary if the iron concentration varied. This effect was not thoroughly investigated in this study, but it is not expected to be very significant at the levels of iron (1 to 2 percent) contained in the RMA soil. The multivariate regression models that can be derived with the X-Met software makes it possible to correct for this type of matrix effect if the variation of iron in the calibration standards varies to the same extent as it does in "unknown" samples. The model to permit the estimation of the concentration of copper, C_{Cu}, in the presence of iron can be represented by

$$C_{Cu} = K_{Cu,O} + K_{Cu,Cu}I_{Cu} + K_{Cu,Fe}I_{Fe}$$

where, the k's are constants determined in the regression modeling with the X-Met 840, and the quantities I_{Cu} and I_{Fe} denote the measured x-ray intensities of the copper and iron respectively. The constant $K_{Cu,Fe}$ is called the matrix effect coefficient. It should be noted that it is not necessary to know the concentrations of iron, but only necessary for the iron concentration to vary over a target range to be able to determine the correct matrix coefficient. The matrix coefficient will not be valid, and the observed concentration of copper will be in error, if the iron concentration falls outside the range for which the matrix coefficient was derived.

Preparation of standards

In the present work, soil standards were prepared from a large well-mixed specimen of soil from the Rocky Mountain Arsenal. Standards prepared in a water matrix made use of well water obtained at Oak Ridge National Laboratory. The multivariate regression calibration of the instrument was performed according to the method of Piorek and Rhodes (1988). Briefly, the calibration was accomplished by preparing 18-21 ten gram RMA soil samples and 18 10 mL ORNL groundwater samples. Each sample was spiked with some multiple of the targeted reporting limit (TRL) arbitrarily established at 35 ppm for the soil and water. The multiples of the TRL ranged from 0, and 0.2 x TRL to 100 x TRL. The spiking was performed in a random sequence, as described in Appendix A, so that there would be no correlation among any of the spiked concentrations of the four target elements. The individual soil samples were then homogenized and counted. The data, combined with the spike level information, was processed using the multivariate regression analysis techniques of the XRF's microprocessor. Information generated by the regression that is sufficient to analyze unknown samples is stored

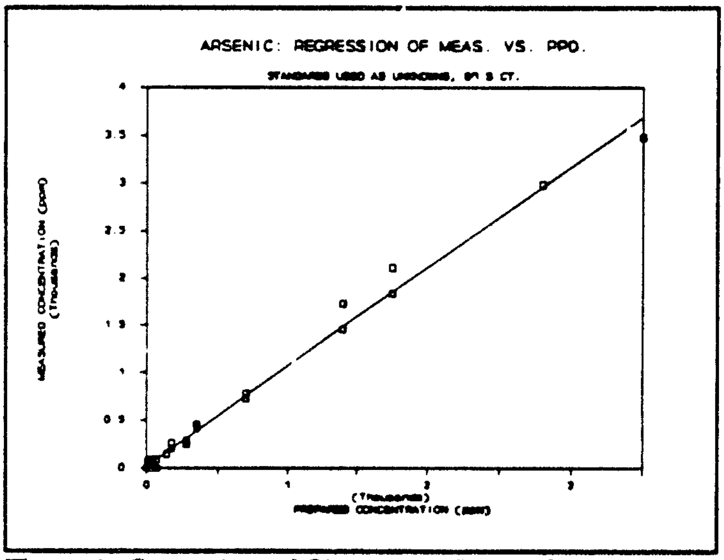

Figure 4. Comparison of Observed vs Spiked Concentrations for Arsenic in RMA Soil in the Presence of Copper, Lead, and Mercury.

in the instrument. For details of the sample assay calibration procedure, the reader is referred to the users manual which is listed as reference 1. A given multivariate regression (eg. wet soil spiked with all four target elements) is referred to as a "model." The X-Met 840 is capable of storing up to 8 such models. A list of the standards used to calibrate the instrument for both wet soil and groundwater is included in Appendix A. An example of the response linearity for a single element (in the presence of the other three target elements) is included in Figure 4.

According to USATHAMA guidelines, actual instrumental responses (peak heights, areas, etc.) must be used to perform Class 1 Certification. That is, standards of known concentration are used to obtain instrumental responses, and the linearity of that response is examined. The X-Met 840 is designed as a user-friendly instrument, and, unless commands are issued to the instrument to provide additional information, and reports observed concentrations of elements for which it has been calibrated previously using the stored multivariate regression program. In order to perform a single-variate regression analysis (such as that which the Class 1 Certification requires), direct instrument responses for each target element in each sample must be extracted from the microprocessor and stored externally, for eventual analysis. For the work reported here, this was accomplished by using a so-called keyboard macro routine written with the Borland program SUPERKEY. The macro was executed while communication existed between the communication environment of Lotus SYMPHONY and the X-Met. The macro would issue a series of commands to the X-Met as if they were being typed on the keyboard. The X-Met would then respond to the commands and send information resulting from the commands to the RS-232 interface where it was captured in the SYMPHONY worksheet. The commands issued by the macro caused the X-Met to transmit results for the gross counts, net counts, and standard deviations of the photopeaks corresponding to the elements of interest. A further discussion of the operating instructions and a list of macro commands are given in Appendix B. Typical output which is generated by the X-Met and stored for later analysis is given in Figure 5.

Precertification and Class 1 Certification were performed in the laboratory and the field, according to USATHAMA specifications. (A schematic diagram of the Class 1 Precertification and Certification as well as the Class 2 Certification used for the reference methods is provided in Appendix C.) Practical limitations necessitated some modifications in the usual procedures. These included not making up fresh standards on a daily basis, since such was impractical to perform in a field setting. Instead, the same standards were used repeatedly. However, this provided an additional quality control component by providing a day-to-day determination of a given standard. Lists of water and soil standards used for the certification and equivalency testing are provided in Appendix D, along with a typical day's output, explaining the use of the individual standards. Also, the IRPQAP software used to process the certification data is not designed to accept negative instrumental responses. However, the net channel intensities reported for many of the standards by the XRF system are negative. In order to compensate for this, many of the net channel intensities were altered by adding a fixed amount to each reported value.

Figure 5
Typical Output from X-Met 840

```
>
SAMPLE NUMBER           AM-16
SAMPLE TITLE

(MODEL 4:    RMA WATER)    Date: 20.12.88        Time: 12.54-17
Measuring    : 200 SECONDS
ASSAYS:CU    94.07 AS      51.41  HG   61.69 PB 486.0


>
STD
STDEVS:CU 3.367 AS 6.127 HG 5.816 PB 13.05


> PUL
CHANNEL PULSE FREQUENCIES:
CU:  161.1850 P


CHANNEL PULSE FREQUENCIES:   (MODEL 4: RMA WATER)
    CU         AS         HG         PB         BS
   161.2       177.0      137.6      501.7      2379


> INT
CHANNEL INTENSITIES:
CU: -210.7582 P

CHANNEL INTENSITIES:   (MODEL 4: RMA WATER)
    CU         AS         HG         PB         BS
  -210.8       28.28      -29.97     71.93      2379
```

This had the affect of making the determination of a nonzero intercept by the IRPQAP data processing package irrelevant.

The comparability of the XRF based method to standard laboratory based procedures was assessed using the EPA Equivalency Testing approach. This has been described in detail elsewhere (EPA, 1987). Briefly, sample aliquots are analyzed in duplicate over the course of ten or more days by both the reference and the test procedures. A schematic diagram of the equivalency test procedure is portrayed in Appendix E. For this work, the reference procedures were the USATHAMA standard analytical methods described above for the target elements. For the laboratory based equivalency test, no real environmental samples were available. Therefore, surrogate samples were fabricated by spiking a suite of ORNL groundwater and RMA soil samples with known amounts of the target contaminants. These spiked samples were treated as unknowns. In the field, samples were acquired from various locations, aliquoted, and analyzed in duplicate over the course of 10 or 11 days.

IV.A.3. Field Operations. The field phase of the study was conducted January 3-18, 1989, at Rocky Mountain Arsenal, Colorado. The ORNL field operation was established approximately 150 meters east of the trailers located north of the South Plants area. The location was chosen based on its proximity to the Ebasco Services support trailer and the decontamination trailer. Also, the orientation of the site provided some additional protection from south and southwesterly winds. The XRF system was set up inside a nylon backpacking tent, on top of a portable slide projector stand. Some portable shelter was required to diminish the potential for hypothermic injury to the instrument operators, and to shield the XRF system from wind-blown dust, rain, and snow. In addition, the shade prevented strong sunlight from excessively darkening the liquid crystal display screen on the laptop personal computer used to run the XRF system. Ambient temperatures ranged from 10°F. to 60°F. during sample analysis. Originally, a sample processing table was set up outside the tent, but was moved inside the tent when wind became a problem. Occasionally, a small backpacking stove was used to increase the temperature inside the tent for the comfort of the operators. In the field, the XRF system, including the laptop PC, was powered by a hand-carriable briefcase unit containing four lead/acid gel cell batteries. A small, battery powered heater for the PC screen was constructed and available to prevent the LCD screen from blacking out at subfreezing temperatures. However, such a problem was not experienced.

Certification samples and standards were identical to those used for the laboratory certification. During the daily experiments, the samples were kept in flat plastic cake containers, the bottoms of which were lined with moistened blotter paper to prevent the samples from drying out. These in turn were stored inside a thermally insulated chest to prevent freezing. At night, the samples and standards were taken indoors.

One water and four soil samples were acquired for the field Equivalency Test at each of five locations. Soil (approximately 2-3 kg) was removed near the surface (in most cases, the ground was frozen) and placed into flat cake pans. In Table 1 are listed the location of the sampling sites and the sample designation.

For the field work, a Health and Safety Plan was developed in close cooperation with Ebasco and USATHAMA personnel, and approved by USATHAMA project management. All environmental samples were acquired in the presence of an Ebasco Services Health and Safety Officer. Strict adherence to all safety and hazardous materials handling procedures was maintained. The samples were returned to the ORNL experimental area. Large stones were removed manually. The soil samples were homogenized by placing them in large plastic bags and manually shaking them.

Table 1

Sampling Locations for Field Equivalency Test Environmental Samples

Sample Designation	Location
Silo (Soil)	Approximately 10 meters S of silos area of South Plants region.
Pit (Soil)	Immediately inside adjacent to the north wall of a spray pond located in Section 2
2-18 (Soil)	Approximately 30 m north of the warehouses at Site 2-18, on the side of a drainage ditch.
2-8 (Soil)	Between two concrete pads at Site 2-8
1703 (Water)	Removed from a sump pit on the north side of the interior of Building 1703

IV.B. Results and Discussion

IV.B1. Automated Data Acquisition. Continued use of the X-Met 840, for all but the most limited data gathering operations, pointed to the need for automated data acquisition and management. Experience in our laboratory indicated that the time required to manually collect the data generated would considerably lengthen the time required to conduct a suite of analyses. The number of commands needed to extract net photopeak count rates (required for Class 1 Certification) from the XRF unit's microprocessor is considerable and requires a substantial amount of time if not performed via computer controlled interrogation. For the field effort at Rocky Mountain Arsenal, attempting to perform both USATHAMA Class 1 Certification measurements and EPA Equivalency Testing on both soil and water samples necessitated the analysis of as many as 90 samples and standards in a given workday. Benefits of computer controlled data acquisition include time savings on site, the lack of data transcription errors, and the ability to capture the data directly into a spreadsheet program for data processing at a later time. An additional advantage is that an actual x-ray pulse height spectrum can be viewed in the field on the computer screen. This provides the operator with a visual confirmation of authenticity of the data being reported by the instrument. The choice of Lotus Symphony as a data acquisition/management software package was based on the operators' familiarity with the system, and its ability to run the XRF and acquire data via its "communication environment," as well as managing the data in its "spreadsheet environment." One significant advantage of Symphony over many other communication programs is that data is captured in the worksheet rather than on a disk drive. It was therefore only necessary to make use of the floppy disk drive of the Toshiba T-1000 twice each day to store the information that had been captured in the worksheet. This method of operation resulted in a considerable savings of power from the portable supply that otherwise would have been used to operate the floppy drive. Other software packages were not evaluated, but presumably would function similarly.

IV.B.2. Choice of Appropriate X-Rays. Given the relatively low resolution of the system detector, the choice of an appropriate x-ray photopeak to use for quantitation can be difficult. For example, in Figure 6 are portrayed the pure element spectra for the target elements, plus iron. Iron is included because of its relatively high concentration in the RMA soil (see Figure 7). The simultaneous determination of lead and arsenic in environmental samples is particularly difficult. This is due to the fact that the K_α x-ray of arsenic and the L_α x-ray of lead possess nearly the same energy. Thus, the L_β x-ray is used to quantitate lead when arsenic is present. The abundance of this x-ray is about half that of the L_α x-ray. As a result, the detection limit for lead was about twice as large as those for the other elements. This also tends to increase the uncertainties associated with the determination of arsenic, since to perform that determination in the multivariate regression analysis approach used by CSI, lead must first be determined on the basis of its L_β x-ray. Then, the combination photopeak is used to estimate the amount of lead plus arsenic present. That sum is then adjusted for the presence of lead, and the remainder is taken as arsenic. The effect of having to compare two values to calculate a third is manifested as an inverse correlation between arsenic and lead concentrations as the photopeak intensities vary due to experimental uncertainties. For example, in Figure 8 is plotted the apparent As level as a function of the apparent Pb concentration for a spiked water sample measured repeatedly in the laboratory over the course of two weeks. Actual spike levels of As and Pb were 35 and 420 ppm, respectively. The reported values were calculated using the manufacturer's multivariate regression analysis. The degree of correlation is quite large ($R^2 = 0.926$).

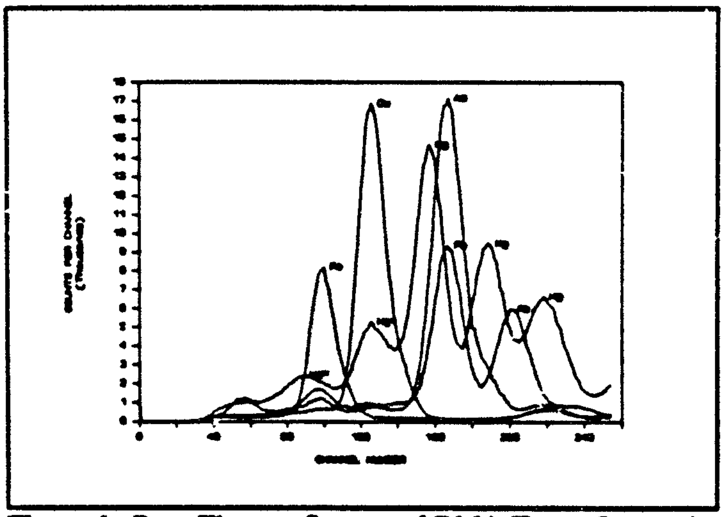

Figure 6. Pure Element Spectra of RMA Target Inorganics Plus Iron Acquired with X-Met 840. [Hg* indicates Xenon x-ray escape peak.]

IV.B.3. Determination of Instrumental Limits of Detection. The instrumental limits of detection (LOD's) for all four elements were determined for both water and wet soil samples. This data is listed in Table 2. This was accomplished by performing careful measurement of the background counting rate at the selected channels for the target elements, and comparing the resulting counting statistic to a concentration which would generate an equivalent counting rate. The lead LOD is higher than for the other elements due to spectral interference with arsenic, necessitating use of a lower intensity peak for detection of lead. Two calibration models were made for the portable XRF to determine lead concentration in the absence of the other compounds of interest, thereby allowing the use of the higher intensity lead peak, for both water and wet soil samples. Subsequent lead LOD determinations using these two models yielded much lower LOD's. However, time constraints did not permit a complete evaluation of the lead-only model in the laboratory or field.

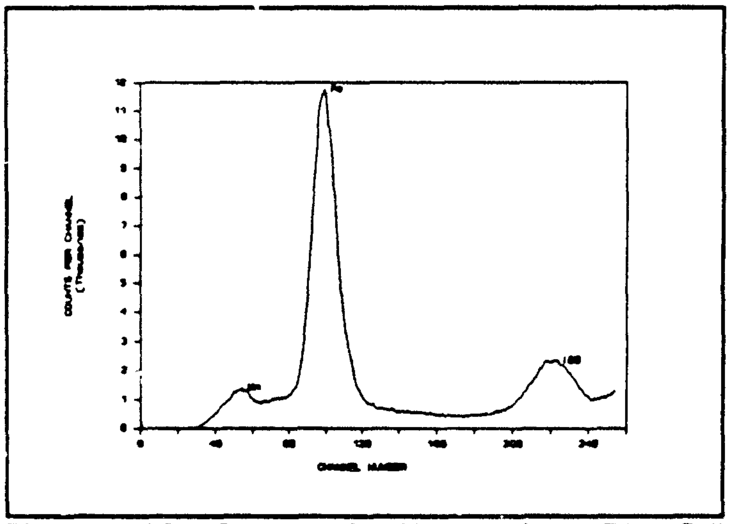
Figure 7. X-Ray Spectrum of an Uncontaminated RMA Soil Acquired with X-Met 840.

IV.B.4. Influence of Environmental Parameters.

Temperature

Changes in ambient temperature can affect the gain of the amplifiers in the X-Met 840. As long as the gain control is permitted to make periodic adjustments, the unit will compensate for the influence of temperature on its energy scale. For example, tests conducted at ORNL at both 35°F. and 72°F. with pure element standards indicated essentially no changes of photopeak maxima for all four of the target elements. However, under the working conditions experienced in the field at RMA (large sample load and widely ranging temperatures), the time required to periodically permit gain adjustment can place an additional burden on the instrument operator. Under such conditions, we found it more practical to insulate the probe head by placing it inside a small thermally insulated chest, which contained warmed "blue ice" as thermal ballast. This eliminated the need for the operator to remember to halt analyses and permit gain control adjustment, as the temperature of the probe remained nearly constant.

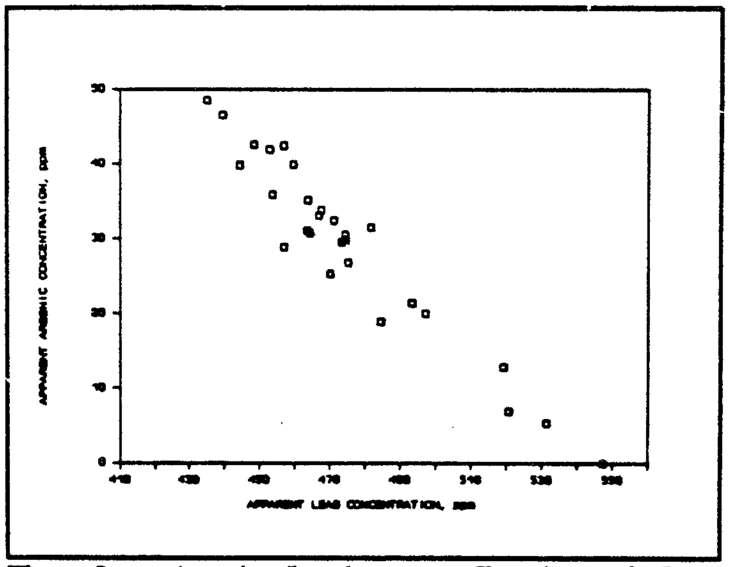

Figure 8. Arsenic Level as a Function of Lead Concentration. Laboratory Analysis of Water Standard AM-14.

Table 2

Instrumental Limits of Detection*
Portable X-Ray Fluorescence Unit
Limits of Detection

	Multi-element	Multi-element	Multi-element	Single-element (L-Alpha)	Multi-element (L-Beta)
Sample Matrix	CU (ppm)	AS (ppm)	HG (ppm)	PB (ppm)	PB (ppm)
Water	8.9	3.5	5.5	9.5	42
Wet Soil	25	12	29	28	60

*Defined at the ppm equivalent to 3 times the square root of the background count rate added to the background count rate.

Atmospheric Pressure

Because of the considerable difference in elevation between ORNL and RMA, the effect of changes in ambient pressure were determined. This was accomplished by determining the response to pure element standards both at ORNL (approximate elevation 800 ft.) and at Newfound Gap in the Great Smoky Mountains National Park (elevation 5040 ft., similar to that of Rocky Mountain Arsenal). The change in elevation caused the count rate of a copper pure element standard to increase by 3%. Estimates of the attenuation of the Curium-244 14 Kev x-ray (used to excite the sample) from the source to the sample and of the 8 Kev copper x-ray from the sample to the detector indicated that the difference in air density at the two altitudes could account for about half the 3% difference. From a practical standpoint, this small change was not considered to be important. No attempt was made to quantify the influences in background radiation at the two comparison sites.

Soil Moisture Content

A systematic study of the impact of soil moisture content on XRF performance revealed that the most efficacious approach to field analysis of soil would be to insure that the soil being tested was visibly moist. The data obtained from the ruggedness test for soil moisture content is listed in Table 3, both as concentrations (determined using the wet soil calibration model) and as percent difference of the mean for 10% moisture content (1 mL water added to 10 grams dry spiked soil). The soil moisture content was varied from zero percent (actually equilibrated with atmospheric moisture) to 20 percent in increments of five percent. The data indicated that although there was a large difference between dry soil (0% soil moisture content) and 10% soil moisture content results, there was a much lower difference between the 5% to 20% soil moisture content means (N=7). (20% soil moisture content was determined to be near the saturation level for RMA soil.) Thus, from a practical standpoint under a field situation, it appeared acceptable to treat all wet soils as equivalent.

Iron Content of Soil

The presence of percent quantities of iron in the soil samples can influence the apparent concentration of copper in two ways. First, because of the proximity of the energies of the Curium-244 exciting x-rays, the emitted x-rays of copper, and the absorption edge of iron, the iron can attenuate the incoming Curium-244 x-ray intensity, as well as that of those emitted by the copper. The fraction of attenuation should be constant over the range of copper concentrations. Also, since the iron photopeak maximum (observed at XRF detector channel #99) is so close to that of the copper (channel #125), the "tail" of a large iron peak can overlap with the copper photopeak maximum. Since the "background" intensity is subtracted from the measured intensity to obtain the net intensity for any given photopeak, small variations in the magnitude of the relatively large iron "tail" due to sample inhomogeneity may have a substantial effect on the net photopeak intensity ascribed to the copper. Changes in the magnitude of the photopeak tail under the copper photopeak which are due to actual concentration changes in the sample iron content would be expected to have an even larger effect.

In order to determine the magnitude of the changes in the iron concentration on the apparent copper concentration, silica was analyzed unspiked, spiked with 2000 ppm copper, and spiked with both 2.2% iron and 2000 ppm copper. Analysis of seven replicates of each of these three samples gave data showing a 12% difference in the mean copper intensity for 0% iron versus 2.2% iron. This is a statistically significant difference; however, we did not expect to find a variation in iron content this large in the environmental samples at RMA. Indeed, the iron content of the RMA reference soil, from which the certification and equivalency standards were prepared, was estimated from XRF analysis to have an iron content of 1.5%. This was in relatively good agreement with the inductively coupled plasma analysis of a sample of RMA soil analyzed at ORNL. Results of that analysis are reported in Table 4. The iron concentration of the field equivalency samples was estimated from the XRF measurements to range from 1.0% to 1.9%. Thus, the variation in the iron content of the soil samples was not expected to alter the apparent concentration of the copper by more than a few percent at high copper concentrations. At low copper concentrations, the effects were expected to be more pronounced.

Sample Homogeneity

The homogeneity of soil samples is an important aspect of quantitation. Soil particle size, and cracks and fissures in the soil can affect the extent to which exciting x-rays can penetrate the sample, and emitted x-rays can leave the sample. The visual assessment of sample homogeneity seems too subjective. In Table 5 are reported the mean net intensities of the iron x-ray photopeaks for various types of soil samples analyzed. The precision of these measurements was taken as an indicator of sample homogeneity, since iron is present in an easily measurable concentration, and it was assumed to be at a constant level within a given sample type. The control sample, a wet soil, was analyzed daily throughout the certification and equivalency testing at RMA. The high precision indicates that it is possible to make repeated measurements of the same sample quite reproducibly. The calibration standards are different aliquots of the same large batch of RMA soil spiked individually. The dry soil standard, run as a control each day, is much more subject to variation, probably due to the settling which occurs with repeated handling. Comparison of the wet and dry soil standards indicate that the former are less susceptible to changes in homogeneity with time. Although the iron content was different for each of the equivalency samples, the precision of the iron measurement was better than ± 10% for all four soil batches. Thus, sample inhomogeneity appears to be only a small contributor to sample-to-sample variation under these conditions.

Table 3

Apparent Response to Analyte Spike as a Function of Soil Moisture Content

Apparent Elemental Concentration[a]
Mean ± Standard Deviation[b]

Moisture Content (% by weight)	CU (ppm)	AS (ppm)	HG (ppm)	PB (ppm)
0%	1066.9 ± 32.9	937.4 ± 28.6	1148.6 ± 45.1	1092.4 ± 37.7
5%	817.6 ± 11.8	716.2 ± 12.7	716.6 ± 9.6	798.6 ± 23.1
10%	733.3 ± 13.7	687.6 ± 12.2	682.1 ± 41.9	764.9 ± 23.7
15%	650.7 ± 13.0	732.6 ± 11.7	744.7 ± 35.0	712.6 ± 27.6
20%	757.2 ± 10.4	756.3 ± 16.5	798.4 ± 30.9	783.4 ± 51.6

[a]Spiked Concentration = 700 ppm
[b]N=7

Percent Difference from 10% Soil Moisture Content

Moisture Content	CU	AS	HG	PB
0%	+45.5	+36.3	+68.4	+42.8
5%	+11.5	+ 4.2	+ 5.1	+ 4.4
10%	+ 0.0	+ 0.0	+ 0.0	+ 0.0
15%	- 11.3	+ 6.5	+ 9.2	- 6.8
20%	+ 3.3	+10.0	+17.0	+ 2.4

Table 4

Levels of Inorganic Species in Rocky Mountain Arsenal Reference Soil as Determined by ICP Analysis

Species	Level, μg/g soil
Silver	< .98
Aluminum	52,000
Arsenic	< 9.8
Boron	18
Barium	770
Beryllium	2.6
Calcium	13,000
Cadmium	< .33
Cobalt	4.7
Chromium	20
Copper	9.3
Iron	19,000
Gallium	< 49
Lithium	45
Magnesium	4,200
Manganese	290
Molybdenum	< 6.5
Sodium	12,000
Nickel	12
Phosphorus	520
Lead	20
Antimony	< 8.1
Selenium	< 9.8
Silicon	4,400
Tin	< 8.1
Strontium	230
Titanium	1,800
Vanadium	46
Zinc	44
Zirconium	80

Table 5

Net Intensities of Iron in Soil Samples as an Indicator of Sample Homogeneity

Sample	Net Intensity of Iron Photopeak (Count Rate) [Mean ± One Standard Deviation, (RSD%)]	
Control Sample SCS-M (Wet Soil Blank)	596.8 ± 1.8	(0.3%)
Calibration Standards [SM-1 thru SM-21]	524.9 ± 33.8	(6.4%)
Dry Soil Standard #26	609.6 ± 74.4	(12.2%)
Equivalency Test Samples		
Silo	542.6 ± 24.1	(4.4%)
2-8	777.2 ± 46.4	(6.0%)
Pit	760.9 ± 34.1	(4.5%)
2-18	392.5 ± 34.6	(8.8%)

IV.B.5. Class 1 Certification

Class 1 Certification was used as a tool to evaluate the efficacy of the analytical method, rather than to certify the method for use. In Table 6 are reported the USATHAMA Class 1 Certified Reporting Limits (CRL's) for the portable XRF system under both field and laboratory conditions for RMA soil and ORNL groundwater. (F-ratio analyses are reported in Appendix F.) That CRL's were obtained for most of the target elements indicates that it is possible to obtain quantitative results in the sub-1000 ppm range very rapidly with a field portable instrument with virtually no sample processing. While variations in the soil iron content did not alter the apparent levels of copper when the latter was present at high concentrations, it was not possible to certify for copper in the RMA soil starting with the relatively low levels of the Targeted Reporting Limit (TRL) chosen for this study (25 and 50 mg/kg). This may be due to the proximity of the tail of the large x-ray photopeak from iron in the soil to that of the copper. However, the calibration curves for copper generated for the equivalency testing indicate that the net intensity of the copper photopeak increases linearly with copper concentration above ca. 100 ppm. (See Figure 9.). It is our opinion that the XRF method could have been certified for copper in soil if higher TRL's had been chosen. In all cases, the CRL's for the water matrix were smaller than those for the soil. This is most likely due to the greater inhomogeneity among the soil samples, and the higher background due to scattering of the x-rays off of the individual soil particles. Indeed, the instrumental limits of detection (see above), largely a function of the background

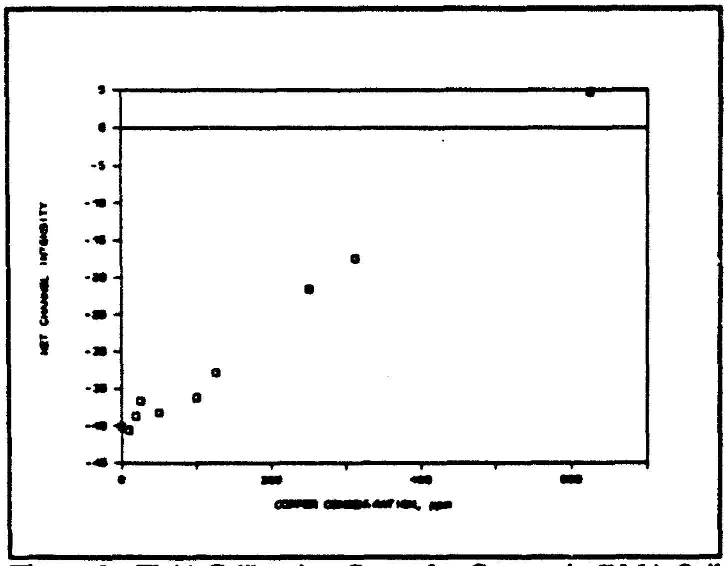

Figure 9. Field Calibration Curve for Copper in RMA Soil from Precertification Runs. Data Included from Both 1x and 2x the Targeted Reporting Limit Experiments.

Table 6

Certified Reporting Limits for CSI X-MET 840 Portable X-Ray Fluorescence Analyzer

Rocky Mountain Arsenal Soil	Copper		Arsenic		Mercury		Lead	
	Laboratory[a]	Field[b]	Laboratory	Field	Laboratory	Field	Laboratory	Field
Certified Reporting Limit (μg/g)	FC	FC	63.2	137	144	187	FC	192
Slope			0.831	1.013	0.947	0.885		0.935
Correlation Coefficient[c]			0.859	0.837	0.965	0.803		0.943
ORNL Groundwater	Laboratory	Field	Laboratory	Field	Laboratory	Field	Laboratory	Field
Certified Reporting Limit (μg/l)	19.4	57.8	44.1	121*	16.7	48.8	82.9	176
Slope	0.969	0.965	1.033	0.925*	0.881	1.046	0.949	0.934
Correlation Coefficient	0.993	0.974	0.809	0.362*	0.948	0.895	0.994	0.975

[a]Laboratory measurements performed at Oak Ridge National Laboratory

[b]Field Measurements Performed at Rocky Mountain Arsenal

[c]Slope and correlation coefficients are for single variate regression analysis of observed vs prepared concentrations, according to USATHAMA IRPQAP software.

*Considered to have failed Class 1 certification because of low correlation coefficient.

FC: Failed Class 1 certification

counting statistics are substantially larger for the soil samples. The relatively low correlation coefficients for low concentrations of arsenic in water appear to be related to the overlapping nature of the arsenic and lead photopeaks.

In general, the CRL's determined in the field are larger than those determined in the laboratory. This appears to be due to the cumulative effects of performing analyses under a much less well controlled environment, since the same samples were analyzed for both the field and the laboratory certifications. This increased field variability is also observed in the multivariate calibrated determination of contaminant levels in spiked soil and water samples in both the field and the laboratory. In Table 7 are reported the means and standard deviations of selected standards repeatedly analyzed through the course of the certification and equivalency testing. These data are portrayed graphically in Figures 10 - 13. In general, the relative standard deviations are greater in the field.

Table 7

Comparison of Spiked vs Measured Concentrations (Multivariate Regression)
Laboratory and Field (RMA) Analyses of Soil and Water Standards*
[Mean ± One Standard Deviation, in µg/g Sample (RSD%)]

	Copper	Arsenic	Mercury	Lead
Soil Standard SM-16				
Spiked Concentration	313	150	363	750
Laboratory Measured[a]	357 ± 11 (3.1%)	123 ± 17 (13.8%)	306 ± 29 (9.5%)	822 ± 38 (4.6%)
Field Measured[b]	371 ± 16 (4.3%)	206 ± 45 (21.5%)	250 ± 31 (12.4%)	695 ± 86 (12.6%)
Water Standard AM-14				
Spiked Concentration	89	35	55	420
Laboratory Measured[c]	83 ± (4.8%)	29 ± 12 (41.4%)	54 ± 8 (14.8%)	475 ± 27 (5.7%)
Field Measured[d]	83 ± (4.8%)	91 ± 31 (34.1%)	38 ± 8 (21.1%)	349 ± 64 (18.3%)

*Determinations performed over the course of 11 days. No data excluded for determination of means.

a: N = 23
b: N = 25
c: N = 28
d: N = 27

It is important to recognize the limitations of the conclusions regarding the certification experiments. As performed here, attempting to develop calibration models using all four of the target contaminants simultaneously, the evaluation is that of a worst case situation in the determination of unknown quantities of elements. Given the relatively low resolution of the XRF detector, which permits the presence of one element to influence the apparent photopeak intensity of another, it would be more likely that in a field situation with repeated use, calibration models or curves would be developed for a number of individual situations.

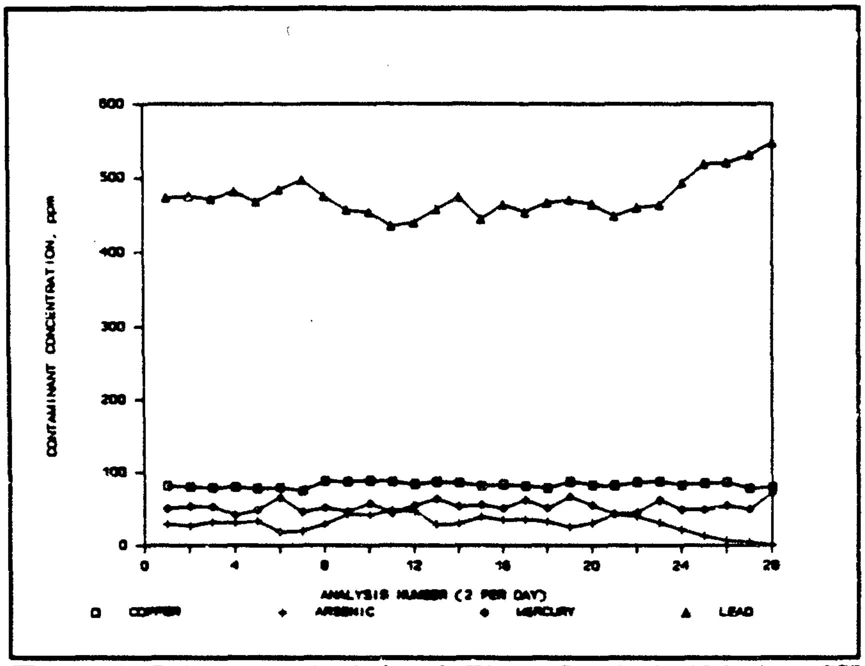

Figure 10. Laboratory Analysis of Water Standard AM-14. CSI (Multivariate) Calibration.

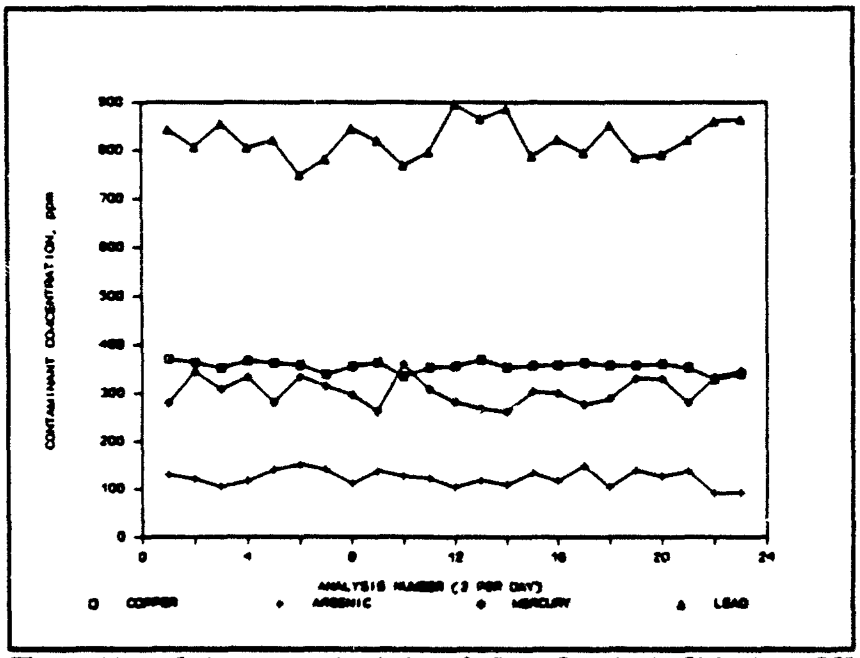

Figure 11. Laboratory Analysis of Soil Standard SM-16. CSI (Multivariate) Calibration.

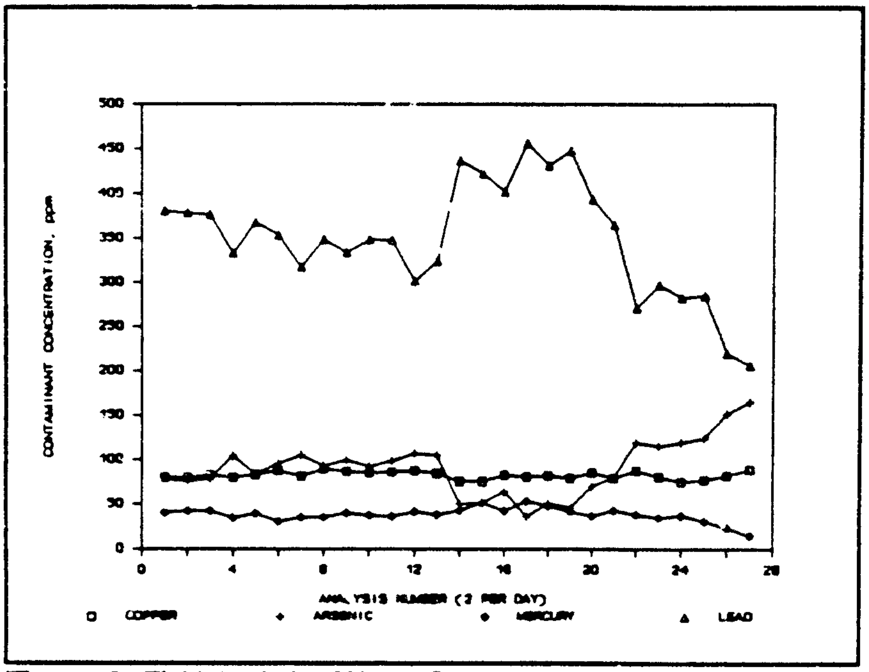

Figure 12. Field Analysis of Water Standard AM-14. CSI (Multivariate) Calibration.

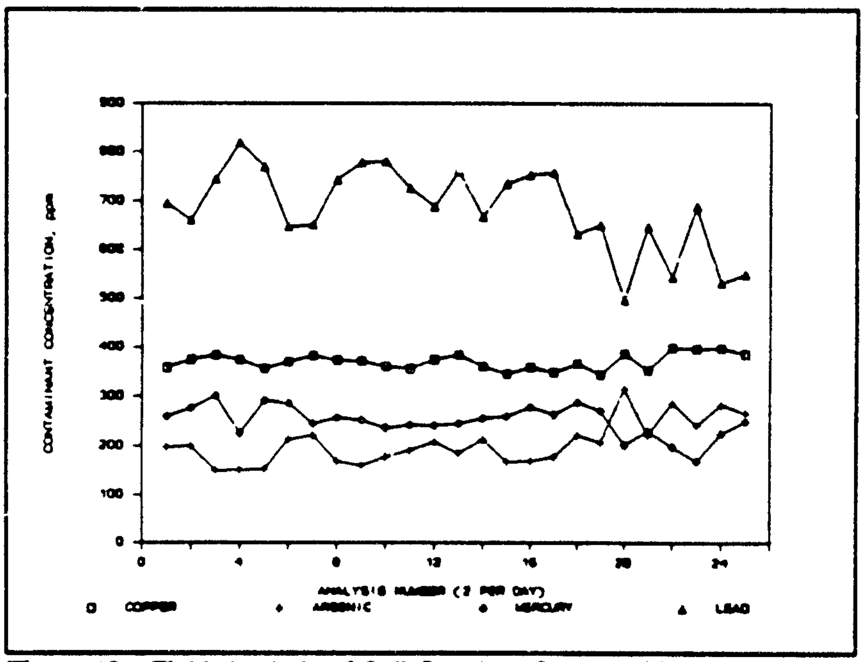

Figure 13. Field Analysis of Soil Standard SM-16. CSI (Multivariate) Calibration.

IV.B.6. Comparison of Single Variate (USATHAMA) Regression Calibration with Multivariate (CSI) Regression Calibration

As part of the certification study, a secondary issue was addressed as to whether the USATHAMA approach to calibration, involving the use of single variate regression, was more accurate or precise than the manufacturer's approach, which involves the use of multivariate regression analysis. The X-Met 840 converts the intensity in the channel of a specific element to elemental concentration by employing an algorithm which uses empirical coefficients and linear multi-parameter regression. The concentration of the analyte (i.e., the element being measured) is assumed to be the linear sum of contributions from all element net intensities, each net intensity being multiplied by a coefficient determined empirically during calibration.

The X-Met 840 contains the software necessary to calculate the regression coefficients, together with statistical criteria to help the operator select the most accurate option in any particular case. The general equation used by the instrument to convert net intensities (I_j) to element concentrations (C_i) is

$$C_i = r_{i0} + \sum_{j=1}^{6} r_{ij} f_j \qquad 1.$$

A maximum of six elements, designated by subscript i, can be selected for concentration readout from the ten element channels maximum in each calibration model.

The f_j, are intensity-related independent variables that can have any of the following forms:

$$f_j = I_j$$

$$f_j = I_j/I_{BS}$$

$$f_j = I_j \cdot I_k$$

$$f_j = I_j \cdot I_k/I^2_{BS}$$

where j, k are any of the ten element channel numbers, I_j & I_k are the net intensities of the corresponding element channels and I_{BS} is the net intensity of the backscatter channel. Note that the backscatter channel must be one of the ten allowed element net intensity channels.

The empirical regression coefficients are r_{ij} and one of them, r_{i0} is the intercept coefficient. r_{ij}, where, $i = j$, is the slope coefficient for element i and r_{ij}, where $i \neq j$, are the matrix correction coefficients.

Equation 1, when written out in full, is:

$$C_1 = r_{10} + r_{11}f_1 + r_{12}f_2 + r_{13}f_3 + r_{14}f_4 + r_{15}f_5 + r_{16}f_6$$

$$C_2 = r_{20} + r_{21}f_1 + r_{22}f_2 + r_{23}f_3 + r_{24}f_4 + r_{25}f_5 + r_{26}f_6$$

.

.

.

$$C_6 = r_{60} + r_{61}f_1 + r_{62}f_2 + r_{63}f_3 + r_{64}f_4 + r_{65}f_5 + r_{66}f_6$$

Thus, in the calibration mode, C_i and f_{i}, are known, and the regression coefficients calculated and stored. When matrix effects are important, or when there is significant overlap between the energies of the element specific channels, the multivariate regression approach seems more likely to give a more accurate result than a single variate regression, which does not take element to element interactions into account. The single variate regression, in the form of $C_i = r_0 + r_1 \cdot I_i$, is used in most calculation routines with conventional instrumentation because minimal element:element interaction is present in such methods. The single variate approach is essentially that used in the USATHAMA QA program.

In Table 8 are compared values for surrogate environmental samples (matrix spikes treated as unknowns) determined using both single and multivariate regression. Several observations are appropriate. First, in only four of the 16 determinations are the calculated means greater than two standard deviations from the spike level. This indicates that under the conditions of measurement, the accuracy of both methods is comparable. However, in five of the 8 pairs of values, there is a statistically significant difference between the means of the two types of calculated values ($p \leq 0.05$). Of those five cases in which a difference exists, the single variate regression calculated mean is somewhat more accurate in three. This data indicates that for these conditions and contaminants, there is no clearly superior method of determining the quantities of unknown constituents. From a field utility standpoint, the multivariate regression approach has the advantage of the calibration curve being stored in the unit's microprocessor, such that an immediate determination of the contaminant concentration can be made.

IV.B.7. Equivalency Testing and Analysis of Field Samples

Results of the field XRF analyses of the samples collected at RMA for the equivalency testing are summarized in Table 9. The original data for all of the analyses are reported in Appendix G. Several observations are in order.

Table 8

Analysis of Surrogate Equivalency Test Samples
Comparison of Single Variate (S) and Multivariate (M) Regression Calibration Methods
Contaminant Levels, $\mu g/g$ Sample Mean ± One Standard Deviation (RSD%)

Sample	Regression Type	Copper	Arsenic	Mercury	Lead
Soil	M	122 ± 11 (9%)[a]	96 ± 20 (21%)	156 ± 21 (14%)[a,b]	107 ± 41 (38%)
Soil	S	106 ± 12 (11%)	93 ± 25 (27%)	118 ± 33 (28%)[a]	115 ± 31 (27%)
Soil Spike Level		100	100	100	100
Water	M	96 ± 4 (5%)[a]	44 ± 14 (33%)[a]	84 ± 5 (6%)[a,b]	111 ± 31 (28%)
Water	S	78 ± 3 (4%)[a,b]	53 ± 9 (17%)	79 ± 5 (6%)[a,b]	107 ± 15 (14%)
Water Spike Level		100	50	100	100

[a]Multivariate calibrated value significantly different from that using single variate regression at 95% confidence level.

[b]Mean value greater than two standard deviations from spike level.

Table 9

Comparison of Apparent Contamination Levels
Soil and Water Samples Collected at Rocky Mountain Arsenal
Multivariate Regression (M) vs Single Variate Regression (S)
(N = 20,22)
Contaminant Levels $\mu g/g$ Soil [Mean ± One Standard Deviation (RSD%)]

Sample	Regression Type	Copper	Arsenic	Mercury	Lead
Soils					
Silo	M	226 ± 27 (11.9%)	2517 ± 124 (4.9%)	0	141 ± 63 (44.7%)
Silo	S	198 ± 20 (10.1%)	3310 ± 277 (8.4%)	0	529 ± 84 (15.9%)
Pit	M	415 ± 43 (10.4%)	0	137 ± 29 (21.1%)	164 ± 110 (67.1%)
Pit	S	385 ± 44 (4.4%)	0	185 ± 83 (44.9%)	301 ± 59 (19.6%)
2-18	M	0	130 ± 50 (38.5%)	66 ± 18 (27.3%)	0
2-18	S	0	10 ± 36 (360%)	0	0
2-8	M	365 ± 28 (7.7%)	9.1 ± 18 (250%)	194 ± 20 (10.3%)	798 ± 117 (14.7%)
2-8	S	334 ± 27 (8.1%)	0	313 ± 60 (19.2%)	852 ± 72 (8.5%)
Field Certified Reporting Limit for Soils			137	187	192
Water					
1703-Diluted	M	0	21,600 ± 6100 (28%)	0	0
1703-Diluted	S	0	11,000 ± 3100 (28%)	0	0
1703-Undiluted	M	15 ± 3 (21%)	3310 ± 18 (0.5%)	0	108 ± 47 (44%)
1703-Undiluted	S	262 ± 10 (4%)	3850 ± 944 (19%)0		234 ± 93 (40%)
Field Certified Reporting Limit for Water		38	-	49	176

The one water sample was collected from a sump pit in an abandoned building. Analysis indicated that the water had a considerable amount of arsenic in it. In Figure 14 is portrayed graphically an XRF spectrum of the undiluted water sample. Exact determination of the amount of arsenic was problematic. The levels determined in the undiluted water sample were considerable beyond the range of both the USATHAMA and the CSI calibration ranges. Interestingly, the precision of the multivariate calibrated determinations was very high. In order to make a measurement within the calibrated range, it was necessary to dilute the sample by 200-fold using distilled water purchased in a local grocery store. This yielded a much higher apparent arsenic level in the sample. However, there is nearly a factor of two difference between the levels determined by the two calibration methods. This is likely due to the relatively high degree of uncertainty in the calibration curve for arsenic being extrapolated over 2 - 3 orders of magnitude. For the soil samples, two possessed contamination above the certified reporting limits: the arsenic levels in the Silo sample and the lead levels in the 2-8 samples.

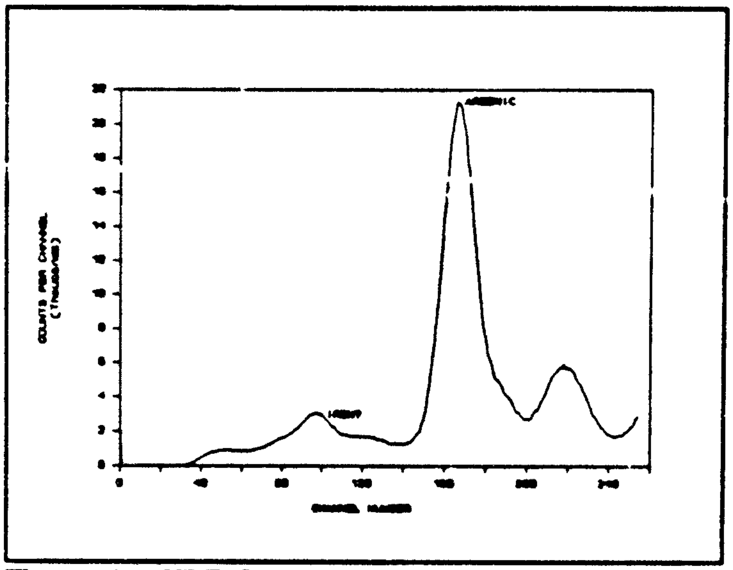

Figure 14. XRF Spectrum of Undiluted Water Sample Acquired from Sump Pit in Building 1703.

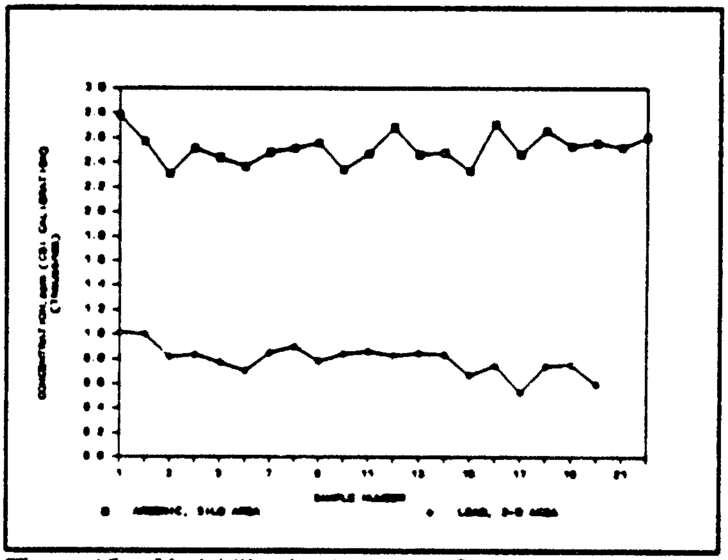

Figure 15. Variability in Apparent Contaminant Level in Soil Samples Acquired at RMA. Multivariate Calibration Used.

Recall however, that the calibration curve for copper is fairly linear above 100 ppm, so that it might be assumed that any copper concentrations reported above ca. 200 ppm (eg., 2-8 and the Pit samples) are probably real. While the arsenic level in the Silo sample is about a factor of 10 beyond the calibration range for arsenic (ca. 300 ppm), there was no practical way to dilute the soil samples in the field, and no additional spiking solutions were available in the field to make up new soil standards. However, precision for all of the determinations of the arsenic and lead in the Silo and 2-8 samples was very good for either the multi- or single-variate calibration ($\leq$ 15%). This is portrayed graphically in Figure 15.

A more definitive approach to comparing the accuracy and precision of the XRF method with that of the laboratory based systems is the use of the EPA Equivalency Test Petition or procedure. The test is portrayed schematically in Appendix E. The values determined by the laboratory methods are given in Appendix H. A detailed description of the statistical analysis of the data is provided in Appendix I. In Table 10, results of the laboratory method analyses for the samples are summarized. A few comments are in order. First, because of difficulties with the laboratory instrumentation, the test samples were not analyzed until the end of July, 1989. This is approximately 6 1/2 months after the samples were acquired in the case of the field samples and approximately 8 months after the surrogate samples were spiked. The USATHAMA reference methods A-8 and L-8 specify pre-analytical holding times of 6 months and 28 days for arsenic and mercury in water, respectively. Clearly, these holding times were exceeded, albeit by a relative small fraction of the allowable time in the case of arsenic. No holding times are specified for the target elements in soil, or copper or lead in water. Interestingly, the agreement between the laboratory analytical results for the surrogate water samples and the spike levels was very good for arsenic and mercury (49 ± 2 ppm vs 50 ppm spike for arsenic, and 102 ± 11 ppm vs 100 ppm spike for mercury). This would suggest that the specified pre-analytical holding times are overly conservative for these elements in water. However, the apparent stability of the samples may be due in part to the addition of ethylene diamine tetracetic acid (EDTA) to the surrogate water samples. This was done because in the calibration process, we discovered that precipitates would form in the higher concentration calibration standards. EDTA was added to prevent the precipitation.
Regarding the possible spectral interference of EDTA, disodium-EDTA consists of sodium, carbon, hydrogen, oxygen, and nitrogen atoms. Of these, sodium emits the highest energy fluorescence x-ray, with an energy of 1.04 keV. Since the lowest energy K x-ray for the analytes of interest is 8.05 keV for copper, none of the elements contained in disodium-EDTA would provide a spectral interference with any of the inorganic analytes of interest. Therefore, the only mechanism for interference by EDTA is matrix interference, i.e., the absorption of source x-rays and the absorption of emitted fluorescent x-rays from the sample. Above were reported results of experiments investigating the effect of iron in the soil samples on the results of copper, which was the analyte of interest most likely to be interfered with by iron. In fact, iron would provide the highest matrix interference for copper of any element, except cobalt. The experimental results indicated a change in the copper results in spiked samples of 12%. Since iron has a K absorption edge at 7.11 keV, and copper has a K-alpha fluorescence emission x-ray of energy 8.05 keV, and the

concentration of iron in RMA soil averages near 2%, this would probably be considered a large matrix interference. In the case of EDTA, the coefficient of absorption at 8.05 keV is much lower than iron, since the K absorption edge of sodium is 1.08 keV, which is vastly different in energy than the K absorption edge of iron. The concentration of EDTA used in the water standards was 1.9%. Therefore, we concluded that the matrix interference of EDTA in the water standards would be much lower than the effect of iron on copper in RMA soil samples and thus did not merit experimental pretesting. However, to be consistent, the EDTA was added to the surrogate water samples. We speculate that the EDTA complexes with the elements, and prevents their volatilization from the sample. EDTA was not added to the field water sample because it appeared as though the high level of contamination in that sample would require the addition of inordinantly large amounts of EDTA.

For the surrogate soil samples, there was reasonable agreement between the laboratory based analytical results and the spike levels. The one exception to this is mercury, where the observed level was less than half that of the spike. Mercury is known to be easily reduced to its volatile, elemental form, and although the USATHAMA Reference method specifies no holding time, EPA methods specify 28 days holding time. This data tends to support such a relatively short holding time.

Table 10

Summary of Laboratory-Based Analysis of Surrogate and Actual Field Equivalency Test Samples

Sample	Concentration (μg/g soil or μg/mL water) Mean ± One Standard Deviation			
	Copper	Arsenic	Mercury	Lead
LWEM*	107 ± 7	49.4 ± 2.3	102 ± 11	103 ± 6
1703	0.9 ± 0.3	5025 ± 227	0 ± 0	22.2 ± 1
LSEM*	118 ± 17	78.6 ± 9.6	41.0 ± 7.5	115 ± 17
Silo	19.8 ± 6.0	1811 ± 133	0.1 ± 0.3	83 ± 33
Pit	219 ± 15	3.0 ± 0.2	0 ± 0	223 ± 54
2-18	6.6 ± 0.9	1.2 ± 0.1	0 ± 0	10.0 ± 1.5
2-8	16.9 ± 4.2	2.8 ± 0.4	0.5 ± 0.5	773 ± 136

* Denotes surrogate samples generated by spiking RMA soil (LSEM) or ORNL groundwater (LWEM) with target elements.

The results of the statistical evaluation of the laboratory/field comparison are summarized below. The sample location and measured elements of the samples to be compared are given as follows:

Sample Designation	Evaluated
LWEM	As, Cu, Pb, Hg
1703	As
LSEM	As
SILO	As
2-8	Pb, Hg

Other sample/element combinations were not evaluated, either because the XRF system did not pass USATHAMA Class I certification for that element, or that the reported level was below the certified reporting limit.

The Test Method Equivalency Petition examines the measurements for each sample and each element using the single-site comparative case by the following steps: The single site test was performed because XRF performance is related to soil composition, which is dependent on the site location. A comparative, rather than absolute, test was performed, since the XRF system was not expected to perform better than the laboratory based method.

1. Screening for Outliers: Measurements are considered outliers if the values falls outside 4 standard deviations of the grand average. The petition recommends replacing these values with representative values to preserve balance. This replacement makes the calculations simpler but is not necessary for the Analysis of Variance tables (ANOVA). Therefore, rejected outliers were not replaced in the data set. In addition to this test, the Shaprio-Wilks test was examined to check the assumption that the data have an approximately normal distribution.

2. Equality of Replicate Variance: An assumption for the analysis of variance to test bias is that the variance of replicated measurements is constant at all concentration levels. To test this assumption, the standard deviations (e.g. S) and averages (e.g. M) are calculated for the logarithm of the concentrations for each day and each method. The replicate values on each day are used to calculate the daily standard deviations and averages. The method of least-squares is used fit the line $\log(S_{ij}) = a \log(M_{ij}) + b$, where "a" and "b" are the estimated slope and intercept for the i-th method and j-th day. If the slope is significantly different than zero (H_o: $a = 0$), the variances are considered to depend on the concentration level and some data transformation should be employed. The recommended transformation is $[\log(Y)]^{1-a}$ for slope "a" and concentration value "Y".

3. Variance Ratio - 95% Confidence Interval: The laboratory determinations of the contaminant levels in the samples using the ICP or AA are assumed to be "correct".

The precision of the proposed method is compared with the correct or reference method by a 95% confidence interval on the ratio of their variances. If the confidence interval does not include 1, the two methods have different precisions and the proposed method fails. Note that this means that the proposed method fails even if it has a smaller variance than the variance of the reference method.

4. Bias Test by Analysis of Variance: A two-way ANOVA table was used to compare bias of the proposed method and the reference method. The sources of variation tested at the 5% significant level are METHOD, DAY, METHOD x DAY, and ERROR. A significant METHOD X DAY interaction indicates that the differences between the two methods are not the same for all days and the proposed method is not acceptable. If there are no significant sources of interaction, the main effects for METHOD are tested for equivalence. In other words, the mean results are compared for the proposed and reference method. If the mean value for the proposed method is significantly different from that of the reference method, then the proposed method fails equivalency.

The single-site comparative equivalency test is designed to test if a proposed analytical method is the same as the accepted analytical method. The proposed analytical method will fail if it's precision and accuracy are either better or worse than the precision and accuracy of the accepted method. The precisions of the two methods are equivalent if the 95% confidence interval on the ratio of the variances includes 1. The accuracy of two methods are compared by the bias test with analysis of variance. This bias test can fail if either the METHOD X DAY interaction effects are significant or the METHOD effect is significant. The equivalency petition requires that the precision and accuracy test be done in the sequence (1) test for precision, (2) test for METHOD X DAY interaction effects, and (3) test for METHOD effect. If any part of the sequence fails, the equivalency test is terminated and the proposed analytical method does not pass equivalency. A summary table of the results of the precision and accuracy test are given in Table 11. For completeness each of the three tests were performed on all of the tested element/sample combinations.

Examination of the data in Table 11 indicates that the XRF system was not equivalent to the laboratory-based analytical systems for any of the element/sample combinations surveyed for this study. Analysis of the sum of the data here suggests a number of factors which contribute to this non-equivalency. First, the CSI X-Met 840 is designed to be a field portable instrument. To accomplish this objective, the system uses a lightweight, low power consumption gas proportional counter as a detector, and 256-channel energy analyzer. Such a combination provides for relatively modest resolution, requiring complex software to compensate. A low resolution system is inherently less precise and accurate than a higher resolution, more specific laboratory based system. Another factor which acts to reduce precision is operation under field conditions. The instrument and samples are subject to temperature extremes and changes in humidity. The data presented in this report (e.g., Figure 8-11) show clearly that there was a greater variation in responses to calibrated standards under field conditions, compared with that observed in the laboratory. Finally, if operator error can play a part in reproducibility, it seems more likely to occur during the stress of field operations.

Clearly, the X-Met 840 portable XRF system was not designed to perform comparably to laboratory based instruments, and indeed, it does not. Perhaps a more relevant question to ask is the extent to which it can provide useful information concerning levels of contamination under field conditions. Clearly, the X-Met 840 can provide quantitative information concerning the concentration of contaminants. It is not as accurate and precise as a laboratory based system, but there is essentially no sample preparation, and the data is available within four minutes of the start of the analysis. In Table 12 is summarized the performance of the XRF system on the samples acquired at RMA.

Table 11

Summary of Equivalency Petition Test

Sample	Method[a]	Element	Precision	Method X Day	Method
LWEM µg/mL	CSI	As	Failed	Failed	Passed
LWEM µg/mL	USA	As	Failed	Passed	Passed
1703 µg/mL	CSI	As	Failed	Failed	Failed
1703 µg/mL	USA	As	Failed	Failed	Passed
LSEM µg/g	CSI	As	Passed	Passed	Failed
LSEM µg/g	USA	As	Failed	Passed	Failed
SILO µg/g	CSI	As	Passed	Passed	Failed
SILO µg/g	USA	As	Passed	Failed	Failed
LWEM µg/mL	CSI	Cu	Failed	Failed	Failed
LWEM µg/mL	USA	Cu	Failed	Failed	Failed
LWEM µg/mL	CSI	Pb	Failed	Failed	Passed
LWEM µg/mL	USA	Pb	Failed	Passed	Passed
2-8 µg/g	CSI	Pb	Failed	Passed	Passed
2-8 µg/g	USA	Pb	Failed	Passed	Failed
LWEM µg/mL	CSI	Hg	Passed	Failed	Failed
LWEM µg/mL	USA	Hg	Passed	Failed	Failed

[a]CSI refers to the manufacturer's procedure for multivariate calibration. USA refers to the USATHAMA single variate approach to calibration.

A more qualitative system has been used, which essentially scores the XRF system as correct if the response is within a factor of two of the laboratory result, or when the XRF and the lab method response are both below the certified reporting limit. For the 20 element/sample combinations, the multi-variate calibration method was correct in 85% of the cases, exhibiting two false positive responses, and one false negative. The single variate method was correct in 75% of the cases, showing five false positive responses. This high degree of correct responses, coupled with the low number of false negative responses, suggests that the XRF system should be a good, semi-quantitative analytical system for screening soil and water samples.

Table 12

Comparison of XRF and Laboratory Analysis of Field Samples
Qualitative Scoring System[b]

Sample Designation	Calibration Method	Copper[a]	Arsenic	Mercury	Lead
Soil					
Silo	Multivariate	Correct	Correct	Correct	Correct
	Single Variate	Correct	Correct	Correct	False Positive
Pit	Multivariate	Correct	Correct	Correct	False Negative
	Single Variate	Correct	Correct	Correct	Correct
2-18	Multivariate	Correct	Correct	Correct	Correct
	Single Variate	Correct	Correct	Correct	Correct
2-8	Multivariate	False Positive	Correct	False Positive	Correct
	Single Variate	False Positive	Correct	False Positive	Correct
Water					
1703	Multivariate	Correct	Correct	Correct	Correct
	Single Variate	False Positive	Correct	Correct	False Positive

[a] The XRF system did not pass certification for copper in soil under field conditions. However, response appears linear above 250 ppm. For the purposes of this table, a surrogate CRL of 250 ppm was assumed.

[b] A "correct" response was scored if the XRF reported value was within a factor of two of the laboratory result, or if the XRF showed the value to be less than the CRL for the element, and the laboratory analysis showed this to be the case also. A false positive response was scored if the XRF response was greater than twice that of the laboratory result, or if the XRF showed the element to be above the CRL, while the laboratory result was below the CRL. A false negative was scored if the XRF result was half that, or less, of the laboratory result, or if the XRF showed the concentration to be less than the CRL, while the laboratory result was greater than the XRF CRL.

IV.C. Recommendations for Use and Further Work

Results from this study have indicated that the CSI X-Met 840 system, in the configuration evaluated here, with the laboratory probe and a laptop personal computer for data acquisition and management, can be a powerful, semi-quantitative tool for screening contaminated soil and water samples. However, it appears that the use of the contact probe could make the system even more versatile, since it can be used to analyze any surface in the field, including samples packaged in the plastic cups normally used with the laboratory probe. The laboratory probe is, however, safer to operate since exposure to direct radiation from the source is not possible, whereas it is possible with the contact probe. The contact probe loaded with 100 mCi of ^{244}Cm that was purchased with the present instrument produced a dose rate at the probe window with the shutter open of 40 mR/hr; the dose rate with the shutter closed was 1.0 mR/hr.

Observations made in the laboratory and field suggest that the practical use of the instrument would involve standardizing the system under laboratory conditions and using the system in the field. We would recommend that a few check standards be analyzed on a daily basis. The time involved is minimal, and response to the standards can be used to determine if excursions in performance are occurring as a result of field operations. In order to achieve the most quantitative results, the importance of developing calibration models which reflect as closely as possible the situation and/or sample to be encountered in the field cannot be overemphasized. For example, if either the arsenic or lead is expected to be present in environmental samples without the presence of the other, the recommended approach would be to construct a multi-variate regression model which is calibrated for either lead-only or arsenic only, plus other noninterfering elements. Presumably, both calibration models could be stored in the XRF's microprocessor. In the field, an initial analysis would be conducted and the photopeak spectrum could be examined visually to determine the presence of either lead or arsenic. Based on that examination, the analysis would be rerun, using the most appropriate calibration model. The time required for the extra screening analysis and visual examination would be 5 - 6 minutes. This approach was not taken in the laboratory or field studies described here, because the time required for certifying lead without arsenic and arsenic without lead calibration models would have lengthened the field portion of the trip from approximately 2 and 1/2 weeks to a minimum of 6 weeks.

In its current configuration, the X-Met 840 performs very well as a screening system. It is not as precise or accurate as the reference laboratory methods, but it is not designed for that requirement. However, none of the laboratory methods can run 90 samples - processing and analysis - in an 8 hour work shift. However, it does appear that it is possible to markedly improve the accuracy and precision of an XRF system and still maintain true field portability. Such an approach would be based on a radioactive x-ray source, similar to that used in the X-Met 840, and a much higher resolution silicon detector. The potential benefit of a higher resolution system is illustrated in Figure 16. This spectrum of dry RMA, soil spiked with 700 ppm of copper, 350 ppm of arsenic and lead and 3500 ppm of mercury, was acquired with a portable x-ray fluorescence system, Model SEFA-P, manufactured by HNU Systems, Inc., of Newton, Massachusetts. The instrument employs a liquid nitrogen cooled silicon detector, a 4096 channel analyzer, and one of three radioactive sources, ^{55}Fe, ^{109}Cd, and ^{241}Am that exist in separate chambers and are available for use in the field. The spectrum of Fig. 16 was obtained with ^{109}Cd as the exciting source. A spectrum of the same sample acquired with the X-Met 840, using ^{244}Cm as the excitation source, is shown in Figure 17. Several differences between the two spectra point out the advantages of the higher resolution silicon detector. The most obvious benefit of the high resolution is the fact that the person operating the instrument can visually determine with much greater confidence the presence or absence of photopeaks of elements of interests. Although, it appears that copper, at a concentration of 700 ppm, is present in the X-Met spectrum, there is no question about its presence in the HNU spectrum. One should note that there is no indication of two iron x-rays from the X-Met spectrum, whereas both are shown by the HNU spectrum. In the case of the arsenic, mercury, and lead, the Kα x-ray of arsenic and the Lα x-rays of mercury and lead are not resolved. Although the Kα x-ray of arsenic and the Lα x-ray of lead are not resolved with the silicon detector, these two x-rays are well resolved from the mercury Lα x-ray. The Kβ x-ray of arsenic and the Lβ x-rays of mercury and lead are also not resolved by the gas proportional detector of the X-Met, whereas, the lead Lβ is separated from the other two x-rays in the spectrum taken with the silicon detector. Overall visual examination of the spectrum from the gas proportional detector reveals very little information about which elements are present in this particular sample. However, in the spectrum from the silicon detector, it is clear that copper, mercury, and lead are present. The presence of arsenic is not strongly indicated by the silicon detector spectrum. It is true that if the sample being measured is described exactly by the regression model being used by the X-Met, the quantitative results obtain by that instrument will be valid. However, for unknown samples, one never knows to what extent the model describes the unknown. It is for this reason that the higher resolution coupled with visual examination of spectra is vital.

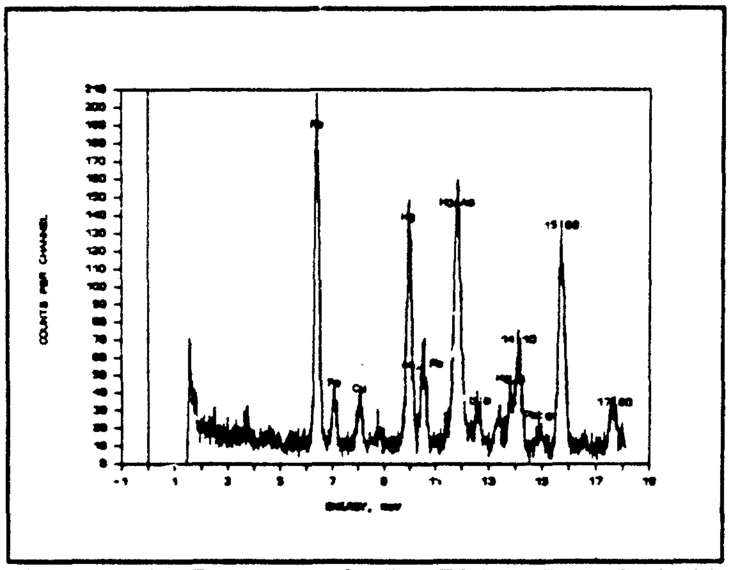

Figure 16. XRF spectrum of spiked RMA soil acquired with Si(Li) detector of HNU Model SEFA-P.

An additional advantage of the silicon detector over the gas proportional detector is the increase of counting efficiency with increasing energy of the fluorescent x-rays. This advantage is shown in Figures 16 and 17 where the $L\beta$ photopeak of mercury actually contains more counts that the $L\alpha$ photopeak even though the yield of the $L\alpha$ is larger. The decreasing counting efficiency with increasing x-ray energy results in the $L\beta$ photopeak having much lower counts than the $L\alpha$. One would expect, therefore, that the detection limits for elements that emit higher energy x-rays would be better with silicon detectors than with gas proportional detectors.

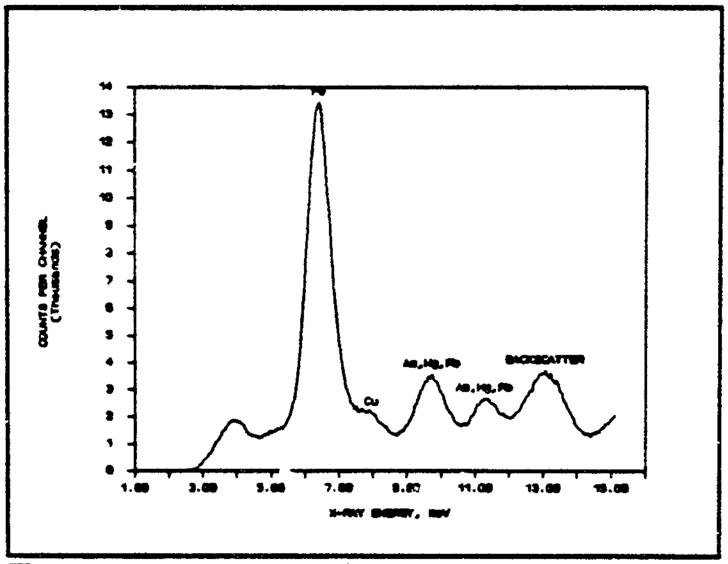

Figure 17. XRF spectrum of spiked RMA soil acquired with the X-Met 840.

The liquid nitrogen dewar has a 24-hour lifetime. Although designed for field use, the system weighs 50 lbs., and thus is not easily carried by one person. Given the current state of technology of laptop personal computers, reducing the weight of a high resolution system by 50% seems easily achieved. A multichannel analyzer card could be installed in a laptop PC, so that all of the data acquisition and manipulation would be performed in a relatively small, lightweight package. Use of a liquid nitrogen cooled Si(Li) detector may appear to limit the use of such a system to sites near urban areas. Of course, many contaminated sites are in fact in or near urban areas. However, a 6 liter dewar, holding sufficient liquid nitrogen for a 5-day work week, can easily be carried in a automobile. If such a system were developed, it should be much more accurate and precise than currently available portable XRF systems, and would maintain true single person portability. It would seem possibility that such a system could actually replace laboratory methods in some circumstances.

REFERENCES

"Operating Instructions: X-Met 840 Portable XRF Analyzer P/N 708-9001, Columbia Sceintific Industries Corporation, Austin, TX, 1988.

S. Piorek and J. R. Rhodes, "A New Calibration Technique for X-Ray Analyzers Used in Hazardous Waste Screening," Proceedings of the Fifth National Conference on Hazardous Wastes and Hazardous Materials, Las Vegas, NV, June 19-21, 1988, pp. 428-433.

United States Environmental Protection Agency, Test Method Equivalency Petitions - A Guidance Manual, Office of Solid Waste, Washington, D.C., EPA/530-SW-87-008, OSWER Policy Directive No. 9433.00-2, February, 1987.

Vic Barnett and Toby Lewis, Outliers in Statistical Data, 2nd edition, John Wiley & Sons, New York, 1984.

APPENDIX A

Water and Soil Standards Used to Calibrate X-Met 840 X-Ray Fluorescence System

METHODS OF PREPARATION

Samples were prepared by spiking soil and water samples with solutions containing individual elements of interest. Although a set of dry soil standards was prepared, they were not used due to the fact that during the winter when the field study was carried out, the soil at RMA is normally damp in most areas. Wet soil samples were prepared by spiking 10 gram soil samples with aliquots of aqueous solutions, drying the samples under a heat lamp, allowing them to equilibrate in air for several hours, mixing the soil particles well and then adding the required amount of water to have all the standards contain the required amount of moisture. Aqueous standard samples were prepared by adding the required aliquot sizes from the stock solutions described below and then adding a computed quantity of water to yield a 20 ml sample with the required analyte concentrations.

Soil standards used to calibrate the X-Met were prepared with analyte concentrations that ranged from 35 to 3500 micrograms per gram of soil (ppm). Water standards had analytes that ranged from 7 to 3500 ppm. Because of the large width of these ranges of concentrations, it was necessary to prepare several stock solutions for each analyte to avoid adding either too little or too much of an aliquot for a spike. A spike that was too small could not be accurately measured, or in the case of a soil sample, would not contact a sufficiently large fraction of the soil particles. A spike that was too large would, in the case of soil, wet the soil too much, or in the case of water cause the final volume of spiked sample to exceed the desired volume. A master stock solution of copper was prepared by dissolving enough reagent grade copper nitrate in a volume of water that was slightly less than 100 ml so that when the solid had disslovedd, and enough water was added to yield exactly 100 ml, the concentration of copper was 35000 micrograms per ml. Master stock solutions of arsenic, mercury, and lead were prepared in a similar manner to yield 35000 micrograms per ml. Table A1 lists the reagent grade compounds and their weights that were used to prepare the stock solutions to give 35000 micrograms of metal ion per ml. Stock solutions containing 3500 micrograms of the analytes per ml were prepared by taking 10 ml of the master stock solutions and diluting to 100 ml. Stock solutions containg 350 micrograms per ml were prepared in the same manner from the second set of stock solutions.

To prepare a complete set of standards, e.g. a set of 20 samples of soils, a concentration scale was established so that each sample represented a multiple of the TRL. As indicated above, 35 ppm was taken for the TRL for soil samples. The concentration scale was selected to cover the range from blanks, to 100 times the TRL. The soil samples were spiked in a random fashion, so that there would be no correlation between the concentrations of any two elements within the suite of standards.

Compound	Molecular Weight	Weight (g) to Yield 35000 μg/ml in 100 mL
$Cu(NO_3)_2 \cdot 2.5H_2O$	232.59	12.810
As_2O	197.84	4.621
$Hg(NO_3)_2 \cdot H_2O$	342.62	5.978
$Pb(NO_3)_2$	331.21	5.594

15-Nov-88

PREPARATION OF CSI WATER CALIBRATION SAMPLES (CONCS RANDOMLY SELECTED)

TRL_Cu = 35
TRL_As = 35
TRL_Hg = 35
TRL_Pb = 35

PRESCRIBED LEVELS RANDOMLY SELECTED

SAMPLE	COPPER CONC. IN WATER (ppm)	ARSENIC CONC. IN WATER (ppm)	MERCURY CONC. IN WATER (ppm)	LEAD CONC. IN WATER (ppm)
1	0	1750	70	175
2	7	13.1	35	1400
3	0	0	0	0
4	13.1	1400	13.1	28
5	13.1	140	17.5	0
6	17.5	7	350	13.1
7	28	70	7	350
8	28	7	0	70
9	70	350	70	3500
10	140	2800	28	280
11	280	35	28	350
12	350	175	35	35
13	700	0	350	17.5
14	700	0	17.5	700
15	1400	7	140	2800
16	1750	35	280	140
17	1750	1750	13.1	1400
18	2800	17.5	0	17.5
19	3500	28	700	7
20	1000	1000	1000	1000
21	1000	1000	1000	1000

15-Nov-88

PREPARATION OF STANDARD SOIL SAMPLES

TRL_Cu	35
TRL_As	35
TRL_Hg	35
TRL_Pb	35

WET SOIL MODEL — PRESCRIBED LEVELS RANDOMLY SELECTED

SAMPLE	MULTIPLES OF TRL (35ppm)	TOTAL MICROGRAMS (PER 10 g SOIL	COPPER CONC. IN SOIL (ppm)	ARSENIC CONC. IN SOIL (ppm)	MERCURY CONC. IN SOIL (ppm)	LEAD CONC. IN SOIL (ppm)
BLANK	0	0	0	0	0	0
1	0	0	0.00	70	350	140
2	1	350	35.00	700	35	700
3	1	350	35.00	1400	350	210
4	2	700	70.00	700	2100	280
5	2	700	70.00	210	280	35
6	4	1400	140.00	2100	140	350
7	4	1400	140.00	140	700	35
8	6	2100	210.00	350	210	140
9	8	2800	280.00	280	1400	280
10	8	2800	280.00	35	140	3500
11	10	3500	350.00	70	1400	2100
12	10	3500	350.00	280	700	1400
13	20	7000	700.00	0	280	70
14	20	7000	700.00	350	3500	350
15	40	14000	1400.00	1400	0	700
16	40	14000	1400.00	35	35	1400
17	60	21000	2100.00	3500	70	70
18	100	35000	3500.00	140	70	0

APPENDIX B

Operating Instructions and Lotus Symphony Macro Commands for Operating the CSI X-Met 840

OPERATING INSTRUCTIONS FOR X-MET 840 AS OPERATED WITH TOSHIBA T1000 LAPTOP COMPUTER

The Toshiba T1000 Computer

The Toshiba T1000 laptop computer is a 6.4 pound instrument with a LCD screen that will display 25 lines of 80 characters. The computer comes standard with 512 kilobytes of memory, and has a 80C88 microprocessor that operates at 4.77 MHz. The T1000 has 256 Kb of read only memory (ROM) that contains MS-DOS 2.11 as the operating system. Unless otherwise instructed, the computer will boot from DOS contained in ROM. Other versions of DOS can be loaded in the floppy diskette drive. An optional memory card with 768 Kb of expanded memory RAM is available. The RAM on the card is supplied with battery power even when the instrument is turned off. A file named SETUP10 is supplied with the software to allow the RAM on the card to be partitioned into conventional memory, expanded memory, or RAM disk memory. A memory expansion card was used in this work and 128 Kb of the memory was used as conventional memory to yield a total of 640 Kb of conventional memory. The remaining 640 Kb of the cards memory was used as a RAM disk. The RAM disk is designated drive "D:", and is formatted as if it were a diskette. Many of the files of SYMPHONY, an integrated program of LOTUS, and SUPERKEY, a keyboard manager program of Borland International, along with several additional files needed to operate the X-Met were stored on the RAM disk. Setup of the T1000 with the SETUP10 file permits options on several additional features, one of which is to configure the keyboard as an 84 or 101 key keyboard. It was found that certain incompatabilities existed with SUPERKEY when the 101 key system was adopted; these problems were eleminated when the 84 key system was chosen. The Setup10 file allows the use of a CONFIG.SYS and an AUTOEXEC.BAT file on drive D: even though the computer boots from the ROM drive. The CONFIG.SYS file makes it possible to have the DOS prompt as the current subdirectory. The AUTOEXEC.BAT file was used, as indicated below, to print instructions on the screen for use of the XRF system and to load SYMPHONY AND SUPERKEY. The reader is directed to the operating manual of the computer for additional information.

Overview of Operation of X-Met with T1000

As explained in the body of this report, the X-Met 840 can be operated from the communications environment (COMM Mode) of SYMPHONY running on the T1000. In the COMM Mode one can issue all of the commands to the X-Met that are available to it from its own keyboard. A list of the X-Met commands are given below along with a description of their function and a reference to the X-Met operating manual supplied by the vendor where the command is discussed. A command is issued simply by typing it on the computer keyboard while SYMPHONY is in the communications environment. Other communications software would also suffice, and one could, with some effort, write a communication program tailored specifically to operate the X-Met. Such a program is being written in Turbo C, a Borland version of the C language, by M. S. Blair of the Instruments and Controls of ORNL for a study directed by J. E. Nyquist of the Health and Safety Division of ORNL. (Personal communication with J. Nyquist, Oak Ridge National Laboratory.)

There are two advantages of SYMPHONY over certain other communication programs in the operation of the X-MET. First, it captures information in the worksheet, rather than on disk, and thus does not frequently use battery power to operate the diskette drive to store information. Second, data is almost immediately available for analysis by all of the mathematical spreadsheet features of SYMPHONY. Note however, that because information is captured as strings (text), numerical information must first be converted to numerical data before it can be manipulated mathematically. SYMPHONY provides a function for this conversion. Most other communication programs will capture to a ram drive, and thus will capture in memory. However getting the data in a form for analysis may be somewhat more complicated, and the data would still have to be imported into a program for analysis. It should be noted that although the Toshiba T1000 is capable of running SYMPHONY and permiting some calculations to be made and other features such as display of X-Met spectra with the SYMPHONY file PRINTGRAPH.COM, the T1000 is too slow to carry out extensive data analysis with SYMPHONY. This slowness is due both to the computer's microprocessor and to the great demands made on the system in running a spreadsheet program. Thus to process large amounts of data with SYMPHONY, it is more convenient to use a more powerful personal computer than the T1000.

In addition to being able to issue the normal X-Met commands from the computer's keyboard, one can make use of keyboard macro routines of SYMPHONY and SUPERKEY to set up the worksheet correctly and ensure that the desired data is collected in the worksheet correctly and expeditiously. SUPERKEY was used in this work to automate data collection as explained below in the list of X-MET commands.

The following exposition gives a detailed account of the use of the X-Met with the Toshiba T1000 laptop computer and describes the functions of the various MACROS and features of SYMPHONY that contributes to the automatic operation of the system.

Initial Software Installation

SYMPHONY is installed on the D: drive as it is on a fixed disk according to the instructions in the SYMPHONY operations manual. The total SYMPHONY software package is too large to place on the 640 Kb ram drive. SUPERKEY.COM copied to the RAM drive, is all that is necessary to provide the necessary features of SUPERKEY. Files necessary to operate the system are shown in Table B1.

Initial System Operations:

The X-Met and T1000 are connected between their respective RS-232 ports by means of the serial cable supplied with the X-Met. Both instruments are then turned on, at which time the T1000 boots with DOS 2.10 incorporated with read only memory (ROM) of the computer. When the battery charger of the X-Met is connected, and the instrument is not being operated on its internal battery for periods longer than a few hours, it is normally left on and connected to a an AC supply.

The AUTOEXEC.BAT file loads SUPERKEY and SYMPHONY. To communicate with the system, it is necessary to define several communications parameters, such as baud rate, parity, etc. By means of the configuration default settings of SYMPHONY, it is possible to defined communications files in which all the necessary communications parameters are predefined and which can be stored on a disk drive and loaded automatically at the time SYMPHONY is loaded. Such a file, named XMET.CCF, listed in Table B1, was established for the X-Met and stored in the T1000 D drive. Thus each time SYMPHONY was loaded, the XMET.CCF file was also loaded to permit communication with the X-Met. In this work, the T1000 and X-Met were operated at 2400 baud.

Table B1

Files Used on the T1000 Ram Drive to Operate System

SYMPHONY Files

AUTOEXEC.BAT CONFIG1.SYS DOS.APP INPUT.APP LOTUS.SET
MACROMGR.APP STAT.APP SYM.BAT SYMPHONY.CMP SYMPHONY.CNF
SYMPHONY.DYN SYMPHONY.EXE SYMPHONY.HLP UTIL.SET XMET.CCF

SYMPHONY Macro

XMET.MLB

SUPERKEY

KEY.COM

SUPERKEY Macro

XMETONE.MAC

SYMPHONY also permits several other automatic features to be predefined to enhance its utility. One such feature is the automatic loading of application add-in managers. A macro manager supplied by LOTUS as MACROMGR.APP (see Table B1) permits macros to be loaded in memory and executed without being in the worksheet. In this work, SYMPHONY was set to load the macro manager which then prompted the operator to load the file XMET.MLB, which contains all of the SYMPHONY macros to operate the X-Met. A list of the SYMPHONY macros and a description of their function are given below.

Summary of Operating Steps

1. Connect the RS-232 port of the T1000 to the RS-232 port of the X-Met

2. Turn on both X-Met and T1000.

3. T1000 boots DOS 2.10 from ROM, loads SUPERKEY and SYMPHONY, and displays the DOS prompt.

4. Change to the D: drive and type SYM

   Note: The D: drive is a battery supported RAM drive that contains SYMPHONY and all the other files needed for the analysis. SYM is a batch file that loads SYMPHONY. SYMPHONY is set up to automatically load the MACRO MANAGER and a set of macros that will operate the X-MET.

5. Type <ALT> S to set up the work sheet for capturing data from the X-MET. The macro has the functions listed below and will leave SYMPHONY in COMM environment ready to operate X-MET.

LIST OF MACROS

SYMPHONY Macros

The Symphony macros below are used to operate the X-Met 840. The macros have the following function.

Setup Macro

<ALT>S	{GOTO}A1~/FD~~/WS70~@NOW~/RV~~ {DOWN}WORKSHEET TITLE: ~{?}~ {DOWN}{TYPE}C{MENU}SCRA1..A8192~Q {HANDSHAKE "\D","013",1}

The symbol <ALT>S signifies that the macro can be executed by holding down the ALT key and presing the S key. This macro is run from the SHEET ENVIRONMENT of SYMPHONY and should be executed when symphony is loaded. The macro writes the date in the worksheet, sets up a worksheet range for capture in the comm environment, and prompts the operator for a worksheet title and the analysis model for the X-MET.

<ALT>A (ACQUIRE SPECTRUM)	{GOTO}A8192~{END}{UP} {DOWN}SAMPLE ID~{?}~ SAMPLE NUMBER~{?}~ {DOWN}{SWITCH}~

This macro is executed from the SHEET ENVIRONMENT and can be used to move cursor to bottom of worksheet, prompt operator for sample identification and change to COMM ENVIRONMENT. NOTE: <ALT> C is normally used instead of <ALT> A. Usually at this point the operator would press the start key on the X-Met panel or type <CTRL>A to cause the X-Met to acquire a spectrum and print the assay values for the sample. After the assay values appear in the SYMPHONY COMM window, the operator would run the SUPERKEY macro <ALT>T to cause the X-Met to print values of the standard deviations, the gross count rates and net count rates for the photo peaks in the spectrum.

<ALT>C (ACQUIRE SPECTRUM)	{SWITCH}{\A}

This macro is run from COMM ENVIRONMENT. It's only function is to switch from the COMM to the SHEET environment and run the <ALT> A macro.

```
<ALT>P      /IR~{DOWN 257}~/IR
(PLOT       {UP 3}{END}{UP}~{RIGHT 2}
SPECTRUM)   /E{END}{DOWN}{END}{RIGHT}~
            @VALUE{LEFT}{LEFT}~
            /C~{TAB}{LEFT}{END}{DOWN}{RIGHT 2}~
            /RV{END}{DOWN}{RIGHT}~~
            /M{END}{DOWN}{RIGHT}~{LEFT2}~{LEFT2}
            /G1CR~QRX{TAB}{END}{DOWN}~A{RIGHT}
             {TAB}{END}{DOWN}~
            QF~~QSOS20~TXCHANNEL NUM
            BER~YCOUNTS PER CHANNEL~QQPQ
```

This macro plots the spectrum on the screen. It is run from the SHEET ENVIRONMENT. To use, the operator should capture a spectrum in COMM MODE with X-Met command SPL, then change to the SHEET ENVIRONMENT, place Cursor on the first channel number of the spectrum and type <ALT> P. The column widths need to be set up correctly before <ALT> P is used.

SUPERKEY Macros

```
<ALT>T      <CMD>OP070<CMD>STD<ENTER>
            PUL<CMD>P<ENTER><ESC>INT<ENTER>
            P<ENTER><ESC>
```

This macro, which resides in the file XMETONE.MAC, can be executed when SUPERKEY is resident and the ALT key is held while the T key is pressed. The macro is used in the COMM environment of SYMPHONY. Commands to SUPERKEY are enclosed in angle brackets. The command OP070 places a delay in the operation of SUPERKEY to cause the keyboard commands to execute more slowly and enable the X-Met to keep up with the commands as they are issued. Keyboard commands such as STD are typed by SUPERKEY to the COMM environment of SYMPHONY as if the operator were typing the commands. The X-Met responds to the STD command and sends the values of standard deviations of photopeaks in the spectrum to the RS232 interface where it is captured in the SYMPHONY worksheet.

SPECIAL KEYS AND COMMANDS OF THE XMET-840

COMMAND OR KEY	COMMAND DESCRIPTION	OPERATOR'S MANUAL REFERENCE SECTION
START	[CONTROL A ON COMPUTER] START MEASUREMENT	4.1, 4.2.1

NOTE: The key commands that use the control key as a prefix key can be issued directly from the COMM environment of SYMPHONY and by a SUPERKEY macro that types to the COMM environment, but no way was found to use a SYMPHONY macro to issue this command to the COMM environment. This failure prompted us to use SUPERKEY to provide a way to rapidly and unerringly obtain the necessary information from the X-Met. The SUPERKEY macro <ALT>T was especially useful in the field to prevent operator error.

MODEL	[CONTROL D] SELECT MODEL	3.6, 4.2.3, 5.2.4
MTIME	[CONTROL T] SELECT COUNTING TIME	3.7,4.2.2, 5.2.5
RECALC	[CONTROL R] RECALCULATE ASSAY IN SELECTED MODEL CAN CHANGE MODEL AND RECALCULATE.	4.2.4
ON	SWITCH ON	3.3
OFF	SWITCH OFF	3.8
"<"	DELETE KEYBOARD ENTRY	1.3.2
"^"	SCROLL BACKWARD	1.3.2
CONT/YES	ACCEPT, CONTINUE, OR SCROLL FORWARD	1.3.2
END/NO	REJECT, OR TERMINATE, OR AGREE TO NEGATIVE QUESTION	1.3.2
ADD	ADD REFERENCE SAMPLES TO IDENTIFICATION LIBRARY	5.3.2
ASY	ENTER ASSAYS OF CALIBRATION SAMPLES	5.4.3
CAL	MEASURE CALIBRATION SAMPLES	5.4.2
CIN	OUTPUT CALIBRATION SAMPLE INTENSITIES	5.4.5
CPU	NOT APPLICABLE IN CURRENT XMET COMMANDS	
CSI	CONFIGURING THE I/O PORT (WITH THE EMP COMMAND)	7.7, 11.2.5
DEL	DELETE MODEL	5.2.10

DTM	DISPLAY TIME AND DATE	7.3
EMP	ENTER MAINTENANCE PROGRAMS (WITH PRM, TSM, CSI)	5.2.12
INI	INITIALIZE GAIN CONTROL	5.2.2
INT	OUTPUT NET COUNT RATES	4.4.7
LIM	EXAMINE AND EDIT CHANNEL LIMITS	3.6, 5.2.11
LOC	LOCK THE KEYBOARD	7.4
MOD	REGRESSION MODELING	5.4.4
NOR	NORMALIZATION	3.7
PAR	ENTER AND EDIT CALIBRATION COEFFICIENTS	5.4.6
PUL	OUTPUT GROSS COUNT RATES	4.2.6
PUR	INSTRUMENT CALIBRATION WITH PURE ELEMENTS	5.2.6, 5.2.7, 5.2.8
PRM	INSTRUMENT CALIBRATION PARAMETERS (WITH EMP)	5.2.12
REF	REFERENCE SAMPLE EXAMINATION AND EDITING	5.3.3
SPE	OUTPUT SPECTRUM	4.2.9, 5.2.13
SPL	PLOT SPECTRA (USED TO TRANSFER TO TOSHIBA T1000 TO PLOT WITH ALT <P> SYMPHONY MACRO.	4.2.9, 5.2.14
STD	OUTPUT STANDARD DEVIATION (CAN USE SUPERKEY MACRO ALT <T> TO ISSUE THIS COMMAND TO XMET)	4.2.5
STM	SET TIME AND DATE	7.2
TCR	OUTPUT TOTAL COUNT RATE OF ENTIRE SPECTRUM	4.2.8
TSM	FIND PEAK (WITH EMP)	7.5
THD	DISPLAY DEFAULT THRESHOLD VALUES	5.3.4.1
THR	IDENTIFICATION THRESHOLD	5.3.4
UNL	UNLOCK KEYBOARD	7.6

APPENDIX C

Schematic Diagrams

USATHAMA Class 1 Precertification and Certification and Class 2 Certification

CLASS 1 (QUANTITATIVE) - LINEAR AND ZERO INTERCEPT MINIMUM TESTING RANGE

X * TRL (TARGET REPORTING LIMIT) = STANDARD OR SAMPLE CONCENTRATION
( ) = TOTAL NUMBER OF SAMPLES AND/OR STANDARDS TO BE RUN

PRECERTIFICATION
(14)
(SECTION 4.3)

INSTRUMENT
BEGINNING OF DAY
EPA CHECK STANDARD

INSTRUMENT BLANK
INSTRUMENT BLANK
0.375*TRL=____
0.375*TRL=____
1.0* TRL=____
1.0* TRL=____
2.0* TRL=____
2.0* TRL=____
5.0* TRL=____
5.0* TRL=____
12.5* TRL=____

END OF DAY
EPA CHECK STANDARD

INITIAL
CALIBRATION
(9)
(SECTION 6.4)

INSTRUMENT
BEGINNING OF DAY
EPA CHECK STANDARD

INSTRUMENT BLANK
0.375*TRL=____
1.0 * TRL=____
2.0 * TRL=____
5.0 * TRL=____
12.5 * TRL=____

END OF DAY
EPA CHECK STANDARD
12.5 * TRL=____

CERTIFICATION
GENERATED EACH DAY FOR
4 DAYS (39)
(SECTION 4.6)

METHOD
FIRST DAY
INITIAL CALIBRATION
STANDARDS

METHOD BLANK
0.5 * TRL=____
1.0 * TRL=____
2.0 * TRL=____
5.0 * TRL=____
10.0* TRL=____

SECOND-FOURTH DAYS
BEGINNING OF DAY
12.5* TRL=____

METHOD BLANK
0.5 *TRL=____
1.0 *TRL=____
2.0 *TRL=____
5.0 *TRL=____
10.0 *TRL=____

END OF DAY
12.5* TRL=____

SAMPLE ANALYSIS
INITIAL FIELD SAMPLE LOT
(13+)
INITIAL CALIBRATION STANDARDS
MATRIX METHOD BLANK
3 MATRIX SPIKES PER LOT
2*CRL=____
10*CRL=____
10*CRL=____

ADDITIONAL FIELD SAMPLE
LOTS
(6+)
BEGINNING OF DAY
12.5* TRL=____
MATRIX METHOD BLANK
3 MATRIX SPIKES PER LOT
2*TRL=____
10*TRL+____
10*TRL=____

END OF DAY
12.5*TRL=____STD

CLASS 1 (QUANTITATIVE) - LINEAR AND ZERO INTERCEPT

INSTRUCTIONS:

PREPARE TWO MASTER STOCK SOLUTIONS ON SEPARATE OCCASIONS BY DIFFERENT PERSONNEL USING IDENTICAL PROCEDURES, ONE FOR CALIBRATION STANDARDS AND ONE FOR CERTIFICATION SPIKES.

PRECERTIFICATION: (SECTION 4.3)
RUN STANDARDS AS LISTED ABOVE
TABULATE AND GRAPH RESPONSE VERSUS CONCENTRATION
ANALYZE CURVES FOR LACK-OF-FIT AND ZERO INTERCEPT
VERIFY CHECK STANDARD RESULTS FOR ACCEPTABILITY
USE LIMITS DEFINED BY ORIGINATOR

INITIAL CALIBRATION: (SECTION 6.4)
RUN STANDARDS AS LISTED ABOVE
CHECK THAT RESPONSE IS WITHIN 10% FOR INORGANIC AND 25% FOR ORGANIC ANALYSES OF THE MEAN RESPONSE FOR THE SAME CONCENTRATION, AS DETERMINED FROM PRECERTIFICATION AND THE CERTIFICATION'S INITIAL CALIBRATION AFTER SEVEN CALIBRATIONS, RESPONSES MUST AGREE WITHIN 2 STANDARD DEVIATIONS

CERTIFICATION: (SECTION 4.6)
SPIKE SOIL AND WATER AS LISTED ABOVE (SECTION 4.5)
PERFORM SAMPLE PREPARATION
AFTER CALIBRATION STANDARDS ARE ANALYZED, CHECK THAT THE RESPONSE FALLS WITHIN THE REQUIRED % OR 2 STANDARD DEVIATIONS OF THE MEAN RESPONSE
TABULATE FOUND VERSUS TARGET CONCENTRATION
TEST DATA FOR LINEARITY USING THE LACK-OF-FIT AND ZERO INTERCEPT LINES
DETERMINE CONFIDENCE BOUND, CONTRACT REPORTING LIMIT, ACCURACY, STANDARD DEVIATION, IMPRECISION, AND INACCURACY

CLASS 2 (QUALITATIVE - LINEAR AND ZERO INTERCEPT MINIMUM TESTING RANGE

TRL = TARGET REPORTING LIMIT
( ) = TOTAL NUMBER OF SAMPLES AND/OR STANDARDS TO BE RUN

PRECERTIFICATION	INITIAL CALIBRATION	CERTIFICATION
(0)	(6)	(14)
(SECTION 4.3)	(SECTION 4.4)	(SECTION 4.6)
INSTRUMENT	INSTRUMENT	METHOD
NOT REQUIRED	INSTRUMENT BLANK INSTRUMENT BLANK INSTRUMENT BLANK TRL TRL TRL	INITIAL CALIBRATION STANDARDS MATRIX METHOD BLANK MATRIX METHOD BLANK MATRIX METHOD BLANK MATRIX METHOD BLANK TRL TRL TRL TRL

SAMPLE ANALYSIS LOTS	SAMPLES LOTS	ADDITIONAL FIELD INITIA FIELD SAMPLE
(3+) INITIAL CALIBRATION STANDARDS MATRIX METHOD BLANK STANDARD MATRIX SPIKE = CRL		(6+) BEGINNING OF DAY INSTRUMENT BLANK CALIBRATION STD = CRL MATRIX METHOD BLANK STD MATRIX SPIKE = CRL END OF DAY INSTRUMENT BLANK CALIBRATION STD = CRL

INSTRUCTIONS:

PREPARE TWO MASTER STOCK SOLUTIONS ON SEPARATE OCCASIONS BY DIFFERENT PERSONNEL USING IDENTICAL PROCEDURES, ONE FOR CALIBRATION STANDARDS AND ONE FOR CERTIFICATION SPIKES

INITIAL CALIBRATION: (SECTION 4.4)

ALL BLANKS MUST YIELD NEGATIVE RESULTS
ALL SPIKED SAMPLES MUST YIELD POSITIVE RESULTS

CERTIFICATION: (SECTION 4.6)

ANALYZE SAMPLES IN A SINGLE DAY
ANALYZE RESULTS USING RANK SUM TEST
(SUM SHALL NOT EXCEED 26)

APPENDIX D

Water and Soil Standards used for USATHAMA Class 1 Certification
in Laboratory and Field

Preparation of Water Samples for Certification Class 1

TRL_Cu = 8.9
TRL_As = 3.5
TRL_Hg = 5.5
TRL_Pb = 42

Levels of All Analytes Equal in Each Sample

Sample	Multiples of TRL	Copper Conc. (ppm)	Arsenic Conc. (ppm)	Mercury Conc. (ppm)	Lead Conc. (ppm)
AM-1	0.45	4.01	1.58	2.48	18.90
AM-2	0.45	4.01	1.58	2.48	18.90
AM-3	0.5	4.45	1.75	2.75	21.00
AM-4	0.9	8.01	3.15	4.95	37.80
AM-5	0.9	8.01	3.15	4.95	37.80
AM-6	1	8.90	3.50	5.50	42.00
AM-7	1	8.90	3.50	5.50	42.00
AM-8	2	17.80	7.00	11.00	84.00
AM-9	2	17.80	7.00	11.00	84.00
AM-10	4	35.60	14.00	22.00	168.00
AM-11	4	35.60	14.00	22.00	168.00
AM-12	5	44.50	17.50	27.50	210.00
AM-13	5	44.50	17.50	27.50	210.00
AM-14	10	89.00	35.00	55.00	420.00
AM-15	10	89.00	35.00	55.00	420.00
AM-16	11	97.90	38.50	60.50	462.00
AM-17	11	97.90	38.50	60.50	462.00
AM-18	20	178.00	70.00	110.00	840.00
AM-19	20	178.00	70.00	110.00	840.00
AM-20	22	195.80	77.00	121.00	924.00
AM-21	22	195.80	77.00	121.00	924.00

Preparation of Wet Soil Samples for Certification Class 1

TRL_Cu = 25
TRL_As = 12
TRL_Hg = 29
TRL_Pb = 60

Levels of All Analytes Equal in Each Sample

Sample	Multiples of TRL	Copper Conc. (ppm)	Arsenic Conc. (ppm)	Mercury Conc. (ppm)	Lead Conc. (ppm)
SM-1	0.375	9.38	4.50	10.88	22.50
SM-2	0.375	9.38	4.50	10.88	22.50
SM-3	0.5	12.50	6.00	14.50	30.00
SM-4	0.75	18.75	9.00	21.75	45.00
SM-5	0.75	18.75	9.00	21.75	45.00
SM-6	1	25.00	12.00	29.00	60.00
SM-7	1	25.00	12.00	29.00	60.00
SM-8	2	50.00	24.00	58.00	120.00
SM-9	2	50.00	24.00	58.00	120.00
SM-10	4	100.00	48.00	116.00	240.00
SM-11	4	100.00	48.00	116.00	240.00
SM-12	5	125.00	60.00	145.00	300.00
SM-13	5	125.00	60.00	145.00	300.00
SM-14	10	250.00	120.00	290.00	600.00
SM-15	10	250.00	120.00	290.00	600.00
SM-16	12.5	312.50	150.00	362.50	750.00
SM-17	12.5	312.50	150.00	362.50	750.00
SM-18	20	500.00	240.00	580.00	1200.00
SM-19	20	500.00	240.00	580.00	1200.00
SM-20	25	625.00	300.00	725.00	1500.00
SM-21	25	625.00	300.00	725.00	1500.00

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
PRE-CERTIFICATION AND DAY 1 EQUIVALENCY.

	SAMPLE	Sample Description	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	Dry soil check std (not required).	2250.0	2086.0	2656.0	2660.0
2	SCS-M	USATHAMA required check std.	297.9	262.4	289.0	313.7
3	INSTRUMENT BLANK	Required by	908.1	0.0	64.1	1014.0
4	INSTRUMENT BLANK 2	USATHAMA, but	426.8	0.0	70.1	982.2
5	RAJ BLANK	not appropriate.	2821.0	0.0	2128.0	336.0
6	RAJ BLANK 2		18.4	0.0	75.6	0.0
7	WET SOIL BLANK	Quasi instrument blk	17.0	0.0	83.3	0.0
		Samples 8-28 are Pre-certification samples.				
8	SM-1	0.375 X TRL	26.8	0.0	88.5	36.4
9	SM-2	0.375 X TRL	0.0	24.4	88.1	0.0
10	SM-3	0.5 X TRL (not required).	47.3	6.2	53.5	44.2
11	SM-4	0.75 X TRL	35.3	24.0	112.8	0.0
12	SM-5	0.75 X TRL	4.2	11.9	87.2	14.5
13	SM-6	1 X TRL	66.3	14.7	107.2	37.8
14	SM-7	1 X TRL	27.4	5.1	90.0	82.0
15	SM-8	2 X TRL	32.2	58.5	109.8	14.1
16	SM-9	2 X TRL	76.8	61.3	93.0	37.9
17	SM-10	4 X TRL	95.7	108.6	155.9	139.8
18	SM-11	4 X TRL	90.7	79.4	154.2	162.8
19	SM-12	5 X TRL	131.7	53.8	177.7	306.3
20	SM-13	5 X TRL	122.1	81.5	203.7	220.2
21	SM-14	10 X TRL	264.7	148.3	279.6	587.9
22	SM-15	10 X TRL	268.4	141.6	243.1	621.3
23	SM-16	12.5 X TRL	372.1	[illegible]0.0	281.3	842.4
24	SM-17	12.5 X TRL	314.0	163.3	322.5	804.3
25	SM-18	20 X TRL	518.5	238.0	526.2	1240.0
26	SM-19	20 X TRL	511.7	248.3	481.2	1198.0
27	SM-20	25 X TRL	635.9	255.0	603.7	1556.0
28	SM-21	25 X TRL	618.7	313.7	604.2	1504.0
29	INST. BLANK	Same as sample 4.	440.6	0.8	99.7	766.0
30	INST BLANK #2	Same as sample 7.	21.9	3.6	64.2	0.0
31	SM-1	Samples 31-39 are	30.9	36.3	59.3	0.0
32	SM-4	Initial Calibration	35.8	19.9	76.7	18.8
33	SM-6	for Day 1	86.1	0.0	87.7	102.4
34	SM-8	Equivalency.	32.9	41.5	93.0	72.3
35	SM-10		75.1	103.6	155.9	142.1
36	SM-12		132.1	61.8	168.9	294.4
37	SM-14		265.5	145.0	294.5	585.2
38	SM-16		363.9	121.3	346.5	807.2
39	SM-20		631.9	275.3	630.1	1453.0
40	SCS-M	QA check.	317.5	260.7	258.6	355.1
41	SM-20	QA check.	618.2	265.8	624.8	1508.0
42	SOIL BLANK	Matrix method blank.	11.7	8.4	78.1	0.0
43	SM-8	Samples 43-48	60.7	52.4	79.2	77.3
44	SM-10	are Day 1	72.7	84.3	174.0	182.3
45	SM-14	Equivalency curve.	268.6	136.8	274.3	629.4
46	SM-14		278.4	97.8	262.8	704.7
47	SM-18		497.0	261.0	504.6	1212.0
48	SM-18		491.0	207.4	501.1	1295.0
49	LSEM-21	Surrogate	114.9	87.4	153.6	122.5
50	LSEM-22	Equivalency sample.	114.2	106.2	160.4	73.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
INITIAL CALIBRATION AND DAY 2 EQUIVALENCY.

	SAMPLE	Sample Description	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SCS-M	QA check.	330.9	249.2	281.9	349.4
2	SOIL BLANK	Quasi instrument blk	0.0	6.3	57.0	0.0
3	SOIL BLANK REPEAT	Quasi instrument blk	31.7	0.2	72.0	18.1
4	SM-1	Samples 4-12 are	14.5	0.0	81.9	41.7
5	SM-4	Initial Calibration.	46.0	5.0	97.0	31.2
6	SM-6	This set of Initial	87.0	0.0	91.9	97.7
7	SM-8	Calibration runs	56.4	38.2	115.1	80.6
8	SM-10	was not required	104.2	50.1	150.6	278.1
9	SM-12	for certification or	136.0	69.7	167.1	295.6
10	SM-14	equivalency	265.3	107.2	311.1	659.0
11	SM-16	testing.	353.3	165.1	309.9	854.7
12	SM-20		641.8	277.0	662.2	1445.0
13	SCS-M	QA check.	326.9	219.9	270.4	378.9
14	SM-16	QA check.	368.4	117.0	334.4	804.7
15	SM-20	QA check.	627.4	267.2	634.7	1508.0
16	SOIL BLANK	Matrix method blank.	28.1	0.0	86.3	7.4
17	SM-8	Samples 17-22	65.6	44.0	94.2	61.5
18	SM-10	are Day 2	86.4	81.2	182.6	165.9
19	SM-14	Equivalency curve.	268.6	129.1	304.8	634.2
20	SM-14		268.3	119.6	261.8	672.5
21	SM-18		495.1	206.1	531.2	1236.0
22	SM-18		498.4	210.8	534.9	1232.0
23	LSEM-21	Surrogate	101.4	78.5	181.7	117.8
24	LSEM-22	Equivalency sample.	106.0	103.6	199.8	57.8
25	SM-16	QA check.	362.9	140.8	281.7	821.4
26	SM-20	QA check.	627.4	230.9	627.6	1559.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Sample Description	Cu ppm	As ppm	Zn ppm	Pb ppm
1	SCS-N	QA check.	305.4	259.8	286.2	305.1
2	SOIL BLANK	Quasi instrument blk	12.0	0.0	92.2	8.7
3	SM-1	Samples 3-11 are	14.1	17.1	78.0	9.2
4	SJ-4	Initial Calibration	38.4	0.0	65.1	101.8
5	SM-6	samples for Day 1	54.1	6.0	131.5	55.8
6	SM-8	Certification.	49.7	26.7	83.5	135.5
7	SM-10		88.3	74.3	176.9	189.4
8	SM-12		114.5	85.2	178.1	255.7
9	SM-14		265.7	135.2	287.5	626.8
10	SM-16		358.9	151.3	336.0	749.4
11	SM-20		626.4	214.8	638.8	1565.0
12	SCS-N	QA check.	387.0	267.9	277.2	342.8
13	SM-16	QA check.	339.8	140.8	315.4	781.9
14	SM-20	QA check.	619.4	285.1	612.2	1442.0
15	SOIL BLANK	Matrix matched blank.	25.1	0.0	47.0	38.8
16	SM-3	Samples 16-22 are	36.9	32.3	75.0	3.1
17	SM-6	Day 1 Certification	78.9	8.2	96.1	64.6
18	SM-8	samples.	45.6	29.6	61.9	106.6
19	SM-10		102.5	85.3	179.0	162.9
20	SM-12		132.8	62.2	188.2	287.1
21	SM-14		273.6	114.9	256.5	705.9
22	SM-18		496.2	227.3	545.2	1208.0
23	SM-16	QA check.	357.7	111.2	296.9	864.3
24	SM-20	QA check.	654.1	263.4	601.1	1539.0
25	SOIL BLANK	Matrix matched blank.	2.3	0.0	48.3	52.0
26	SM-8	Samples 26-31	48.6	43.0	111.1	78.6
27	SM-10	are Day 3	103.8	69.7	114.5	236.1
28	SM-14	Equivalency curve.	259.4	120.0	284.4	657.7
29	SM-14		280.3	111.6	286.4	657.6
30	SM-18		497.6	235.8	473.2	1241.0
31	SM-18		495.6	241.5	495.3	1187.0
32	LSEM-21	Surrogate	138.4	68.2	141.1	172.5
33	LSEM-22	Equivalency sample.	118.8	92.1	152.4	106.4
34	SM-6		98.8	35.9	81.7	10.4
35	SM-16	QA check.	364.8	137.7	262.7	818.9
36	SM-20	QA check.	632.1	230.8	585.7	1598.0

APPENDIX E

Schematic Diagram of EPA Equivalency Test

a) Absolute

	Spiked Concentration:	
	Low	High
Day 1 (Sample 1)	x x	x x
Day 2 (Sample 2)	x x	x x
Day 3 (Sample 3)	x x	x x
Day 4 (Sample 4)	x x	x x
Day 5 (Sample 5)	x x	x x
Day 6 (Sample 6)	x x	x x
Day 7 (Sample 7)	x x	x x
Day 8 (Sample 8)	x x	x x
Day 9 (Sample 9)	x x	x x
Day 10 (Sample 10)	x x	x x

b) Comparative

	Test Method:	
	Proposed	Approved
Day 1 (Sample 1)	x x	x x
Day 2 (Sample 2)	x x	x x
Day 3 (Sample 3)	x x	x x
Day 4 (Sample 4)	x x	x x
Day 5 (Sample 5)	x x	x x
Day 6 (Sample 6)	x x	x x
Day 7 (Sample 7)	x x	x x
Day 8 (Sample 8)	x x	x x
Day 9 (Sample 9)	x x	x x
Day 10 (Sample 10	x x	x x

Figure E-1. Layout of Experimental Designs for Single Site Case

APPENDIX F

F-Ratio Analyses from USATHAMA Class 1 Certifications

TABLE: PRE-CERTIFICATION DATA FOR RMA PROJECT.
LABORATORY AND FIELD LACK-OF-FIT AND ZERO INTERCEPT.
INSTRUMENT RESPONSES VS. PREPARED CONCENTRATIONS.

SAMPLE		F-Ratios CU	AS	HG	PB
Lab, soil, w/o blank	LOF, Model with Intercept	0.35	1.96	2.57	4.28
	LOF, Model through Origin	0.26	1.46	1.93	3.21
	Zero Intercept	0.00	0.00	0.00	0.00
	Target Reporting Limit	25	12	29	60
Lab, water, w/o blank	LOF, Model with Intercept	1.81	1.32	0.87	2.14
	LOF, Model through Origin	1.36	0.99	0.65	1.61
	Zero Intercept	0.00	0.00	0.00	0.00
	Target Reporting Limit	8.9	7	5.5	86
Field, soil, w/o blank	LOF, Model with Intercept	0.60	0.67	0.66	0.67
	LOF, Model through Origin	0.45	0.51	0.50	0.50
	Zero Intercept	0.00	0.00	0.00	0.00
	Target Reporting Limit	25	12	29	60
Field, water, w/o blank	LOF, Model with Intercept	1.93	0.26	0.93	0.26
	LOF, Model through Origin	1.45	0.19	0.70	0.20
	Zero Intercept	0.00	0.00	0.00	0.00
	Target Reporting Limit	8.9	3.5	5.5	42

Critical 95% F-ratios:
w/o blanks - LOF, Model with Intercept, 5.41.
LOF, Model through Origin, 5.19.
Zero Intercept, 5.32.

TABLE F-2: CERTIFICATION DATA FOR RMA PROJECT.
LABORATORY AND FIELD LACK-OF-FIT AND ZERO INTERCEPT.
MEASURED CONCENTRATIONS VS. PREPARED CONCENTRATIONS.

SAMPLE		F-ratio CU	AS	HG	PB
Lab	Intercept	49.13	*1.88	4.11	7.16
soil	Origin	51 19	*1.53	3.23	6.32
(1 x TRL)	Zero Intercept	6.36	*0.41	*0.37	*1.86
	TRL (ppm)	25	12	29	60
Lab	Intercept	105.77	5.88	*3.10	14.49
soil	Origin	88.63	7.31	*2.32	12.01
(2 x TRL)	Zero Intercept	*2.02	6.40	*0.00	*1.41
	TRL (ppm)	50	24	58	120
Lab	Intercept	*1.23	*1.49	*0.15	*0.83
water	Origin	*3.03	*1.38	*0.61	*1.87
(1 x TRL)	Zero Intercept	8.12	*2.79	*2.34	5.16
	TRL (ppm)	8.9	3.5	5.5	42
Lab	Intercept	*0.11	*1.03	*0.33	*0.14
water	Origin	*0.12	*1.05	*2.12	*0.49
(2 x TRL)	Zero Intercept	*0.17	*1.09	8.46	*1.77
	TRL (ppm)	17.8	7	11	84
Field	Intercept	15.36	*0.66	*2.04	*1.21
soil	Origin	11.60	3.09	*1.77	4.81
(1 x TRL)	Zero Intercept	*0.10	10.99	*0.81	15.05
	TRL (ppm)	25	12	29	60
Field	Intercept	22.19	*2.23	3.34	3.67
soil	Origin	17.49	3.27	*2.61	4.44
(2 x TRL)	Zero Intercept	*0.75	5.30	*0.29	4.65
	TRL (ppm)	50	24	58	120
Field	Intercept	3.41	*0.92	*0.30	*1.10
water	Origin	9.65	*1.29	*1.69	4.69
(1 x TRL)	Zero Intercept	20.23	*2.45	6.64	15.21
	TRL (ppm)	8.9	3.5	5.5	42
Field	Intercept	*2.30	*0.29	*0.07	*0.35
water	Origin	5.36	*2.04	*0.05	5.37
(2 x TRL)	Zero Intercept	11.94	8.25	*0.01	22.94
	TRL (ppm)	17.8	7	11	84

Critical 95% F-ratios:
- F-ratio, Intercept, 3.29
- F-ratio, Origin, 3.06
- F-ratio, Zero Intercept, 4.41

"*" indicates that XRF method passes certification under given conditions

APPENDIX G

Original Data: Laboratory and Field Certification and Equivalency Measurements

TABLE: COMPILATION OF LABORATORY CONCENTRATIONS, SOIL

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, PRE-CERTIFICATION AND DAY 1 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
	SAMPLE	Cu	As	Hg	Pb
1	26	2250.0	2086.0	2456.0	2660.0
2	SCS-M	297.9	262.4	289.0	313.7
3	INSTRUMEN	908.1	0.0	64.1	1014.0
4	INSTRUMEN	426.8	0.0	70.1	982.2
5	RAJ BLANK	2821.0	0.0	2128.0	336.0
6	RAJ BLANK	18.4	0.0	75.6	0.0
7	WET SOIL	17.0	0.0	83.3	0.0
8	SM-1	26.8	0.0	88.5	36.4
9	SM-2	0.0	24.4	88.1	0.0
10	SM-3	47.3	6.2	53.5	44.2
11	SM-4	35.3	24.0	112.8	0.0
12	SM-5	4.2	11.9	87.2	14.5
13	SM-6	66.3	14.7	107.2	37.8
14	SM-7	27.4	5.1	90.0	82.0
15	SM-8	32.2	58.5	109.8	14.1
16	SM-9	76.8	61.3	93.0	37.9
17	SM-10	95.7	108.6	155.9	139.8
18	SM-11	90.7	79.4	154.2	162.8
19	SM-12	131.7	53.8	177.7	306.3
20	SM-13	122.1	61.5	203.7	220.2
21	SM-14	264.7	148.3	279.6	587.9
22	SM-15	268.4	141.6	243.1	621.3
23	SM-16	372.1	130.0	281.3	842.4
24	SM-17	314.0	163.3	322.5	804.3
25	SM-18	518.5	230.0	524.2	1240.0
26	SM-19	511.7	248.3	481.2	1198.0
27	SM-20	635.9	255.0	603.7	1556.0
28	SM-21	618.7	313.7	[illegible]	1504.0
29	INST. BLA	440.6	0.0	99.7	766.0
30	INST BLAN	21.9	3.6	64.2	0.0
31	SM-1	30.9	36.3	59.3	0.0
32	SM-4	35.8	19.9	76.7	18.8
33	SM-6	86.1	0.0	87.7	102.4
34	SM-8	32.9	41.5	93.0	72.3
35	SM-10	75.1	103.6	155.9	142.1
36	SM-12	132.1	61.8	168.9	294.4
37	SM-14	265.5	145.0	294.5	585.2
38	SM-16	363.9	121.3	346.5	807.2
39	SM-20	631.9	275.3	630.1	1453.0
40	SCS-M	317.5	260.7	258.6	355.1
41	SM-20	618.2	265.8	624.8	1508.0
42	SOIL BLAN	11.7	8.4	78.1	0.0
43	SM-8	60.7	52.4	79.2	77.3
44	SM-10	72.7	84.3	174.0	182.3
45	SM-14	268.6	136.8	274.3	629.4
46	SM-14	278.4	97.8	262.8	704.7
47	SM-18	497.0	261.0	504.6	1212.0
48	SM-18	491.0	207.4	501.1	1295.0
49	LSEM-21	114.9	87.4	153.6	122.5
50	LSEM-22	114.2	106.2	160.4	73.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, INITIAL CALIBRATION AND DAY 2 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SCS-M	330.9	249.2	281.9	349.4
2	SOIL BLAN	0.0	6.3	57.0	0.0
3	SOIL BLAN	31.7	0.2	72.0	18.1
4	SM-1	14.5	0.0	81.9	41.7
5	SM-4	46.0	5.0	97.0	31.2
6	SM-6	87.0	0.0	91.9	97.7
7	SM-8	56.4	38.2	115.1	80.6
8	SM-10	104.2	50.1	150.6	278.1
9	SM-12	136.0	69.7	167.1	295.6
10	SM-14	265.3	107.2	311.1	659.0
11	SM-16	353.3	105.1	309.9	854.7
12	SM-20	641.8	277.0	662.2	1445.0
13	SCS-M	326.9	219.9	270.4	378.9
14	SM-16	368.4	117.0	334.4	804.7
15	SM-20	627.4	247.2	634.7	1508.0
16	SOIL BLAN	28.1	0.0	86.3	7.4
17	SM-8	65.6	44.0	94.2	61.5
18	SM-10	86.4	81.2	132.6	165.9
19	SM-14	268.6	129.1	304.8	634.2
20	SM-14	268.3	119.6	261.8	672.5
21	SM-18	495.1	206.1	531.2	1236.0
22	SM-18	498.4	210.8	534.9	1232.0
23	LSEM-21	101.4	78.5	181.7	117.8
24	LSEM-22	106.0	103.6	199.8	57.8
25	SM-16	362.9	140.8	281.7	821.4
26	SM-20	627.4	230.9	627.6	1559.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SCS-M	305.4	259.8	286.2	305.1
2	SOIL BLAN	12.0	0.0	92.2	8.7
3	SM-1	14.1	17.1	78.0	9.2
4	SM-4	38.4	0.0	65.1	101.8
5	SM-6	54.1	6.0	131.5	55.8
6	SM-8	49.7	24.7	83.5	135.5
7	SM-10	88.3	74.3	176.9	189.4
8	SM-12	114.5	85.2	178.1	255.7
9	SM-14	260.7	135.2	287.5	624.8
10	SM-16	358.9	151.3	336.0	749.4
11	SM-20	624.4	214.8	630.8	1565.0
12	SCS-M	307.0	247.9	277.2	362.8
13	SM-16	339.8	140.8	315.4	781.9
14	SM-20	619.4	285.1	612.2	1442.0
15	SOIL BLAN	25.1	0.0	47.0	38.8
16	SM-3	36.9	32.3	75.0	3.1
17	SM-6	70.9	8.2	96.1	64.6
18	SM-8	45.6	29.6	61.9	104.6
19	SM-10	102.5	85.3	179.0	162.9
20	SM-12	132.8	62.2	188.2	287.1
21	SM-14	273.6	114.9	256.5	705.9
22	SM-18	494.2	227.3	545.2	1208.0
23	SM-16	357.7	111.2	296.9	844.3
24	SM-20	654.1	243.4	601.1	1539.0
25	SOIL BLAN	2.3	0.0	48.3	52.0
26	SM-8	48.6	43.0	111.1	78.6
27	SM-10	103.8	69.7	114.5	236.1
28	SM-14	259.4	120.0	284.4	657.7
29	SM-14	289.3	111.6	286.4	657.6
30	SM-18	497.6	235.8	473.2	1241.0
31	SM-18	493.6	241.5	495.3	1187.0
32	LSEM-21	130.4	68.2	141.1	172.5
33	LSEM-22	118.8	92.1	152.4	104.4
34	SM-6	90.8	35.9	81.7	10.4
35	SM-16	364.8	137.7	262.7	818.9
36	SM-20	632.1	230.8	585.7	1598.0

TITLE: X-MET 840 LABORATORY CONCENTRATION, WET SOIL, DAY 2 CERTIFICATION AND DAY 4 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	335.6	126.0	361.1	768.4
2	SM-20	641.3	242.2	596.1	1550.0
3	SM-3	56.4	0.0	83.6	64.7
4	SM-6	73.8	11.3	117.6	36.6
5	SM-8	35.1	44.1	81.0	93.1
6	SM-10	94.5	57.3	169.0	236.1
7	SM-12	106.2	62.0	173.2	284.9
8	SM-14	275.0	103.7	303.9	694.9
9	SM-18	494.3	181.7	494.0	1300.0
10	SOIL BLAN	8.8	7.8	73.7	0.0
11	SM-8	62.7	3.6	99.0	159.2
12	SM-10	77.4	82.3	180.0	175.5
13	SM-14	259.3	145.3	317.7	590.3
14	SM-16	275.9	131.5	293.4	627.1
15	SM-18	491.0	199.2	548.3	1231.0
16	SM-18	515.6	226.2	463.2	1274.0
17	LSEM-21	123.4	91.9	138.1	135.6
18	LSEM-22	126.2	105.6	154.8	92.2
19	SM-16	354.8	123.1	308.9	796.4
20	SM-20	618.1	224.4	643.3	1537.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL,
DAY 3 CERTIFICATION AND DAY 5 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	357.6	104.1	282.2	894.2
2	SM-20	639.5	272.6	577.6	1512.0
3	SM-3	58.4	7.0	117.4	0.0
4	SM-6	85.4	10.6	108.0	73.1
5	SM-8	38.7	31.0	99.6	104.7
6	SM-10	98.5	73.8	194.1	188.8
7	SM-12	132.4	54.7	153.9	325.5
8	SM-14	279.7	76.9	319.4	711.3
9	SM-18	504.6	203.0	467.1	1291.0
10	SOIL BLAN	28.8	0.0	86.7	9.8
11	SM-8	58.1	42.8	127.6	47.8
12	SM-10	100.3	56.2	182.8	200.1
13	SM-14	270.3	135.4	265.9	629.8
14	SM-14	284.2	121.1	298.6	636.3
15	SM-18	496.6	227.5	409.1	1298.0
16	SM-18	498.2	235.1	486.6	1217.0
17	LSEM-21	126.5	79.6	175.1	106.4
18	LSEM-22	135.4	103.7	170.6	91.4
19	SM-16	370.8	118.6	268.5	855.2
20	SM-20	616.7	245.0	615.3	1520.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL,
DAY 4 CERTIFICATION AND DAY 6 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	355.0	109.2	262.3	886.6
2	SM-20	645.4	254.6	570.4	1522.0
3	SM-3	45.7	0.0	116.2	59.3
4	SM-6	73.5	0.0	75.2	128.8
5	SM-8	31.8	31.5	77.8	97.7
6	SM-10	96.0	82.0	161.9	188.8
7	SM-12	116.6	85.7	175.7	277.5
8	SM-14	268.8	117.7	252.4	686.1
9	SM-18	488.7	205.3	522.8	1256.0
10	SOIL BLAN	13.9	0.0	95.8	0.0
11	SM-8	70.9	52.6	74.4	81.1
12	SM-10	90.2	91.8	132.7	183.0
13	SM-14	259.1	122.2	322.1	664.1
14	SM-14	278.0	122.8	283.5	663.5
15	SM-18	409.1	217.9	497.4	1255.0
16	SM-18	503.3	222.1	408.4	1324.0
17	LSEM-21	111.1	80.7	117.2	159.2
18	LSEM-22	136.0	119.2	148.7	80.2
19	SM-16	358.5	133.6	304.4	787.3
20	SM-20	621.0	234.1	624.5	1560.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, DAY 7 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	359.7	116.8	300.9	822.3
2	SM-20	646.5	260.9	621.6	1481.0
3	SOIL BLAN	10.3	0.0	116.8	0.0
4	SM-8	50.1	27.5	85.1	122.4
5	SM-10	108.1	63.4	182.7	196.3
6	SM-14	276.0	110.5	277.1	688.8
7	SM-14	275.3	116.9	294.2	645.5
8	SM-18	485.8	238.3	419.0	1270.0
9	SM-18	515.5	251.4	438.6	1229.0
10	LSEM-21	125.4	108.2	137.3	91.4
11	LSEM-22	126.[illegible]	136.6	114.0	80.0
12	SM-16	364.9	148.3	277.2	794.9
13	SM-20	648.6	251.2	569.4	1509.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, DAY 3 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	358.6	104.3	289.7	851.0
2	SM-20	658.7	252.0	587.6	1525.0
3	SOIL BLAN	14.8	0.0	100.4	0.0
4	SM-8	60.1	41.8	89.4	80.4
5	SM-10	101.7	70.5	164.1	206.0
6	SM-14	276.7	116.1	281.5	666.0
7	SM-14	262.7	128.9	331.7	595.0
8	SM-18	483.9	222.3	469.0	1254.0
9	SM-18	504.4	262.3	426.4	1249.0
10	LSEM-21	106.[illegible]	94.1	162.3	98.2
11	LSEM-22	123.4	131.2	183.6	14.5
12	SM-16	358.0	138.5	331.5	785.2
13	SM-20	642.4	261.4	581.4	1510.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, DAY 9 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	361.9	126.5	330.9	791.6
2	SM-20	630.5	222.7	603.5	1558.0
3	SOIL BLAN	21.4	0.0	84.8	40.5
4	SM-8	51.6	13.8	87.0	135.3
5	SM-10	105.7	78.8	157.4	196.9
6	SM-14	273.7	116.5	299.4	634.2
7	SM-14	283.6	111.0	298.4	673.6
8	SM-18	529.2	226.8	455.7	1242.0
9	SM-18	508.4	222.4	456.3	1270.0
10	LSEM-21	145.5	93.8	159.4	103.5
11	LSEM-22	123.0	97.5	171.2	92.2
12	SM-16	353.7	137.5	281.1	821.7
13	SM-20	652.8	236.7	634.1	1469.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, DAY 10 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	329.2	91.1	334.1	860.5
2	SM-20	613.7	196.5	640.9	1546.0
3	SOIL BLAN	20.8	0.0	57.5	58.2
4	SM-8	44.5	25.2	107.3	111.8
5	SM-10	93.3	54.1	156.1	257.9
6	SM-14	265.5	100.8	330.8	647.8
7	SM-14	264.8	107.2	274.0	681.3
8	SM-18	505.5	181.8	485.0	1311.0
9	SM-18	489.1	209.0	466.9	1259.0
10	LSEM-21	129.5	51.0	150.4	189.0
11	LSEM-22	123.2	82.8	139.8	152.8
12	SM-16	341.3	93.0	346.7	863.4
13	SM-20	648.0	186.5	625.4	1584.0

TABLE: COMPILATION OF LABORATORY CONCENTRATIONS, WATE

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
PRE-CERTIFICATION AND DAY 1 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	ACS-M	88.3	122.4	77.3	67.0
2	BACKSCATT	471.9	0.0	195.2	41.4
3	BACKSCATT	465.4	0.0	189.4	34.7
4	WATER BLA	0.0	1.9	0.0	0.0
5	WATER BLA	0.0	12.0	0.0	0.0
6	AM-1	0.0	8.1	0.0	0.0
7	AM-2	0.0	15.4	0.0	0.0
8	AM-4	0.0	21.0	3.9	0.0
9	AM-5	1.8	5.7	0.0	9.8
10	AM-6	0.0	18.0	0.0	0.0
11	AM-7	0.0	20.9	0.3	0.0
12	AM-8	9.0	4.6	4.1	78.6
13	AM-9	10.9	4.3	5.2	81.7
14	AM-10	32.6	34.7	11.3	131.8
15	AM-11	27.0	7.5	18.4	185.4
16	AM-16	94.2	42.3	55.3	504.1
17	AM-17	89.8	42.9	53.6	516.3
18	AM-20	185.8	72.3	141.3	1021.0
19	AM-21	205.1	78.4	121.3	1011.0
20	WATER BLA	0.0	9.1	0.0	0.0
21	REPEAT OF	0.0	0.0	0.0	0.0
22	BACKSCATT	461.7	0.0	189.3	58.2
23	AM-1	0.0	0.0	0.1	20.6
24	AM-4	0.0	0.0	6.5	13.7
25	AM-6	0.0	7.0	0.0	27.0
26	AM-8	2.4	0.0	3.9	88.1
27	AM-10	33.0	9.8	20.3	185.3
28	AM-12	38.4	9.4	19.9	243.8
29	AM-13	31.8	8.3	30.4	234.4
30	AM-14	82.0	29.5	50.9	473.4
31	AM-15	77.4	19.1	57.4	476.3
32	AM-12	33.1	17.9	14.8	234.2
33	AM-14	80.1	26.8	53.8	475.3
34	AM-16	88.3	34.1	59.4	523.5
35	AM-20	198.0	74.6	127.6	1041.0
36	ACS-M	89.9	120.3	79.9	77.5
37	AM-20	198.1	69.1	130.6	1046 0
38	WATER BLA	0.0	0.0	0.0	0.0
39	AM-8	7.6	11.1	0.0	70.1
40	AM-10	31.0	14.3	17.7	172.5
41	AM-14	79.7	32.4	53.4	471.2
42	AM-18	173.9	51.0	122.1	957.8
43	AM-14	80.0	31.5	41.9	481.7
44	AM-18	173.7	52.0	111.9	978.6
45	AM-16	96.2	39.2	53.3	514.2
46	LWEM-21	98.9	45.[illegible]	85.1	107.6
47	LWEM-22	99.3	48.3	76.5	103.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
INITIAL CALIBRATION AND DAY 2 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	ACS-M	94.4	115.0	71.5	85.6
2	WATER BLA	0.0	1.0	0.0	0.0
3	AM-1	0.0	0.0	0.0	0.0
4	AM-4	0.0	0.0	0.7	28.2
5	AM-6	0.0	10.2	0.8	5.8
6	AM-8	11 1	4.8	5.3	79.2
7	AM-10	23.3	15.9	14.3	175.8
8	AM-12	39.8	0.0	33.3	261.2
9	AM-14	77.7	33.8	48.8	467.6
10	AM-16	97.0	32.1	73.2	510.0
11	AM-20	192.9	50.3	138.3	1076.0
12	ACS-M-2	89.8	102.3	82.6	103.7
13	AM-16	97.4	22.9	69.2	533.8
14	AM-20	197.3	83.0	133.5	1016.0
15	WATER BLA	0.0	0.0	0.0	8.1
16	AM-8	12.7	0.4	0.0	94.8
17	AM-10	25.0	11.8	11.5	190.2
18	AM-14	79.1	18.9	65.7	484.5
19	AM-14	75.6	20.0	45.8	497.1
20	AM-18	175.4	69.7	116.0	938.3
21	AM-18	178.7	34.9	127.2	1003.0
22	LWEM-21	93.3	32.0	81.9	135.8
23	LWEM-22	98.6	56.0	79.7	88.8
24	AM-16	97.1	27.9	67.9	525.6
25	AM-20	197.7	60.9	142.1	1051.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	ACS-M	108.8	111.8	78.9	94.4
2	WATER BLA	0.0	0.0	0.0	3.8
3	AM-1	0.0	22.4	0.0	0.0
4	AM-4	0.0	23.6	0.0	0.0
5	AM-6	3.8	19.2	0.0	0.0
6	AM-8	12.2	10.7	0.9	72.5
7	AM-10	53.6	36.6	12.1	127.1
8	AM-12	40.1	34.0	27.5	192.2
9	AM-14	88.7	29.8	52.0	474.5
10	AM-16	95.6	42.7	59.2	497.3
11	AM-20	194.5	61.0	144.7	1059.0
12	ACS-M	90.7	110.8	84.5	87.3
13	AM-16	92.3	45.4	62.4	498.0
14	AM-20	195.9	68.5	132.2	1054.0
15	WATER BLA	0.0	0.0	0.0	0.0
16	AM-3	0.0	13.6	0.0	0.0
17	AM-6	0.0	7.2	2.2	12.8
18	AM-8	9.8	21.6	0.0	36.4
19	AM-10	34.8	33.3	6.9	143.6
20	AM-12	48.7	28.1	27.3	198.2
21	AM 14	87.9	42.4	47.0	456.9
22	AM-18	175.8	62.5	133.4	959.6
23	AM-16	99.4	41.6	60.2	505.1
24	AM-20	196.7	87.0	124.8	1028.0
25	WATER BLA	0.6	0.0	0.0	10.9
26	AM-8	5.4	20.3	0.0	46.7
27	AM-10	30.6	29.8	16.0	146.1
28	AM-14	89.1	41.9	58.1	453.0
29	AM-14	87.6	48.5	44.7	435.1
30	AM-18	181.8	69.0	110.2	949.1
31	AM-18	176.0	68.3	110.7	947.8
32	LWEM-21	104.3	53.2	83.5	91.4
33	LWEM-22	98.2	55.2	86.8	88.9
34	AM-16	97.7	37.8	59.1	516.9
35	AM-20	197.5	60.2	146.0	1042.0

TITLE: X-MET 840 LABORATORY CONCENTRATION, WATER, ALL
DAY 2 CERTIFICATION AND DAY 4 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	103.1	43.6	62.5	497.9
2	AM-20	196.6	60.2	133.9	1071.0
3	AM-3	0.0	11.6	0.0	0.0
4	AM-6	1.4	0.0	4.0	36.9
5	AM-8	11.2	0.0	12.5	80.7
6	AM-10	26.3	19.0	18.0	168.5
7	AM-12	36.5	34.0	16.3	194.8
8	AM-14	84.0	46.5	55.2	[illegible]
9	AM-18	180.0	54.0	111.6	978.1
10	WATER BLA	0.0	0.0	0.0	34.7
11	AM-6	10.1	10.2	9.9	61.5
12	AM-10	29.3	25.0	22.5	143.3
13	AM-14	87.4	28.9	64.4	457.0
14	AM-14	86.1	30.5	53.2	476.4
15	AM-18	172.2	71.6	119.7	919.5
16	AM-18	184.9	79.4	115.7	917.0
17	LWEN-21	98.0	54.4	86.7	95.3
18	LWEN-22	95.9	55.1	73.6	98.4
19	AM-16	99.2	43.8	50.6	516.8
20	AM-20	182.5	71.6	139.0	1051.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
DAY 3 CERTIFICATION AND DAY 5 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	90.9	41.9	56.7	514.0
2	AM-20	189.7	69.6	128.4	1060.0
3	AM-3	0.0	9.2	0.0	0.0
4	AM-6	0.0	7.8	1.7	7.8
5	AM-8	11.1	14.0	9.5	55.4
6	AM-10	25.3	28.0	14.9	139.8
7	AM-12	41.3	22.4	25.8	204.9
8	AM-14	81.7	39.8	56.3	444.4
9	AM-18	177.1	74.6	116.1	922.7
10	WATER BLA	0.0	0.0	5.4	23.1
11	AM-8	8.7	11.1	5.1	60.9
12	AM-10	29.0	23.3	16.7	153.1
13	AM-14	84.1	35.1	51.2	443.8
14	AM-14	81.4	33.9	63.0	453.8
15	AM-18	175.9	78.0	112.3	932.9
16	AM-18	177.6	58.1	118.9	947.5
17	LWEN-21	104.3	65.9	82.0	66.1
18	LWEN-22	97.6	65.2	95.1	96.7
19	AM-16	96.5	51.6	71.9	473.5
20	AM-20	194.4	69.1	133.0	1065.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
DAY 4 CERTIFICATION AND DAY 6 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	96.8	46.4	56.9	504.5
2	AM-20	189.8	52.4	137.3	1088.0
3	AM-3	0.0	0.0	0.0	1.1
4	AM-6	0.0	0.0	0.0	33.2
5	AM-8	4.0	3.4	0.1	91.2
6	AM-10	30.1	2.4	19.7	194.0
7	AM-12	39.6	22.8	19.5	214.8
8	AM-14	78.9	33.0	51.4	467.0
9	AM-18	174.5	68.6	111.0	936.0
10	WATER BLA	0.0	0.0	2.7	32.4
11	AM-8	10.8	3.9	10.1	77.6
12	AM-10	32.1	14.5	15.6	171.9
13	AM-14	87.4	25.3	67.0	470.2
14	AM-14	87.6	30.7	55.2	464.4
15	AM-18	182.2	68.6	117.7	925.0
16	AM-18	181.3	62.4	113.9	953.4
17	LWEN-21	94.5	50.8	85.7	94.5
18	LWEN-22	93.7	38.1	82.3	129.6
19	AM-16	93.2	36.3	66.8	512.2
20	AM-20	192.8	60.8	135.3	1066.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
DAY 7 EQUIVALENCY

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	94.1	51.4	61.7	486.0
2	AM-20	193.9	60.5	146.5	1050.0
3	WATER BLA	0.0	0.0	1.3	0.0
4	AM-8	9.2	7.6	1.0	73.5
5	AM-10	33.4	16.8	14.3	166.7
6	AM-14	82.2	42.6	43.5	448.4
7	AM-14	86.6	39.9	45.4	459.7
8	AM-18	181.7	71.4	117.8	929.7
9	AM-18	177.1	76.8	106.4	923.8
10	LWEN-21	94.2	56.1	85.0	83.0
11	LWEN-22	97.5	58.3	82.1	78.2
12	AM-16	99.4	55.4	59.4	475.9
13	AM-20	194.4	75.8	125.4	1051.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
DAY 8 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	94.2	50.6	59.7	497.5
2	AM-20	198.4	70.3	138.9	1039.0
3	WATER BLA	0.0	0.0	8.5	54.6
4	SP-8	9.4	9.0	6.0	65.8
5	AM-10	28.2	10.1	21.0	177.2
6	AM-14	87.6	31.1	62.5	463.6
7	AM-14	82.5	21.4	49.5	493.3
8	AM-18	176.6	64.4	120.6	947.1
9	AM-18	178.6	72.7	114.5	939.9
10	LWEN-21	97.3	46.4	78.8	112.0
11	LWEN-22	98.3	32.4	89.9	128.8
12	AM-16	96.4	34.0	59.8	528.8
13	AM-20	199.8	58.0	142.1	1069.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
DAY 9 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	97.4	25.1	58.0	547.6
2	AM-20	194.4	52.3	142.6	1077.0
3	WATER BLA	0.0	0.0	4.8	56.1
4	AM-8	2.5	0.0	4.5	128.7
5	AM-10	28.2	0.0	17.4	220.0
6	AM-14	85.2	12.8	49.4	519.2
7	AM-14	86.9	6.9	54.9	520.6
8	AM-18	173.4	49.1	128.9	981.5
9	AM-18	177.0	58.2	115.4	975.0
10	LWEN-21	93.2	33.3	87.2	134.8
11	LWEN-22	95.7	25.1	90.1	149.7
12	AM-16	95.1	30.3	62.4	536.3
13	AM-20	195.4	40.2	143.3	1116.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WATER, AL
DAY 10 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	91.7	17.2	71.0	545.3
2	AM-20	197.8	37.9	145.3	1109.0
3	WATER BLA	0.0	0.0	2.0	98.7
4	AM-8	6.2	0.0	4.5	152.1
5	AM-10	24.4	0.0	24.1	257.3
6	AM-14	78.8	5.3	50.6	531.4
7	AM-14	80.2	0.0	71.7	547.3
8	AM-18	175.3	31.8	128.5	1009.0
9	AM-18	176.3	41.3	129.0	990.9
10	LWEN-21	97.3	28.0	82.3	148.9
11	LWEN-22	84.6	6.0	93.5	191.8
12	AM-16	92.1	0.0	75.4	588.2
13	AM-20	199.7	26.1	162.8	1119.0

TABLE: COMPILATION OF LABORATORY NET INTENSITIES, SOIL, ALL ELEMENTS.

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
PRE-CERTIFICATION AND DAY 1 EQUIVALENCY.

	SAMPLE	Fe NET INT	CU CONC	Cu NET INT	AS CONC	As NET INT	HG CONC	Hg NET INT	PB CONC	Pb NET INT	BS NET INT
1	26	665.20		112.50		207.30		64.83		93.54	643.3
2	SCS-M	600.30		-22.56		54.91		1.5[illegible]		-11.57	730.2
3	INSTRUMEN	1.31		19.68		-7.88		-0.84		8.52	1152.0
4	INSTRUMEN	-0.41		-13.63		-8.11		-0.61		7.40	1204.0
5	RAJ BLANK	127.10		152.00		-11.96		72.54		-3.64	286.6
6	SOIL BLAN	545.40	0.0	-41.90	0.0	32.75	0.0	-4.01	0.0	-26.06	738.8
7	SOIL BLAN	540.00	0.0	-41.99	0.0	32.84	0.0	-3.75	0.0	-25.46	736.2
8	SM-1	538.10	9.4	-41.32	4.5	32.06	10.9	-3.50	22.5	-23.93	741.6
9	SM-2	517.90	9.4	-43.64	4.5	35.02	10.9	-3.77	22.5	-25.66	744.0
10	SM-3	578.90	12.5	-39.90	6.0	33.50	14.5	-4.86	30.0	-23.78	733.7
11	SM-4	542.00	18.8	-40.73	9.0	34.99	21.8	-2.89	45.0	-25.33	741.8
12	SM-5	507.50	18.8	-42.88	9.0	33.97	21.8	-3.71	45.0	-24.62	752.0
13	SM-6	581.80	25.0	-38.58	12.0	34.21	29.0	-3.02	60.0	-23.65	736.0
14	SM-7	512.70	25.0	-41.27	12.0	33.40	29.0	-3.56	60.0	-22.23	740.6
15	SM-8	493.70	50.0	-40.94	24.0	37.87	58.0	-3.25	120.0	-24.27	740.4
16	SM-9	536.10	50.0	-37.85	24.0	38.10	58.0	-3.87	120.0	-23.51	738.2
17	SM-10	472.30	100.0	-36.55	48.0	42.05	116.0	-1.99	240.0	-19.28	739.1
18	SM-11	508.00	100.0	-36.89	48.0	39.61	116.0	-1.84	240.0	-18.61	731.8
19	SM-12	493.70	125.0	-34.06	60.0	37.47	145.0	-0.82	300.0	-13.48	733.3
20	SM-13	512.80	125.0	-34.72	60.0	39.79	145.0	-0.10	300.0	-16.26	740.6
21	SM-14	516.10	250.0	-24.85	120.0	45.38	290.0	2.09	600.0	-2.39	728.5
22	SM-15	524.90	250.0	-24.59	120.0	44.82	290.0	0.85	600.0	-1.45	727.8
23	SM-16	621.00	312.0	-17.41	150.0	43.85	362.0	2.29	750.0	6.61	711.5
24	SM-17	509.30	312.0	-21.44	150.0	46.62	362.0	3.50	750.0	5.65	724.6
25	SM-18	491.00	500.0	-7.29	240.0	52.20	580.0	10.15	1200.0	22.69	713.8
26	SM-19	486.60	500.0	-7.76	240.0	53.73	580.0	8.49	1200.0	21.03	715.6
27	SM-20	589.90	625.0	0.84	300.0	54.29	725.0	12.77	1500.0	34.53	706.4
28	SM-21	494.60	625.0	-0.35	300.0	59.20	725.0	12.36	1500.0	32.98	712.9
29	INST. BLA	0.73		-12.68		0.29		-0.30		0.35	1202.0
30	INST BLAN	543.00		-41.66		33.28		-4.46		-26.17	734.7
31	SM-1	532.70		-41.04		36.01		-4.88		-26.30	739.1
32	SM-4	543.10		-40.69		34.65		-4.14		-24.49	747.5
33	SM-6	585.10		-37.21		32.61		-3.57		-21.56	732.7
34	SM-8	484.20		-40.90		36.45		-3.72		-22.38	741.2
35	SM-10	471.00		-37.97		41.64		-1.96		-19.23	740.6
36	SM-12	488.40		-34.03		38.14		-1.19		-13.92	732.6
37	SM-14	518.00		-24.80		45.09		2.65		-2.41	730.1
38	SM-16	618.70		-17.99		43.12		4.66		5.70	712.0
39	SM-20	589.00		0.56		55.98		13.56		31.13	707.9
40	SCS-M	597.7[illegible]		-21.20		54.76		0.52		-10.28	723.1
41	SM-20	589.70		-0.39		55.19		13.44		33.01	708.3
42	SOIL BLAN	540.90		-42.36		33.69		-4.01		-26.70	735.6
43	SM-8	485.30		-38.97		37.36		-4.29		-22.23	738.5
44	SM-10	468.16		-38.14		40.03		-1.17		-17.78	738.3
45	SM-14	517.90		-24.58		44.42		1.99		-1.00	728.0
46	SM-14	516.30		-25.90		41.15		1.87		1.44	726.7
47	SM-18	487.30		-8.78		54.79		9.22		21.72	718.7
48	SM-18	488.50		-9.19		50.31		9.49		24.43	722.4
49	LSEM-21	567.70		-35.[illegible]		40.28		-1.92		20.0*	736.9
50	LSEM-22	538.00		-35.27		41.85		-1.81		-21.65	737.5

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
INITIAL CALIBRATION AND DAY 2 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SCS-M	595.80	-20.27	53.80	1.43	-10.40	721.4
2	SOIL BLAN	536.00	-43.54	33.51	-4.74	-25.40	736.6
3	SOIL BLAN	537.60	-40.97	33.00	-4.16	-24.63	729.6
4	SM-1	535.00	-42.17	32.49	-3.77	-23.76	737.1
5	SM-4	539.50	-39.99	33.40	-3.31	-23.99	740.5
6	SM-6	585.50	-37.15	32.33	-3.40	-21.71	733.6
7	SM-8	488.60	-39.26	36.17	-2.92	-21.97	734.4
8	SM-10	466.60	-35.96	37.16	-1.75	-14.66	730.6
9	SM-12	492.00	-33.76	38.80	-1.31	-13.85	730.3
10	SM-14	513.90	-24.81	41.94	3.51	0.14	722.5
11	SM-16	617.90	-18.72	41.76	3.48	7.10	712.7
12	SM-20	584.80	1.25	56.13	14.68	31.05	704.1
13	SCS-M	595.00	-20.55	51.36	1.24	-9.56	722.0
14	SM-16	613.90	-17.67	42.76	4.26	5.52	708.5
15	SM-20	591.C0	0.25	53.64	13.93	32.99	704.8
16	SOIL BLAN	543.50	-41.22	31.85	-3.56	-25.00	733.8
17	SM-8	488.70	-38.63	36.66	-3.70	-22.75	737.5
18	SM-10	467.30	-37.19	39.77	-0.85	-18.32	736.2
19	SM-14	514.30	-24.58	43.77	3.13	-0.68	721.4
20	SM-14	516.30	-24.60	42.97	1.67	0.38	725.5
21	SM-18	489.70	-8.91	50.21	10.57	22.49	716.3
22	SM-18	490.50	-8.68	50.60	10.67	22.37	717.3
23	LSEM-21	561.90	-36.15	39.54	-0.86	-20.03	733.0
24	LSEM-22	535.10	-35.84	41.64	-0.40	-21.97	735.1
25	SM-16	615.10	-18.06	44.75	2.22	5.91	715.6
26	SM-20	585.70	0.25	52.27	13.80	34.68	705.8

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SCS-M	592.50	-22.04	54.69	1.51	-11.91	724.9
2	SOIL BLAN	542.70	-42.34	31.64	-3.33	-24.92	733.5
3	SM-1	538.70	-42.20	34.41	-4.08	-24.33	733.8
4	SM-4	536.30	-40.51	31.39	-4.27	-21.78	736.0
5	SM-6	584.90	-39.43	33.49	-2.10	-22.91	734.0
6	SM-8	493.20	-39.73	35.05	-3.94	-20.26	738.1
7	SM-10	470.60	-37.06	39.19	-1.00	-17.55	733.5
8	SM-12	490.40	-35.25	40.10	-1.03	-15.13	733.1
9	SM-14	516.60	-24.78	44.28	2.47	-1.09	724.6
10	SM-16	615.00	-18.33	45.62	4.07	3.72	714.4
11	SM-20	593.30	0.05	50.93	14.03	34.83	706.4
12	SCS-M	594.60	-21.92	53.69	1.28	-9.96	725.4
13	SM-[illegible]	618.70	-19.65	44.75	3.41	4.71	713.7
14	SM-2[illegible]	595.80	-0.30	56.80	12.85	30.69	707.3
15	SOIL BLAN	544.00	-41.43	31.67	-4.93	-24.12	735.5
16	SM-3	577.10	-40.62	35.68	-4.29	-25.00	729.4
17	SM-6	586.70	-38.26	33.67	-3.37	-22.79	735.7
18	SM-8	491.90	-40.01	35.46	-4.74	-21.47	735.1
19	SM-10	470.10	-36.08	40.11	-1.00	-18.43	732.7
20	SM-12	490.80	-33.98	38.18	-0.51	-14.06	730.0
21	SM-14	518.00	-24.24	42.53	1.52	1.52	722.4
22	SM-18	488.70	-8.97	51.98	10.91	21.66	719.5
23	SM-16	619.20	-18.42	42.27	2.98	6.68	712.6
24	SM-20	592.00	2.10	53.32	12.77	33.89	703.7
25	SOIL BLAN	540.20	-43.01	31.30	-4.85	-23.66	735.9
26	SM-8	490.20	-39.81	36.57	-3.09	-22.04	732.7
27	SM-10	470.00	-35.99	38.81	-3.17	-16.28	733.4
28	SM-14	514.20	-25.22	43.01	2.47	-0.01	724.3
29	SM-14	516.10	-23.15	42.30	2.60	-0.04	721.0
30	SM-18	487.50	-8.73	52.69	8.30	22.47	718.8
31	SM-18	493.20	-9.01	53.16	9.04	20.70	720.1
32	LSEM-21	566.90	-34.15	38.68	-2.22	-18.40	735.8
33	LSEM-22	533.70	-34.95	40.67	-1.99	-20.65	738.7
34	SM-6	584.10	-36.89	35.98	-4.08	-24.68	738.0
35	SM-16	612.60	-17.92	44.48	1.57	5.70	713.8
36	SM-20	589.20	0.58	52.27	12.31	35.81	709.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 2 CERTIFICATION AND DAY 4 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SM-16	613.50	-19.95	43.51	5.14	4.42	715.0
2	SM-20	588.30	1.22	53.22	12.60	34.23	704.6
3	SM-3	572.20	-39.27	31.56	-3.63	-22.98	727.8
4	SM-6	587.80	-38.06	33.92	-2.63	-23.65	733.4
5	SM-8	490.60	-40.74	36.66	-4.17	-21.70	738.3
6	SM-10	471.20	-36.63	37.77	-1.15	-16.02	733.9
7	SM-12	491.00	-35.62	38.16	-1.04	-14.23	729.6
8	SM-14	517.10	-24.14	41.64	3.28	1.36	723.6
9	SM-18	488.40	-8.96	48.16	9.43	24.41	716.0
10	SOIL BLAN	538.00	-42.56	33.63	-4.16	-25.61	736.7
11	SM-8	489.50	-38.83	33.29	-3.23	-19.42	732.3
12	SM-10	468.20	-37.81	39.86	-0.95	-17.99	737.9
13	SM-14	519.20	-25.23	45.12	3.46	-2.10	729.1
14	SM-14	516.30	-24.08	43.97	2.71	-0.99	727.1
15	SM-18	489.90	-9.19	49.63	11.22	22.38	717.6
16	SM-18	494.60	-7.49	51.88	8.02	23.52	715.7
17	LSEM-21	567.30	-34.63	40.66	-2.50	-19.62	735.0
18	LSEM-22	538.60	-34.44	41.81	-2.01	-21.00	729.5
19	SM-16	615.90	-18.61	43.27	3.32	5.10	713.7
20	SM-20	588.90	-0.39	51.73	14.40	33.97	702.2

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SCS-M	592.50	-22.04	54.69	1.51	-11.91	724.9
2	SOIL BLAN	542.70	-42.34	31.64	-3.33	-24.92	733.5
3	SH-1	538.70	-42.20	34.41	-4.08	-24.83	738.8
4	SH-4	536.30	-40.51	31.39	-4.27	-21.78	736.0
5	SH-6	584.90	-39.43	33.49	-2.10	-22.91	734.0
6	SH-8	493.20	-39.73	35.05	-3.94	-20.26	738.1
7	SH-10	470.60	-37.06	39.19	-1.00	-17.55	733.5
8	SH-12	490.40	-35.25	40.10	-1.03	-15.13	733.1
9	SH-14	516.60	-24.78	44.28	2.47	-1.09	724.6
10	SH-16	615.00	-18.33	45.62	4.07	3.7[illegible]	714.4
11	SH-20	593.30	0.05	50.73	14.03	34.83	706.4
12	SCS-M	594.60	-21.92	53.69	1.28	-9.96	725.4
13	SH-16	618.70	-19.65	44.75	3.41	4.71	713.7
14	SH-20	595.80	-0.30	56.80	12.85	30.69	707.3
15	SOIL BLAN	544.00	-41.43	31.67	-4.93	-24.12	735.5
16	SH-3	577.10	-40.62	35.68	-4.29	-25.00	729.4
17	SH-6	586.70	-38.26	33.67	-3.37	-22.79	735.7
18	SH-8	491.90	-40.01	35.46	-4.74	-21.47	735.1
19	SH-10	470.10	-36.08	40.11	-1.00	-18.43	732.7
20	SH-12	490.[illegible]	-33.98	38.18	-0.51	-14.06	730.0
21	SH-14	518.00	-24.24	42.58	1.52	1.52	722.4
22	SH-18	488.70	-8.97	51.98	10.91	21.66	719.5
23	SH-16	619.20	-18.42	42.27	2.98	6.68	712.6
24	SH-20	592.00	2.10	53.32	12.77	33.89	703.7
25	SOIL BLAN	540.20	-43.01	31.30	-4.85	-23.66	735.9
26	SH-8	490.20	-39.81	36.57	-3.09	-22.04	732.7
27	SH-10	470.00	-35.99	38.81	-3.17	-16.28	733.4
28	SH-14	514.20	-25.22	43.01	2.47	-0.01	724.3
29	SH-14	516.10	-23.15	42.30	2.60	-0.04	721.0
30	SH-18	487.50	-8.73	52.69	8.30	22.47	718.8
31	SH-18	493.20	-9.01	53.16	9.04	20.70	720.1
32	LSEM-21	566.90	-34.15	38.68	-2.22	-18.40	735.8
33	LSEM-22	533.70	-34.95	40.67	-1.99	-20.65	738.7
34	SH-6	584.10	-36.89	35.98	-4.08	-24.68	738.0
35	SH-16	612.60	-17.92	44.48	1.57	5.70	713.8
36	SH-20	589.20	0.58	52.27	12.31	35.81	709.0

TITLE: X-MET 840 LABORATORY CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 2 CERTIFICATION AND DAY 4 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SH-16	613.50	-19.95	43.51	5.14	4.42	715.0
2	SH-20	588.30	1.22	53.22	12.60	34.23	704.6
3	SH-3	572.20	-39.27	31.56	-3.63	-22.98	727.8
4	SH-6	587.80	-38.06	33.92	-2.63	-23.65	733.4
5	SH-8	490.60	-40.74	36.66	-4.17	-21.70	738.3
6	SH-10	471.20	-36.63	37.77	-1.15	-16.02	733.9
7	SH-12	491.00	-35.82	38.16	-1.04	-14.23	729.6
8	SH-14	517.10	-24.14	41.64	3.28	1.36	723.6
9	SH-18	488.40	-8.96	48.16	9.43	24.41	716.0
10	SOIL BLAN	538.00	-42.56	33.63	-4.16	-25.61	736.7
11	SH-8	489.50	-38.83	33.29	-3.23	-19.42	732.3
12	SH-10	468.20	-37.81	39.86	-0.95	-17.99	737.9
13	SH-14	519.20	25.23	45.12	3.44	-2.10	729.1
14	SH-14	514.30	-24.08	43.97	2.71	-0.99	727.1
15	SH-18	489.90	-9.19	49.63	11.22	22.38	717.6
16	SH-18	494.60	-7.49	51.88	8.02	23.52	715.7
17	LSEM-21	567.30	-34.63	40.66	-2.50	-19.62	735.0
18	LSEM-24	538.60	-34.44	41.81	-2.01	-21.00	729.5
19	SH-16	615.90	-18.61	43.27	3.32	5.10	713.7
20	SH-20	588.90	-0.39	51.73	14.40	33.97	702.2

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 7 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SM-16	618.00	-18.28	42.74	3.08	5.94	709.3
2	SM-20	587.90	1.57	54.78	13.37	32.01	703.0
3	SOIL BLAN	544.20	-42.46	32.79	-2.56	-26.60	730.5
4	SM-8	491.30	-39.70	35.28	-3.90	-20.71	735.0
5	SM-10	469.00	-35.69	38.28	-0.71	-17.33	732.7
6	SM-14	516.90	-24.07	42.22	2.28	1.01	724.5
7	SM-14	516.50	-24.12	42.75	2.84	-0.40	719.8
8	SM-18	488.30	-9.55	52.89	6.36	23.19	720.3
9	SM-18	490.20	-7.49	53.99	6.96	21.88	716.5
10	LSEM-21	567.30	-34.49	42.02	-2.65	-21.12	729.5
11	LSEM-22	535.70	-34.43	44.39	-3.68	-21.53	734.3
12	SM-16	617.60	-17.92	45.37	2.01	4.98	712.0
13	SM-20	590.00	1.72	53.97	11.59	32.64	700.8

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 8 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SM-16	618.10	-18.35	41.70	2.78	6.84	713.8
2	SM-20	592.20	2.42	54.06	12.23	33.33	704.6
3	SOIL BLAN	543.60	-42.15	31.39	-3.02	-25.46	731.2
4	SM-8	488.80	-39.01	36.47	-3.85	-22.11	733.2
5	SM-10	468.30	-36.13	38.87	-1.42	-17.06	733.2
6	SM-14	516.00	-24.02	42.68	2.40	0.25	721.9
7	SM-14	516.40	-26.38	43.75	4.08	-1.92	723.1
8	SM-18	490.80	-9.68	51.56	8.25	22.82	718.1
9	SM-18	488.80	-8.26	53.23	6.60	22.48	716.5
10	LSEM-21	566.50	-35.79	40.84	-1.66	-20.80	733.8
11	LSEM-22	539.10	-34.63	43.95	-1.18	-23.48	734.0
12	SM-16	615.40	-18.40	44.55	4.00	4.90	711.8
13	SM-20	588.60	1.29	54.82	11.94	32.81	703.0

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 9 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SM-16	615.70	-18.12	43.55	4.07	5.07	709.0
2	SM-20	588.30	0.47	51.59	13.00	34.46	703.6
3	SOIL BLAN	541.10	-41.69	29.98	-3.44	-23.93	729.7
4	SM-8	487.00	-39.60	34.13	-3.73	-20.30	734.1
5	SM-10	470.60	-35.86	39.57	-1.72	-17.38	734.5
6	SM-14	518.20	-26.23	42.71	3.03	-0.77	721.7
7	SM-14	514.90	-23.54	42.26	3.03	0.60	721.0
8	SM-18	488.10	-6.55	51.93	7.75	22.34	715.2
9	SM-18	491.50	-7.99	51.56	7.80	23.34	715.3
10	LSEM-21	565.90	-33.10	40.82	-1.76	-20.63	730.6
11	LSEM-22	535.70	-34.46	41.13	-1.37	-20.96	733.3
12	SM-16	621.20	-18.69	44.47	2.23	5.90	713.7
13	SM-20	591.10	1.96	52.76	13.98	31.53	703.8

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 10 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SN-16	606.60	-20.38	40.59	4.44	7.38	714.8
2	SN-20	575.50	-0.70	49.40	14.52	34.13	706.7
3	SOIL BLAN	540.20	-41.73	30.94	-4.49	-23.40	732.3
4	SN-8	487.80	-40.09	35.08	-3.10	-20.97	732.5
5	SN-10	468.60	-36.72	37.50	-1.58	-15.33	730.0
6	SN-14	514.20	-24.79	41.41	4.26	-0.18	722.8
7	SN-14	515.20	-24.84	41.94	2.20	0.71	723.6
8	SN-18	489.10	-8.19	48.17	9.11	24.77	713.7
9	SN-18	409.40	-9.32	50.44	8.27	22.91	715.8
10	LSEN-21	561.10	-34.21	37.24	-1.76	-17.84	726.2
11	LSEN-22	533.00	-34.64	39.90	-2.37	-19.04	727.1
12	SN-16	615.40	-19.55	40.75	4.87	7.57	707.8
13	SN-20	590.70	1.68	48.56	14.05	35.36	699.7

TABLE: COMPILATION OF LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
PRE-CERTIFICATION AND DAY 1 EQUIVALENCY.

			Cu		As		Hg		Pb	BS
	SAMPLE		NET INT		NET INT		NET INT		NET INT	NET INT
1	ACS-N		-220.30		55.32		-26.69		7.99	2436
2	INSTRUMENT BLANK, 1		-2.77		-3.78		-0.75		4.08	1198
3	INSTRUMENT BLANK, 2		-4.38		-2.51		-1.90		3.06	1199
4	WATER BLANK 1	0.00	-252.40	0.00	9.44	0.00	-44.86	0.00	-8.18	2497
5	WATER BLANK 2	0.00	-251.50	0.00	13.28	0.00	-46.37	0.00	-10.77	2499
6	AN-1	4.01	-249.40	1.58	11.78	2.48	-44.79	18.90	-6.07	2494
7	AN-2	4.01	-250.60	1.58	14.56	2.48	-44.77	18.90	-8.45	2499
8	AN-4	8.01	-249.40	3.15	16.70	4.95	-43.76	37.80	-6.61	2503
9	AN-5	8.01	-248.10	3.15	10.86	4.95	-45.42	37.80	-0.74	2499
10	AN-6	8.90	-248.40	3.50	15.58	5.50	-44.77	42.00	-4.55	2493
11	AN-7	8.90	-248.30	3.50	16.68	5.50	-44.60	42.00	-4.24	2490
12	AN-8	17.80	-243.80	7.00	10.47	11.00	-43.61	84.00	9.76	2478
13	AN-9	17.80	-242.40	7.00	10.33	11.00	-43.34	84.00	10.24	2471
14	AN-10	35.60	-237.80	14.00	21.92	22.00	-41.83	168.00	17.88	2479
15	AN-11	35.60	-237.60	14.00	11.57	22.00	-40.17	168.00	26.06	2463
16	AN-12	44.50	-232.80	17.50	12.27	27.50	-39.76	210.00	34.97	2449
17	AN-13	44.50	-234.60	17.50	11.86	27.50	-37.36	210.00	33.54	2445
18	AN-14	89.00	-216.00	35.00	19.95	55.00	-32.44	420.00	70.[illegible]	2398
19	AN-15	89.00	-216.80	35.00	15.97	55.00	-31.00	420.00	70.45	2392
20	AN-16	97.90	-212.90	38.50	24.81	60.50	-31.42	462.00	74.70	2399
21	AN-17	97.90	-212.90	38.50	25.02	60.50	-31.79	462.00	76.56	2390
22	AN-20	196.00	-177.90	77.00	36.25	121.00	-12.56	924.00	153.60	2285
23	AN-21	196.00	-171.70	77.00	38.56	121.00	-16.65	924.00	152.00	2285
24	WATER BLANK W-1		-249.70		12.19		-45.98		-10.80	2498
25	REPEAT OF WATER BLANK		-252.20		5.17		-44.76		-4.88	2495
26	BACKSCATTER SAMPLE BS-1		-4.95		-6.83		-1.92		6.66	1192
27	AN-1		-248.10		3.80		-44.63		0.91	2478
28	AN-4		-248.30		8.57		-43.12		-0.15	2487
29	AN-6		-247.10		11.37		-45.87		1.88	2487
30	AN-8		-245.40		8.29		-43.68		11.21	2477
31	AN-10		-235.30		12.43		-39.69		26.04	2456
32	AN-12		-233.90		15.50		-40.96		33.51	2449
33	AN-14		-216.80		18.90		-31.81		70.30	2400
34	AN-16		-213.40		21.67		-30.52		77.65	2388
35	AN-20		-174.70		37.12		-15.37		156.60	2292
36	ACS-N		-218.20		54.51		-26.09		9.59	2422
37	AN-20		-174.20		35.02		-14.74		157.30	2287
38	WATER BLANK		-250.70		6.90		-47.11		-4.53	2492
39	AN-8		-243.20		12.92		-44.75		8.47	2472
40	AN-10		-236.20		14.16		-40.32		24.09	2440
41	AN-14		-218.10		21.05		-31.93		69.67	2410
42	AN-18		-182.80		28.13		-16.60		143.90	2306
43	AN-14		-217.30		20.69		-34.49		71.27	2408
44	AN-18		-161.30		28.49		-18.74		147.10	2297
45	AN-16		-210.10		23.63		-31.86		76.24	2386
46	LWEN-21		-218.30		25.88		-24.93		14.19	2440
47	LWEN-22		-220.40		27.10		-26.84		13.48	2461

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
INITIAL CALIBRATION AND DAY 2 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1 ACS-H	-215.90	52.48	-27.91	10.82	2417
2 WATER BLANK B-1	-250.30	9.08	-46.01	-6.28	2493
3 AM-1	-251.10	8.72	-44.79	-2.96	2502
4 AM-4	-250.70	7.17	-44.55	2.07	2496
5 AM-6	-249.90	12.57	-44.49	-1.35	2497
6 AM-8	-243.10	10.52	-43.33	9.86	2477
7 AM-10	-239.40	14.76	-41.16	24.60	2470
8 AM-12	-232.70	7.33	-36.67	37.62	2446
9 AM-14	-218.20	21.56	-32.95	69.13	2408
10 AM-16	-211.30	20.91	-27.43	75.59	2385
11 AM-20	-174.40	27.85	-13.14	162.00	2273
12 ACS-H-2	-218.10	47.67	-25.51	13.60	2420
13 AM-16	-210.80	17.43	-28.32	79.22	2334
14 AM-20	-173.50	40.31	-14.14	152.80	2278
15 WATER BLANK B-2	-248.50	4.76	-46.72	-1.01	2478
16 AM-8	-241.80	8.86	-46.35	12.24	2475
17 AM-10	-237.40	13.18	-41.78	26.79	2459
18 AM-14	-218.10	15.91	-29.18	71.71	2403
19 AM-14	-218.00	16.33	-33.62	73.62	2403
20 AM-18	-180.70	35.25	-17.87	140.90	2294
21 AM-18	-180.30	21.99	-15.50	150.80	2294
22 LWEN-21	-220.50	20.89	-25.66	18.49	2447
23 LWEN-22	-219.70	30.02	-26.13	11.32	2453
24 AM-16	-210.90	19.33	-28.58	77.97	2384
25 AM-20	-174.10	31.89	-12.34	158.10	2280

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1 ACS-H	-215.20	51.26	-26.28	12.18	2423
2 WATER BLANK	-250.90	3.43	-45.92	-0.90	2484
3 AM-1	-252.50	17.22	-45.64	-10.60	2514
4 AM-4	-249.80	17.69	-46.72	-6.49	2509
5 AM-6	-249.80	16.03	-45.51	-5.22	2516
6 AM-8	-244.70	12.77	-44.38	8.83	2493
7 AM-10	-236.80	22.62	-41.64	17.16	2473
8 AM-12	-234.40	21.66	-38.03	27.10	2462
9 AM-14	-217.00	20.06	-32.21	70.18	2421
10 AM-16	-213.20	24.95	-30.55	73.65	2403
11 AM-20	-174.30	31.93	-11.80	159.30	2273
12 ACS-H	-218.70	50.92	-25.10	11.09	2426
13 AM-16	-214.30	25.98	-29.86	73.76	2403
14 AM-20	-171.90	34.80	-14.38	158.60	2262
15 WATER BLANK	-249.10	4.18	-45.16	-2.89	2473
16 AM-3	-250.90	13.90	-45.66	-5.54	2504
17 AM-6	-249.50	11.44	-44.16	-0.28	2497
18 AM-8	-246.30	14.93	-44.88	3.31	2502
19 AM-10	-236.80	21.38	-42.86	19.68	2478
20 AM-12	-230.90	19.40	-38.02	28.01	2453
21 AM-14	-216.60	24.85	-33.34	67.49	2418
22 AM-18	-183.70	32.53	-14.26	141.10	2314
23 AM-16	-211.60	24.56	-30.31	74.84	2398
24 AM-20	-173.30	41.83	-15.95	154.70	2273
25 WATER BLANK	-247.10	3.96	-46.69	-0.56	2462
26 AM-8	-247.70	16.42	-45.45	4.89	2503
27 AM-10	-237.50	20.03	-40.74	20.06	2471
28 AM-14	-216.80	24.64	-30.84	66.89	2418
29 AM-14	-216.60	27.15	-33.85	64.16	2418
30 AM-18	-182.10	34.98	-19.11	142.60	2321
31 AM-18	-183.50	34.69	-19.03	142.40	2321
32 LWEN-21	-218.90	28.98	-25.29	11.71	2457
33 LWEN-22	-220.90	29.74	-24.59	10.11	2459
34 AM-16	-211.80	23.11	-30.57	76.65	2396
35 AM-20	-173.60	35.06	-11.53	156.80	2874

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 2 CERTIFICATION AND DAY 4 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
2 AM-20	-172.80	31.63	-14.03	161.30	2270
3 AM-3	-250.20	13.14	-45.92	-5.82	2504
4 AM-6	-247.50	7.21	-43.71	3.40	2491
5 AM-8	-244.60	8.66	-41.66	10.08	2486
6 AM-10	-238.70	15.93	-40.29	23.48	2470
7 AM-12	-235.10	21.63	-40.63	27.50	2464
8 AM-14	-217.90	26.41	-31.50	64.85	2416
9 AM-18	-180.60	29.25	-18.80	147.00	2306
10 WATER BLANK	-247.30	-2.02	-44.71	3.06	2457
11 AM-8	-244.30	12.60	-42.26	7.15	2482
12 AM-10	-238.70	18.23	-39.25	19.64	2475
13 AM-14	-216.10	19.70	-29.44	67.50	2406
14 AM-14	-215.30	20.31	-31.92	70.15	2401
15 AM-18	-182.10	35.97	-17.11	138.10	2311
16 AM-18	-179.90	38.95	-17.92	137.70	2310
17 LWEM-21	-221.10	29.43	-24.63	12.30	2460
18 LWEM-22	-221.20	29.70	-27.49	12.79	2461
19 AM-16	-211.80	25.39	-32.45	76.63	2403
20 AM-20	-177.20	35.98	-13.03	158.20	2273

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 3 CERTIFICATION AND DAY 5 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1 AM-16	-212.90	24.65	-31.10	76.20	2391
2 AM-20	-172.90	35.19	-15.18	159.50	2257
3 AM-3	-247.50	12.19	-45.22	-5.52	2483
4 AM-6	-248.10	11.66	-44.26	-1.05	2488
5 AM-8	-244.10	14.02	-42.35	6.22	2483
6 AM-10	-238.50	19.35	-41.01	19.10	2468
7 AM-12	-233.10	17.24	-38.40	29.04	2455
8 AM-14	-217.30	23.85	-31.25	65.57	2406
9 AM-18	-181.50	37.12	-17.85	138.60	2305
10 WATER BLANK	-245.40	-1.33	-43.34	1.29	2444
11 AM-8	-245.50	12.94	-43.42	7.06	2491
12 AM-10	-238.30	17.57	-40.59	21.13	2473
13 AM-14	-215.60	22.08	-32.37	68.55	2400
14 AM-14	-217.70	22.37	-29.77	67.02	2406
15 AM-18	-181.50	38.41	-18.66	140.10	2304
16 AM-18	-181.80	30.83	-17.27	142.40	2308
17 LWEM-21	-218.70	33.79	-25.62	7.86	2457
18 LWEM-22	-220.30	25.93	-22.79	12.22	2450
19 AM-16	-212.80	28.34	-27.76	70.03	2397
20 AM-20	-171.60	35.00	-14.21	157.20	2255

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 4 CERTIFICATION AND DAY 6 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	AM-16	-210.70	26.37	-31.04	74.76	2387
2	AM-20	-172.50	28.67	-13.32	163.70	2250
3	AM-3	-249.50	8.01	-44.82	-2.07	2493
4	AM-6	-247.50	8.37	-45.26	2.84	2489
5	AM-8	-245.00	9.99	-44.59	11.68	2478
6	AM-10	-236.90	9.63	-39.86	27.36	2463
7	AM-12	-233.00	17.77	-39.85	30.55	2453
8	AM-14	-215.80	21.28	-32.33	69.03	2390
9	AM-18	-180.70	34.84	-18.92	140.60	2295
10	WATER BLANK	-245.70	-1.94	-43.98	2.71	2442
11	AM-8	-244.00	10.18	-42.22	9.61	2481
12	AM-10	-235.90	14.23	-40.80	24.00	2462
13	AM-14	-215.70	18.34	-28.86	69.51	2402
14	AM-14	-216.30	20.40	-31.48	68.64	2401
15	AM-18	-180.10	34.83	-17.51	138.90	2304
16	AM-18	-180.40	32.48	-18.31	143.30	2306
17	LWEM-21	-220.90	28.06	-24.83	12.18	2451
18	LWEM-22	-220.10	23.20	-25.57	17.54	2444
19	AM-16	-211.40	22.52	-28.84	75.93	2380
20	AM-20	-172.50	31.85	-13.74	160.40	2258

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 7 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	AM-16	-210.80	28.28	-29.97	71.93	2379
2	AM-20	-172.00	31.75	-11.42	157.90	2257
3	WATER BLANK	-244.90	4.39	-44.29	-2.49	2446
4	AM-8	-243.60	11.61	-44.35	8.98	2476
5	AM-10	-234.90	15.09	-41.09	23.21	2457
6	AM-14	-215.80	24.91	-34.11	66.20	2400
7	AM-14	-214.70	23.90	-33.66	67.91	2400
8	AM-18	-179.90	35.89	-17.49	139.60	2302
9	AM-18	-181.20	37.95	-19.90	138.70	2306
10	LWEM-21	-221.40	30.05	-25.00	10.44	2455
11	LWEM-22	-219.10	30.92	-25.59	9.70	2445
12	AM-16	-210.80	29.81	-30.49	70.39	2392
13	AM-20	-170.90	37.57	-15.80	158.10	2252

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 8 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	AM-16	-210.30	27.96	-32.42	73.68	2380
2	AM-20	-172.10	35.48	-13.00	156.30	2267
3	WATER BLANK	-263.80	-3.42	-42.58	3.05	2437
4	SP-8	-245.20	12.14	-43.19	7.81	2489
5	AM-10	-237.50	12.56	-39.58	24.91	2464
6	AM-14	-214.90	20.54	-29.85	68.52	2398
7	AM-14	-215.80	16.87	-32.75	73.04	2399
8	AM-18	-181.20	33.24	-16.89	142.30	2299
9	AM-18	-181.10	36.38	-18.19	141.20	2306
10	LWEM-21	-219.30	26.39	-26.31	14.85	2447
11	LWEM-22	-221.40	21.03	-23.92	17.41	2444
12	AM-16	-209.00	21.46	-30.36	78.33	2370
13	AM-20	-170.90	30.80	-12.32	160.90	2259

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 9 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1 AM-16	-210.80	18.26	-30.78	81.34	2388
2 AM-20	-172.00	28.63	-12.22	162.10	2255
3 WATER BLANK	-247.80	-5.77	-43.51	6.32	2444
4 AM-8	-244.40	3.38	-43.53	17.41	2469
5 AM-10	-236.70	5.90	-40.38	31.34	2459
6 AM-14	-214.70	13.60	-32.75	77.00	2396
7 AM-14	-214.60	11.33	-31.52	77.21	2397
8 AM-18	-183.00	27.39	-15.18	147.60	2304
9 AM-18	-181.10	30.85	-17.99	146.50	2302
10 LWEM-21	-220.50	21.38	-24.51	18.34	2445
11 LWEM-22	-220.60	18.25	-23.88	20.62	2450
12 AM-16	-211.40	20.25	-29.82	79.60	2386
13 AM-20	-171.40	24.00	-12.07	165.10	2251

TITLE: X-MET 840 LABORATORY NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 10 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1 AM-16	-211.80	15.26	-27.93	80.98	2378
2 AM-20	-169.70	23.15	-11.63	167.00	2242
3 WATER BLANK	-243.10	-11.74	-44.11	12.83	2422
4 AM-8	-242.90	-1.89	-43.50	20.97	2465
5 AM-10	-236.80	-1.28	-38.84	37.03	2449
6 AM-14	-215.20	10.74	-32.49	78.85	2385
7 AM-14	-215.70	5.02	-27.81	81.28	2384
8 AM-18	-181.00	20.83	-15.23	151.70	2291
9 AM-18	-181.10	24.44	-15.14	149.00	2295
10 LWEM-21	-219.30	19.36	-25.55	20.48	2446
11 LWEM-22	-221.50	10.99	-23.15	27.03	2432
12 AM-16	-210.10	6.96	-26.93	87.52	2364
13 AM-20	-169.90	18.63	-8.05	168.50	2241

TABLE: COMPILATION OF FIELD NET INTENSITIES, WATER, ALL ELEMENTS.

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.

	SAMPLE	CU CONC	Cu NET INT	AS CONC	As NET INT	HG CONC	Hg NET INT	PB CONC	Pb NET INT	BS NET INT
1	ACS-M		-216.7		74.3		-27.4		-8.6	2434.0
2	WATER BLANK		-241.4		18.8		-42.7		-16.3	2417.0
3	AM-1		-249.3		29.6		-46.6		-20.8	2499.0
4	AM-4		-250.4		34.0		-45.9		-22.8	2515.0
5	AM-6		-247.5		24.7		-44.8		-13.2	2491.0
6	AM-8		-245.9		31.6		-43.9		-10.4	2501.0
7	AM-10		-239.5		36.4		-43.9		5.2	2488.0
8	AM-12		-235.4		42.8		-39.5		6.2	2473.0
9	AM-14		-216.3		38.6		-34.7		55.8	2404.0
10	AM-16		-213.0		47.8		-33.2		55.5	2411.0
11	AM-20		-169.9		46.3		-21.4		152.1	2253.0
12	1703-1		-144.1		1276.0		-98.6		12.5	1693.0
13	WATER BLANK		-242.3		13.4		-42.9		-12.2	2422.0
14	AM-8		-245.7		32.6		-44.2		-9.9	2494.0
15	AM-10		-238.1		35.7		-41.8		4.4	2477.0
16	AM-14		-217.1		37.7		-34.4		55.6	2406.0
17	AM-14		-210.4		38.6		-34.3		55.2	2407.0
18	AM-18		-180.5		43.1		-21.8		136.7	2296.0
19	AM-18		-130.0		44.2		-22.8		135.2	2295.0
20	1703-2		-145.8		1266.0		-95.8		12.2	1695.0

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
PRE-CERTIFICATION (SOIL DAY 1 EQUIV.)

	SAMPLE	CU CONC	Cu NET INT	AS CONC	As NET INT	HG CONC	Hg NET INT	PB CONC	Pb NET INT	BS NET INT	Fe NET INT
1	SILO-2		-28.4		247.6		-30.8		-9.3	717.2	546.2
2	2-18		-49.0		40.1		-5.5		-31.5	805.4	500.0
3	PIT-2		-20.3		32.6		-3.9		-14.5	832.3	810.0
4	2-18-1		-56.2		43.7		-6.6		-35.4	864.5	391.1
5	2-18-2		-45.1		33.1		-4.1		-26.4	781.3	575.8
6	2-18-2		-53.7		42.6		-5.2		-35.0	830.8	409.1
7	2-18-3		-50.3		41.1		-3.7		-33.9	786.9	342.1
8	PIT-1		-21.5		33.6		-2.8		-19.9	839.2	712.9
9	SILO-1		-27.9		265.5		-27.6		-9.4	705.3	532.3
10	26		87.2		146.1						572.9
11	ACS-M		-217.4		84.9		-27.8		-18.8	2438.0	
12	INST BLK H2O	0.0	-241.6	0.0	24.4	0.0	-43.4	0.0	-20.4	2415.0	
13	INST BLK H2O	0.0	-240.4	0.0	17.8	0.0	-43.4	0.0	-15.3	2410.0	
14	AM-1	4.0	-251.1	1.6	35.6	2.5	-47.0	18.9	-26.3	2509.0	
15	AM-2	4.0	-251.9	1.6	36.5	2.5	-45.0	18.9	-29.2	2511.0	
16	AM-4	8.0	-251.8	3.2	40.5	5.0	-46.5	37.8	-28.1	2522.0	
17	AM-5	8.0	-250.8	3.2	37.6	5.0	-45.3	37.8	-26.7	2515.0	
18	AM-6	8.9	-249.2	3.5	35.8	5.5	-45.4	42.0	-22.3	2496.0	
19	AM-7	8.9	-250.8	3.5	37.1	5.5	-46.1	42.0	-24.1	2515.0	
20	AM-8	17.8	-244.7	7.0	34.7	11.0	-43.7	84.0	-13.0	2487.0	
21	AM-9	17.8	-245.2	7.0	41.1	11.0	-45.3	84.0	-17.1	2502.0	
22	AM-10	35.6	-240.6	14.0	48.6	22.0	-42.4	168.0	-6.1	2484.0	
23	AM-11	35.6	-237.9	14.0	39.3	22.0	-41.5	168.0	1.4	2470.0	
24	AM-12	44.5	-235.0	17.5	53.6	27.5	-43.3	210.0	-0.6	2486.0	
25	AM-13	44.5	-234.9	17.5	41.9	27.5	-41.8	210.0	7.7	2469 0	
26	AM-14	89.0	-217.6	35.0	48.5	55.0	-36.2	420.0	48.6	2413.0	
27	AM-15	89.0	-216.3	35.0	50.5	55.0	-36.2	420.0	45.8	2400.0	
28	AM-16	97.9	-212.7	38.5	52.6	60.5	-33.8	462.0	50.8	2404.0	
29	AM-17	97.9	-212.4	38.5	60.3	60.5	-35.3	462.0	46.4	2411.0	
30	AM-20	196.0	-171.9	77.0	60.7	121.0	-24.6	924.0	142.4	2269.0	
31	ACS-M		-217.9		83.0		-28.1		-16.3	2439.0	
32	AM-16		-211.3		53.7		-35.8		50.2	2408.0	
33	AM-20		-170.2		57.7		-22.1		143.4	2267.0	
34	WATER BLANK		-241.9		20.5		-43.1		-18.2	2409.0	
35	AM-8		-244.6		31.9		-44.5		-9.9	2488.0	
36	AM-10		-238.7		42.4		-43.1		-0.9	2483.0	
37	AM-14		-216.0		51.4		-34.6		42.9	2410.0	
38	AM-14		-216.4		48.6		-33.8		45.9	2405.0	
39	AM-18		-182.0		51.6		-24.1		128.6	2304.0	
40	AM-18		-179.2		54.8		-24.1		126.5	2302.0	
41	1703-3		-145.0		1271.0		-95.0		5.0	1700.0	
42	1703-4		-145.5		1265.0		-94.8		8.7	1696.0	
43	AM-16		-212.9		46.1		-33.8		58.4	2399.0	
44	AM-20		-171.4		49.4		-19.9		149.7	2265.0	
45	AM-21	196.0	-175.8	77.0	54.1	121.0	-23.6	924.0	140.3	2299.0	
46	1703-1A, DF 3:100		-250.0		84.9		-49.0		-18.6	2488.0	
47	1703-2A, DF 3:100		-246.6		191.8		-54.2		-31.3	2479.0	
48	1703-1B, DF 1:100		-254.3		77.9		-46.8		-52.4	2535.0	
49	1703-2B, DF 1:100		-253.5		80.1		-49.5		-52.9	2542.0	
50	1703-1C, DF 1:200		-254.0		58.3		-47.7		-43.4	2534.0	
51	1703-2C, DF 1:200		-255.2		60.5		-50.1		-44.3	2540.0	

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 1 AND 2 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	AM-16	-211.0	45.1	-34.7	58.3	2387.0
2	AM-20	-171.7	57.6	-20.6	143.1	2259.0
3	WATER BLANK	-238.8	16.7	-42.5	-13.3	2395.0
4	AM-8	-245.0	30.0	-43.7	-8.5	2482.0
5	AM-10	-236.5	36.3	-40.5	3.4	2458.0
6	AM-14	-213.5	40.7	-34.9	53.9	2386.0
7	AM-14	-213.3	45.1	-37.0	51.7	2398.0
8	AM-18	-178.1	55.9	-25.1	128.0	2288.0
9	AM-18	-179.0	52.7	-24.4	129.5	2286.0
10	1703-1D	-252.9	41.1	-47.2	-26.8	2512.0
11	1703-2D	-252.6	48.3	-48.0	-34.3	2535.0
12	1703-3	-255.9	38.6	-45.8	-26.7	2521.0
13	1703-4	-252.1	29.8	-47.3	-16.4	2501.0
14	1703-3U	-146.1	1264.0	-97.7	18.2	1687.0
15	1703-4U	-142.9	1274.0	-100.2	12.1	1695.0
16	AM-16	-210.3	43.2	-34.5	60.3	2386.0
17	AM-20	-172.4	54.2	-18.8	144.6	2267.0

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	86.9	146.4	17.4	97.8	668.2
2	ACS-M	-216.8	90.5	-28.9	-23.4	2423.0
3	WATER BLANK	-239.8	32.6	-42.0	-28.9	2392.0
4	AM-1	-248.2	43.8	-45.1	-34.5	2478.0
5	AM-4	-248.0	42.1	-45.2	-29.0	2489.0
6	AM-6	-245.4	40.0	-43.7	-27.2	2467.0
7	AM-8	-241.8	39.1	-43.9	-17.3	2470.0
8	AM-10	-236.7	50.9	-45.2	-7.2	2468.0
9	26, GAIN TEST.	88.6	157.0	28.9	96.9	663.0
10	AM-12	-233.5	59.6	-42.9	-7.0	2469.0
11	AM-14	-215.6	48.8	-35.9	46.2	2400.0
12	AM-16	-211.8	51.4	-37.3	53.7	2388.0
13	AM-20	-170.3	53.4	-24.4	147.0	2256.0
14	ACS-M	-214.2	70.8	-29.3	-5.8	2416.0
15	AM-16	-209.9	44.0	-36.6	59.4	2383.0
16	AM-20	-169.1	51.8	-20.6	147.0	2254.0
17	WATER BLANK	-239.5	20.3	-42.0	-17.4	2400.0
18	AM-3	-252.6	38.8	-45.1	-30.8	2508.0
19	AM-6	-246.6	34.8	-45.2	-20.3	2479.0
20	AM-8	-242.9	33.9	-45.7	-10.2	2473.0
21	AM-10	-235.3	42.2	-42.4	-1.0	2464.0
22	26, GAIN TEST	92.6	160.2	35.3	99.7	661.3
23	AM-12	-233.7	46.2	-40.0	4.0	2468.0
24	AM-14	-214.1	44.3	-35.9	51.0	2406.0
25	AM-18	-178.6	54.1	-26.5	129.0	2295.0
26	AM-16	-210.8	46.1	-32.1	55.7	2395.0
27	AM-20	-169.1	54.3	-23.4	147.5	2256.0
28	WATER BLANK	-238.7	14.4	-39.5	-13.5	2393.0
29	AM-8	-244.2	36.0	-41.5	-14.8	2484.0
30	AM-10	-236.9	40.2	-43.6	2.3	2479.0
31	AM-14	-214.4	46.7	-34.8	48.7	2400.0
32	AM-14	-215.2	44.0	-35.4	50.9	2405.0
33	AM-18	-177.0	50.7	-25.2	132.3	2295.0
34	AM-18	-178.3	52.4	-22.7	128.8	2285.0
35	26	94.7	156.0	39.1	99.2	659.1
36	1703-5	-251.1	48.2	-45.5	-33.9	2507.0
37	1703-6	-255.3	53.5	-47.1	-38.3	2537.0
38	1703-5U	-144.4	1278.0	-98.9	13.2	1697.0
39	1703-6U	-143.5	1281.0	-105.4	12.5	1690.0
40	AM-16	-209.7	46.0	-35.4	59.2	2395.0
41	AM-20	-169.4	59.7	-23.3	143.4	2272.0

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 2 CERTIFICATION AND DAY 4 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	98.8	163.1	37.3	103.2	665.5
2	ACS-M	-215.7	71.2	-28.9	-4.8	2410.0
3	AM-16	-212.2	48.4	-35.7	56.2	2388.0
4	AM-20	-170.3	55.6	-23.3	146.4	2257.0
5	AM-3	-249.0	41.8	-46.8	-31.5	2510.0
6	AM-6	-247.0	33.1	-44.3	-20.9	2491.0
7	AM-8	-244.3	34.9	-42.8	-13.9	2484.0
8	AM-10	-238.3	47.9	-43.3	-5.2	2473.0
9	AM-12	-234.9	54.5	-41.5	-2.1	2476.0
10	AM-14	-213.3	46.2	-35.8	50.9	2392.0
11	AM-18	-180.7	54.7	-27.6	130.1	2288.0
12	WATER BLANK	-239.4	20.4	-41.4	-17.1	2388.0
13	26, GAIN TEST	102.5	171.6	42.7	103.2	656.1
14	AM-8	-244.6	38.8	-43.6	-16.3	2474.0
15	AM-10	-236.6	42.1	-41.0	-2.0	2468.0
16	AM-14	-213.9	49.6	-34.5	43.7	2399.0
17	AM-14	-214.5	48.7	-35.2	47.3	2397.0
18	AM-18	-180.1	56.6	-25.8	126.0	2289.0
19	AM-18	-180.3	61.5	-30.3	125.9	2290.0
20	1703-7	-253.7	57.7	-47.2	-40.8	2519.0
21	1703-8	-254.5	54.2	-46.1	-39.1	2531.0
22	1703-7U	-141.3	1275.0	-118.0	17.5	1692.0
23	1703-8U	-140.3	1272.0	-119.6	18.5	1700.0
24	AM-16	-212.1	58.4	-34.7	46.4	2398.0
25	AM-20	-168.8	63.7	-25.8	140.7	2253.0
26	26, GAIN TEST	101.1	170.9	43.4	102.6	657.9

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 3 CERTIFICATION AND DAY 5 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	106.3	181.9	51.9	102.8	657.0
2	ACS-M	-213.1	59.5	-27.7	4.6	2394.0
3	AM-16	-209.7	30.8	-31.1	69.7	2360.0
4	AM-20	-170.1	42.1	-21.2	155.3	2238.0
5	26, GAIN TEST	109.7	185.7	51.0	105.1	654.0
6	AM-3	-248.0	19.3	-46.0	-11.7	2475.0
7	AM-6	-245.5	17.1	-44.1	-6.5	2461.0
8	AM-8	-240.2	18.0	-44.0	2.9	2443.0
9	AM-10	-237.8	25.6	-42.6	14.8	2453.0
10	AM-12	-235.2	31.7	-40.6	17.0	2460.0
11	AM-14	-213.4	27.7	-34.2	64.4	2367.0
12	AM-18	-177.7	33.5	-18.9	139.3	2257.0
13	WATER BLANK	-236.5	-2.3	-39.8	3.1	2365.0
14	26	108.1	185.2	60.5	101.4	655.7
15	AM-8	-241.3	16.4	-44.4	6.3	2449.0
16	AM-10	-236.1	23.7	-40.5	15.2	2458.0
17	26	104.7	179.2	55.6	104.6	660.0
18	AM-14	-215.5	28.3	-32.3	62.1	2380.0
19	AM-14	-213.2	33.2	-34.3	59.2	2381.0
20	AM-18	-176.0	37.9	-21.0	139.5	2259.0
21	AM-18	-179.6	36.6	-22.0	140.6	2270.0
22	1703-9	-253.8	41.8	-47.5	-26.1	2523.0
23	1703-10	-253.5	38.7	-47.3	-25.2	2524.0
24	1703-9U	-147.9	1276.0	-88.6	5.7	1699.0
25	1703-10U	-145.8	1278.0	-94.4	11.1	1698.0
26	AM-16	-208.8	23.6	-30.3	73.6	2358.0
27	AM-20	-169.2	38.9	-20.5	157.4	2240.0
28	26	108.2	186.8	53.9	101.4	657.9

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 4 CERTIFICATION AND DAY 6 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1 26	81.9	136.2	23.9	81.3	669.4
2 ACS-M	-212.2	61.7	-25.3	2.9	2393.0
3 AM-16	-210.5	32.7	-31.1	69.6	2362.0
4 AM-20	-166.2	42.3	-18.0	155.2	2228.0
5 AM-3	-245.9	20.7	-44.7	-13.5	2473.0
6 AM-6	-245.4	19.0	-44.2	-6.2	2473.0
7 AM-9	-239.9	19.5	-43.6	1.2	2466.0
8 AM-10	-236.4	23.2	-37.6	14.0	2443.0
9 AM-12	-233.9	28.2	-37.4	18.0	2445.0
10 AM-14	-213.8	22.5	-31.8	67.3	2377.0
11 AM-18	-178.7	33.5	-20.5	143.4	2275.0
12 WATER BLANK	-252.2	16.2	-46.3	-15.0	2519.0
13 26	79.7	121.9	27.9	77.7	665.1
14 AM-8	-241.9	8.6	-41.2	10.6	2448.0
15 AM-10	-236.5	24.2	-41.0	15.7	2460.0
16 AM-14	-214.1	28.0	-33.0	63.6	2385.0
17 AM-14	-212.8	26.3	-34.5	66.1	2371.0
18 AM-18	-178.3	28.8	-21.3	146.7	2272.0
19 AM-18	-178.0	35.0	-19.4	140.9	2270.0
20 1703-11	-251.5	30.1	-43.8	-19.5	2504.0
21 1703-12	-250.0	32.0	-48.1	-16.5	2501.0
22 1703-11U	-150.4	1257.0	-72.1	1.0	1695.0
23 1703-12U	-148.9	1258.0	-75.6	4.0	1687.0
24 AM-16	-208.1	34.9	-31.3	66.4	2367.0
25 AM-20	-169.5	46.1	-17.0	153.6	2246.0
26 26	84.1	129.2	30.2	80.4	664.5

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 7 EQUIVALENCY.

SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1 26	115.0	188.6	48.0	114.7	647.4
2 SCS-M	-21.5	58.4	0.9	-15.7	730.0
3 ACS-M	-212.9	61.3	-25.7	0.0	2375.0
4 AM-16	-208.5	35.2	-33.9	65.8	2358.0
5 AM-20	-169.4	47.4	-22.7	151.4	2230.0
6 WATER BLANK	-253.8	38.0	-46.8	-34.8	2536.0
7 AM-9	-242.5	31.6	-44.3	-8.7	2464.0
8 AM-10	-235.4	31.5	-40.7	7.6	2447.0
9 AM-14	-211.1	35.4	-35.6	57.8	2373.0
10 AM-14	-212.6	39.2	-34.2	53.5	2369.0
11 AM-18	-176.4	43.5	-24.6	134.9	2264.0
12 AM-18	-176.1	48.4	-24.8	133.2	2267.0
13 26	102.8	165.8	41.5	108.6	644.3
14 SCS-M	-21.0	59.3	1.9	-16.8	727.5
15 1703-13	-255.3	44.7	-47.0	-30.8	2537.0
16 1703-14	-249.8	43.5	-46.6	-28.5	2489.0
17 1703-13U	-144.4	1267.0	-98.6	11.7	1694.0
18 1703-14U	-145.5	1272.0	-96.4	9.3	1702.0
19 AM-16	-208.5	38.2	-32.7	63.4	2367.0
20 AM-20	-170.2	49.8	-21.0	149.3	2250.0
21 26	95.1	157.6	40.2	102.6	652.5
22 QA 26, SMALL PARTICLE	105.2	174.4	58.5	112.8	642.9
23 SCS-M	-21.2	57.0	0.1	-12.6	728.7
24 RABBIT FECES.	14.9	-5.2	1576.0		

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 8 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	95.5	166.0	19.2	107.2	664.3
2	SCS-M	-20.8	62.4	-1.0	-16.9	730.8
3	AM-16	-207.8	57.5	-35.0	45.3	2370.0
4	AM-20	-167.9	65.9	-27.6	137.1	2235.0
5	WATER BLANK	-254.8	59.3	-46.6	-52.2	2538.0
6	AM-9	-244.2	51.3	-43.9	-27.7	2478.0
7	AM-10	-235.6	50.0	-43.4	-6.9	2463.0
8	AM-14	-212.1	54.2	-35.3	39.2	2385.0
9	AM-14	-214.3	52.8	-36.1	43.1	2388.0
10	ACS-M	-214.7	83.6	-28.1	-17.1	2406.0
11	AM-18	-176.8	60.1	-30.6	126.0	2277.0
12	AM-18	-177.3	57.9	-26.9	123.4	2271.0
13	26	97.4	165.7	23.5	104.1	661.3
14	SCS-M	-21.2	61.3	0.6	-17.2	727.5
15	1703-15	-251.6	57.9	-48.2	-42.6	2530.0
16	1703-16	-253.6	62.0	-46.3	-46.0	2524.0
17	1703-15U	-137.1	1264.0	-126.5	21.0	1688.0
18	1703-16U	-135.7	1266.0	-129.7	23.3	1695.0
19	AM-16	-209.7	58.8	-35.0	46.9	2378.0
20	AM-20	-170.4	68.9	-26.8	136.6	2264.0
21	26	98.5	173.5	24.6	107.9	658.3
22	SCS-M	-20.9	60.0	-0.2	-15.4	727.9

TITLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
DAY 9 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	88.5	149.0	13.3	92.8	671.4
2	SCS-M	-18.9	62.8	-0.5	-18.2	731.6
3	ACS-M	-214.9	91.6	-29.6	-25.1	2406.0
4	AM-16	-208.6	61.7	-36.7	44.6	2368.0
5	AM-20	-167.6	72.6	-31.5	133.8	2253.0
6	WATER BLANK	-251.7	58.9	-47.6	-52.6	2528.0
7	AM-9	-241.7	50.0	-44.3	-25.2	2465.0
8	AM-10	-237.4	54.3	-42.3	-12.3	2458.0
9	AM-14	-215.6	54.3	-35.6	40.9	2386.0
10	AM-14	-215.2	56.1	-37.1	41.3	2389.0
11	AM-18	-178.4	64.9	-28.2	119.1	2277.0
12	AM-18	-176.4	67.8	-29.9	117.7	2285.0
13	26, GAIN TEST	88.4	152.4	17.8	98.8	664.6
14	SCS-M	-20.3	60.4	0.9	-16.6	728.1
15	1703-17	-250.6	63.9	-48.6	-44.7	2516.0
16	1703-18	-256.4	62.7	-47.1	-46.4	2546.0
17	1703-17U	-139.0	1271.0	-131.2	20.7	1701.0
18	1703-18U	-137.3	1269.0	-128.9	24.0	1689.0
19	AM-16	-209.4	49.2	-34.6	55.0	2367.0
20	AM-20	-169.3	63.8	-27.6	141.8	2249.0
21	26	89.3	154.8	20.8	95.5	661.7
22	SCS-M	-20.0	59.6	0.6	-15.7	726.7

TABLE: X-MET 840 FIELD NET INTENSITIES, WATER, ALL ELEMENTS.
rAY 10 EQUIVALENCY.

	SAMPLE	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	103.7	174.6	6.9	104.7	665.4
2	SCS-M	-20.1	62.9	-0.7	-17.9	730.4
3	ACS-M	-212.9	93.6	-30.4	-22.9	2401.0
4	AM-16	-207.6	57.4	-35.3	46.4	2368.0
5	AM-20	-167.4	70.0	-28.5	133.4	2248.0
6	WATER BLANK	-253.8	60.5	-46.8	-54.1	2534.0
7	AM-9	-244.0	51.2	-41.4	-29.2	2476.0
8	AM-10	-235.9	62.2	-41.7	-18.6	2461.0
9	AM-14	-215.1	66.6	-38.7	31.4	2403.0
10	AM-14	-213.7	71.6	-40.7	29.3	2411.0
11	AM-18	-176.8	73.9	-32.4	111.7	2268.0
12	AM-18	-174.8	61.5	-29.5	120.0	2261.0
13	26, GAIN TEST	100.2	170.6	7.7	99.9	663.4
14	SCS-M	-19.4	62.0	-0.6	-17.6	730.1
15	1703-19	-250.9	67.5	-44.9	-52.6	2508.0
16	1703-20	-248.8	60.1	-47.5	-43.9	2486.0
17	1703-19U	-135.7	1280.0	-136.3	22.9	1697.0
18	1703-20U	-135.7	1277.0	-142.6	28.1	1695.0
19	AM-16	-206.7	51.7	-35.0	52.4	2356.0
20	AM-20	-170.6	68.3	-25.9	133.7	2243.0
21	26	102.3	169.5	16.2	97.3	662.6
22	SCS-M	-19.7	58.8	1.1	-16.6	725.9
23	TENT-1	-38.9	39.3	-2.5	-31.7	722.9
24	TENT-2	-39.3	40.5	-4.0	-32.3	734.1

TABLE: COMPILATION OF FIELD CONCENTRATIONS, SOIL, ALL ELEMENTS

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
1ST PART PRE-CERTIFICATION.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
	SAMPLE	Cu	As	Hg	Pb
1	26	1279.0	1284.0	751.9	1550.0
2	SCS-M	329.4	344.7	264.6	141.5
3	SOIL BLAN	40.8	102.8	70.2	0.0
4	SOIL BLAN	36.4	69.8	123.0	0.0
5	SM-1	54.4	113.5	87.5	0.0
6	SM-2	40.4	116.0	114.1	0.0
7	SM-4	42.3	103.9	71.6	0.0
8	SM-5	40.7	96.9	121.8	0.0
9	SM-6	89.1	115.2	76.4	0.0
10	SM-7	25.0	100.8	96.7	0.0
11	SM-8	60.3	126.9	84.7	0.0
12	SM-9	106.3	90.7	112.4	0.0
13	SM-10	101.4	130.5	137.1	94.6
14	SM-11	107.9	134.1	157.8	47.5
15	SM-12	126.3	147.3	164.6	113.5
16	SM-13	133.2	133.6	134.5	166.6
17	SM-14	297.3	220.0	225.4	489.5
18	SOIL TEST	74.1	577.3	52.5	0.0
19	SM-15	285.8	210.0	233.9	476.8
20	SM-16	358.8	197.4	260.1	695.5
21	SM-17	328.3	210.0	272.8	718.5
22	SM-20	623.1	277.7	583.8	1476.0
23	SM-21	641.2	352.0	559.9	1410.0
24	SOIL BLAN	32.0	45.3	58.1	0.0
25	SM-1	45.8	63.9	83.7	0.0
26	SM-4	42.9	58.6	81.9	0.0
27	PIT-1	372.8	0.0	114.9	346.6
28	PIT-2	836.8	0.0	28.3	1301.0
29	PIT-3	0.0	85.8	61.7	0.0
30	SM-6	86.9	35.8	107.2	0.0
31	SM-8	46.3	78.4	111.5	0.0
32	SM-10	94.3	123.8	187.0	69.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
2ND PART PRE-CERTIFICATION.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	RMA 2-18	0.0	89.5	36.3	0.0
2	SM-12	117.2	100.9	178.3	186.7
3	SM-14	278.4	194.6	261.1	506.8
4	SM-16	375.0	199.5	277.2	661.7
5	RMA 2-18	26.7	31.8	78.6	0.0
6	RMA 2-18	0.0	94.8	38.6	0.0
7	SM-20	639.6	314.5	496.3	1460.0
8	SCS-M	330.7	299.2	281.7	236.8
9	SM-16	384.4	149.7	302.0	744.9
10	SM-20	677.7	304.2	484.9	1473.0
11	SOIL BLAN	32.4	39.1	78.2	0.0
12	SM-8	41.4	108.6	114.7	0.0
13	SM-10	101.3	114.6	164.4	107.3
14	SM-14	289.0	172.0	242.4	570.1
15	SM-14	281.8	158.1	225.5	599.6
16	SM-18	511.8	285.8	418.5	1119.0
17	SM-18	520.3	252.5	402.5	1223.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 1 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SILO-1	220.7	2783.0	0.0	89.6
2	SILO-2	214.1	2569.0	0.0	143.9
3	PIT-1	313.0	7.7	111.2	142.2
4	PIT-2	330.5	0.0	77.9	302.5
5	2-18-1	0.0	128.7	30.4	0.0
6	2-18-2	0.0	115.5	66.4	0.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 2 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	374.7	151.0	225.3	818.6
2	SM-20	654.5	276.8	516.0	1529.0
3	SOIL BLAN	14.9	43.8	107.7	0.0
4	SM-8	37.2	85.6	115.2	0.0
5	SM-10	108.0	120.2	149.6	135.2
6	2-8-A	34.8	10.1	114.4	0.0
7	2-8-B	224.5	0.0	141.6	518.5
8	SM-14 #1	288.6	135.4	239.1	649.3
9	SM-14 #2	291.3	166.2	252.0	549.8
10	SM-18 #1	523.6	271.8	450.4	1144.0
11	SM-18 #2	502.2	261.2	444.2	1189.0
12	SILO-3	199.4	2307.0	62.8	115.3
13	SILO-4	226.4	2515.0	3.3	94.4
14	PIT-3	418.8	0.0	123.7	409.3
15	PIT-4	472.0	0.0	162.4	277.9
16	2-18-3	0.0	30.5	33.7	0.0
17	2-18-4	0.0	34.4	64.1	0.0
18	2-8-1	373.7	0.0	219.3	1018.0
19	2-8-2	407.5	0.0	224.9	1003.0
20	SM-16	357.2	152.0	292.1	769.6
21	SM-20	659.9	271.6	515.8	1513.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1666.0	1770.0	1224.0	2186.0
2	SCS-M	324.1	339.7	272.2	146.5
3	SOIL BLAN	44.7	96.5	118.3	0.0
4	SM-1	37.4	115.2	124.8	0.0
5	SM-4	65.0	124.1	90.3	0.0
6	SM-6	93.8	153.7	107.2	0.0
7	SM-8	71.1	144.6	88.2	0.0
8	SM-10	100.4	178.5	145.0	0.0
9	SM-12	148.6	164.6	122.2	107.6
10	SM-14	311.7	236.5	218.7	462.4
11	SM-16	371.1	212.4	287.8	648.4
12	SM-20	692.5	355.3	467.6	1382.0
13	SCS-M	352.3	306.3	263.8	199.8
14	SM-16	384.0	220.4	245.4	651.5
15	SM-20	669.1	337.5	460.2	1443.0
16	SOIL BLAN	40.1	92.3	81.8	0.0
17	SM-3	89.0	84.6	99.0	0.0
18	SM-6	93.2	115.8	122.9	0.0
19	SM-8	53.7	129.4	120.4	0.0
20	SM-10	98.9	167.4	174.3	0.0
21	SM-12	156.5	150.3	157.5	112.6
22	SM-14	287.2	177.7	228.0	557.4
23	SM-18	539.5	279.7	420.9	1154.0
24	SM-14, CM	293.4	193.4	252.9	516.0
25	SM-16	373.7	168.6	257.4	743.3
26	SM-20	662.3	331.2	440.9	1457.0
27	SOIL BLAN	32.9	80.1	59.7	0.0
28	SM-8	55.9	99.3	123.8	0.0
29	SM-10	93.5	120.1	151.8	85.8
30	SM-14	301.4	168.9	220.6	573.2
31	SM-14	287.0	191.5	231.9	557.0
32	SM-18	514.9	286.4	462.5	1152.0
33	SM-18	519.1	253.2	458.1	1218.0
34	SILO-5	222.1	2439.0	0.0	169.8
35	SILO-6	180.8	2346.0	0.0	59.2
36	PIT-5	389.3	3.7	98.8	168.1
37	PIT-6	407.1	15.2	179.7	106.0
38	2-18-5	0.0	133.5	41.0	0.0
39	2-18-6	0.0	77.2	63.2	0.0
40	2-8-3	396.5	0.0	213.3	819.4
41	2-8-4	356.9	0.0	187.5	836.5
42	SM-16	371.8	160.5	252.0	778.5
45	SM-20	639.4	293.2	543.1	1473.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 4 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SM-16	361.1	178.2	236.5	780.3
2	SM-20	650.8	339.7	523.1	1369.0
3	SOIL BLAN	26.3	69.2	42.3	0.0
4	SM-8	38.4	95.9	89.8	0.0
5	SM-10	99.2	130.1	170.5	60.8
6	26	1514.0	1588.0	1238.0	1805.0
7	SM-14	258.7	189.9	268.4	506.1
8	SM-14	280.9	185.3	246.4	540.3
9	SM-18	512.6	279.1	491.4	1110.0
10	SM-18	506.3	281.5	409.0	1194.0
11	SILO-7	196.0	2484.0	0.0	167.8
12	SILO-8	179.1	2514.0	0.0	66.1
13	PIT-7	414.0	0.0	113.9	190.5
14	PIT-8	424.2	0.0	125.7	215.1
15	2-18-7	0.0	122.9	83.1	0.0
16	2-18-8	0.0	112.6	86.7	0.0
17	2-8-5	336.9	0.0	194.8	774.7
18	2-8-6	301.6	0.0	166.2	704.3
19	26	1251.0	1246.0	959.9	1489.0
20	SM-16	356.3	191.6	241.8	725.9
21	SM-20	680.5	317.0	510.1	1436.0
22	SCS-M	322.7	301.9	282.2	213.9

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 2 CERTIFICATION AND DAY 5 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1452.0	1497.0	884.4	1887.0
2	SCS-M	349.7	368.2	259.9	94.0
3	SM-16	375.0	207.3	240.7	687.9
4	SM-20	659.7	346.4	442.5	1417.0
5	SM-3	82.6	129.2	98.7	0.0
6	SM-6	91.3	110.7	52.7	0.0
7	SM-8	53.9	146.5	96.9	0.0
8	SM-10	97.6	197.6	166.6	0.0
9	SM-12	135.5	191.0	181.2	10.7
10	SM-14	295.0	220.9	221.6	486.6
11	SM-18	499.0	307.2	415.6	1085.0
12	SOIL BLAN	20.4	90.6	93.5	0.0
13	26, GAIN	1481.0	1536.0	1088.0	1734.0
14	SM-8	77.4	114.0	101.1	0.0
15	SM-10	93.1	156.5	169.5	0.0
16	SM-14	268.0	210.8	280.1	451.5
17	SM-14	294.3	221.2	231.2	464.6
18	SM-18	511.5	315.7	445.2	1049.0
19	SM-18	505.3	318.5	355.7	1113.0
20	SILO-9	253.4	2560.0	0.0	188.1
21	SILO-10	229.9	2342.0	0.0	135.3
22	26, GAIN	1477.0	1544.0	1105.0	1771.0
23	PIT-9	403.1	0.0	131.9	158.3
24	PIT-10	370.6	1.1	128.6	132.3
25	2-18-9	0.0	108.8	55.4	0.0
26	2-18-10	0.0	108.5	64.4	0.0
27	2-8-7	372.6	0.0	218.3	851.8
28	2-8-8	356.1	0.0	162.6	901.3
29	SM-16	384.8	184.5	245.0	761.0
30	SM-20	663.5	322.4	498.3	1496.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 3 CERTIFICATION AND DAY 6 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1324.0	1277.0	765.2	1612.0
2	SCS-M	310.7	305.3	276.8	201.2
3	SM-16	360.8	211.8	256.3	667.9
4	SM-20	631.8	310.6	593.2	1383.0
5	26, GAIN	1314.0	1267.0	797.4	1546.0
6	SM-3	58.7	70.0	120.1	0.0
7	SM-6	64.2	59.8	107.9	0.0
8	SM-8	51.1	114.6	87.8	0.0
9	SM-10	84.2	148.9	208.2	1.6
10	SM-12	129.0	118.8	196.4	153.1
11	SM-14	279.6	186.1	248.9	530.3
12	SM-18	510.0	292.0	455.6	1150.0
13	SOIL BLAN	7.8	46.7	90.2	0.0
14	26, GAIN	1378.0	1324.0	829.3	1586.0
15	SM-8	72.8	35.0	55.5	11.6
16	SM-10	86.5	139.9	149.9	51.0
17	SM-14	282.2	189.3	258.3	511.9
18	SM-14	293.4	175.3	263.4	531.8
19	SM-18	515.3	272.6	430.9	1143.0
20	SM-18	507.9	281.2	390.3	1179.0
21	SILO-11	209.0	2471.0	0.0	86.0
22	SILO-12	203.0	2687.0	0.4	110.5
23	PIT-11	452.6	0.0	138.0	279.5
24	PIT-12	516.9	0.0	130.1	320.2
25	2-18-11	0.0	84.2	61.1	0.0
26	2-18-12	0.0	93.8	45.0	0.0
27	2-8-9	343.7	0.0	199.6	781.3
28	2-8-10	360.9	0.0	180.7	841.2
29	SM-16	345.9	167.7	260.1	735.9
30	SM-20	617.0	283.1	574.0	1447.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 4 CERTIFICATION AND DAY 7 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1328.0	1299.0	988.8	1462.0
2	SCS-M	327.9	280.7	261.0	305.5
3	SM-16	359.5	168.2	278.0	753.7
4	SM-20	630.5	276.4	572.6	1457.0
5	SM-3	51.3	43.2	62.9	0.0
6	SM-6	63.9	41.3	117.6	0.0
7	SM-8	50.9	83.5	58.2	18.7
8	SM-10	90.9	130.7	195.8	48.8
9	SM-12	111.5	128.8	187.5	153.4
10	SM-14	274.2	163.0	267.3	569.8
11	SM-18	507.7	257.0	457.6	1201.0
12	SOIL BLAN	30.9	23.3	52.8	0.0
13	26	1219.0	1195.0	819.7	1355.0
14	SM-8	58.2	66.2	70.2	29.2
15	SM-10	103.9	104.5	161.9	132.0
16	SM-14	253.5	156.2	303.1	578.3
17	SM-14	286.9	150.1	238.7	615.6
18	SM-18	506.5	270.7	446.2	1188.0
19	SM-18	503.2	247.8	426.6	1232.0
20	SILO-13	225.4	2464.0	0.0	231.3
21	SILO-14	213.9	2479.0	11.6	98.9
22	PIT-13	400.6	0.0	115.5	183.8
23	PIT-14	407.3	0.0	114.3	201.7
24	2-18-13	0.0	98.0	80.1	0.0
25	2-18-14	0.0	64.5	54.5	0.0
D	2-8-11	355.3	0.0	231.6	861.5
27	2-18-12	339.6	0.0	175.6	828.1
28	SM-16	349.1	177.3	263.0	737.8
29	SM-20	626.6	265.2	585.3	1462.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 8 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1507.0	1552.0	998.4	1801.0
2	SCS-M	321.2	333.5	253.4	174.2
3	SM-16	367.1	221.0	288.2	632.3
4	SM-20	672.1	351.0	530.9	1387.0
5	SOIL BLAN	39.2	80.4	103.8	0.0
6	SM-8	60.0	149.8	126.1	0.0
7	SM-10	110.4	171.6	164.6	0.0
8	SM-14	288.5	206.7	292.8	433.0
9	SM-14	307.0	205.1	227.8	519.1
10	SM-18	504.8	286.0	421.7	1113.0
11	SM-18	509.6	281.8	414.2	1161.0
12	26	1786.0	1964.0	1678.0	2142.0
13	SCS-M	320.4	318.1	271.3	195.9
14	SILO-15	226.8	2332.0	0.0	68.7
15	SILO-16	232.8	2716.0	0.0	139.1
16	PIT-15	425.6	0.0	141.4	175.9
17	PIT-16	409.8	0.0	170.3	169.1
18	2-18-15	0.0	122.5	76.9	0.0
19	2-18-16	0.0	104.4	85.3	0.0
20	2-8-13	401.8	0.0	180.8	868.4
21	2-8-14	373.5	0.0	179.6	836.2
22	SM-16	344.3	206.2	270.6	651.4
23	SM-20	636.8	329.6	498.6	1437.0
24	RABBIT FE	0.0	0.0	0.0	1884.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 9 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1408.0	1495.0	473.7	2104.0
2	SCS-M	340.5	422.8	210.7	15.6
3	SM-16	387.1	316.2	201.7	496.5
4	SM-20	685.1	409.8	355.6	1364.0
5	SOIL BLAN	29.3	178.9	86.4	0.0
6	SM-8	85.8	195.5	74.9	0.0
7	SM-10	127.8	233.8	150.5	0.0
8	SM-14	292.6	289.0	207.4	339.2
9	SM-14	308.2	303.3	248.3	290.1
10	SM-18	545.8	363.6	315.2	1069.0
11	SM-18	527.3	359.0	346.8	1022.0
12	26, GAIN	1423.0	1532.0	637.1	1931.0
13	SCS-M	350.5	392.5	246.1	38.7
14	SILO-17	248.6	2468.0	0.0	240.7
15	SILO-18	269.9	2661.0	0.0	82.4
16	PIT-17	428.2	53.4	141.7	53.5
17	PIT-18	415.3	53.5	198.3	22.3
18	2-18-17	0.0	171.1	66.4	0.0
19	2-18-18	0.0	172.4	86.4	0.0
20	2-8-15	381.0	22.4	165.5	671.2
21	2-18-16	394.8	17.7	204.6	749.5
22	SM-16	352.8	221.5	229.9	648.3
23	SM-20	666.7	374.8	464.7	1363.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 10 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1265.0	1265.0	373.6	1707.0
2	SCS-N	339.1	401.5	217.4	29.4
3	SN-16	399.5	287.4	197.6	544.7
4	SN-20	674.0	387.5	342.7	1406.0
5	SOIL BLAN	46.0	158.1	93.5	0.0
6	SN-8	75.3	197.9	87.9	0.0
7	SN-10	119.7	221.7	157.4	0.0
8	SN-14	317.7	244.5	225.8	415.2
9	SN-14	292.0	264.0	243.6	382.9
10	SN-18	530.3	373.6	342.7	1027.0
11	SN-18	526.2	367.5	329.4	1071.0
12	26, GAIN	1354.0	1408.0	574.8	1694.0
13	SCS-N	356.4	354.5	277.4	113.9
14	SILO-19	264.7	2529.0	0.0	217.3
15	SILO-20	225.7	2559.0	0.0	121.3
16	PIT-19	441.7	47.1	171.1	16.6
17	PIT-20	426.2	56.1	156.2	47.6
18	2-18-19	0.0	182.0	62.4	0.0
19	2-18-20	0.0	203.5	68.5	0.0
20	2-8-17	323.5	56.8	190.1	531.9
21	2-8-18	400.9	0.0	196.7	747.0
22	SN-16	396.7	261.2	168.4	689.9
23	SN-20	653.9	321.7	413.6	1459.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WET SOIL, ALL ELEMENTS.
DAY 11 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1116.0	1111.0	261.3	1594.0
2	SCS-N	356.5	412.3	236.7	0.0
3	SN-16	398.3	283.1	225.0	531.6
4	SN-20	683.4	398.5	350.6	1379.0
5	SOIL BLAN	64.6	166.8	114.5	0.0
6	SN-8	64.9	183.9	122.1	0.0
7	SN-10	112.8	254.6	139.4	0.0
8	SN-14	318.3	287.8	188.9	371.1
9	SN-14	298.7	265.4	261.8	401.8
10	SN-18	532.1	366.0	353.5	1003.0
11	SN-18	531.4	350.4	339.1	1041.0
12	26, GAIN	1394.0	1405.0	479.5	1683.0
13	SCS-N	334.2	379.7	223.0	94.3
14	2-8-19	374.2	36.4	190.1	755.1
15	2-8-20	343.1	48.9	190.8	597.4
16	SILO-21	[illegible]	2521.0	0.0	195.1
17	SILO-22	270.4	2607.0	0.0	277.7
18	PIT-21	426.2	44.6	121.6	24.3
19	PIT-22	445.9	[illegible]	158.6	3.8
20	2-18-21	0.0	185.9	92.8	0.0
21	2-18-22	0.0	205.7	81.5	0.0
22	SN-16	386.3	266.2	249.9	550.1
23	SN-20	678.8	386.0	441.1	1366.0

TABLE: COMPILATION OF FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
	SAMPLE	Cu	As	Hg	Pb
1	ACS-M	99.8	172.3	74.0	0.0
2	INST. BLA	0.0	26.6	7.9	0.0
3	AM-1	0.0	54.8	0.0	0.0
4	AM-4	0.5	66.3	0.0	0.0
5	AM-6	0.7	42.0	0.0	0.0
6	AM-8	11.6	60.2	2.9	0.0
7	AM-10	29.2	72.8	2.6	48.5
8	AM-12	40.2	89.4	21.2	55.4
9	AM-14	79.9	78.5	40.9	380.4
10	AM-16	97.9	102.6	47.3	378.2
11	AM-20	194.3	98.7	99.1	1011.0
12	1703-1	16.4	3328.0	0.0	96.7
13	WATER BLA	0.0	12.3	7.1	0.0
14	AM-8	9.0	62.2	1.9	0.0
15	AM-10	30.9	70.9	11.7	43.7
16	AM-14	79.8	76.1	42.4	378.7
17	AM-14	82.7	78.4	42.8	376.3
18	AM-18	173.6	90.3	97.4	910.2
19	AM-18	174.4	93.1	93.0	900.3
20	1703-2	12.9	[illegible]	0.0	94.8
21	SILO 1	258.8	640.4	92.0	0.0
22	SILO-2	259.6	649.7	80.9	0.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
PRE-CERTIFICATION.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	SILO-2	214.1	2569.0	0.0	143.9
2	2-18	0.0	85.1	50.7	0.0
3	PIT-2	330.5	0.0	77.9	302.5
4	2-18-1	0.0	128.7	30.4	0.0
5	2-18-2	0.0	0.8	74.1	0.0
6	2-18-2	0.0	115.5	66.4	0.0
7	2-18-3	0.0	96.7	105.9	0.0
8	PIT-1	313.0	7.7	111.2	142.2
9	SILO-1	220.7	2783.0	0.0	89.6
10	26	1357.0	1363.0	875.1	1713.0
11	ACS-M	98.7	200.2	72.4	0.0
12	INSTRUMEN	0.0	41.2	4.9	0.0
13	INSTRUMEN	0.0	23.8	4.8	0.0
14	AM-1	0.0	70.7	0.0	0.0
15	AM-2	0.0	72.9	0.0	0.0
16	AM-4	0.0	83.6	0.0	0.0
17	AM-5	0.0	76.0	0.0	0.0
18	AM-6	0.0	71.1	0.0	0.0
19	AM-7	0.0	74.5	0.0	0.0
20	AM-8	9.5	68.4	4.0	0.0
21	AM-9	13.6	85.1	0.0	0.0
22	AM-10	24.3	104.8	9.3	0.0
23	AM-11	28.4	80.4	13.0	23.6
24	AM-12	44.7	117.9	5.0	11.0
25	AM-13	38.0	87.2	11.2	65.2
26	AM-14	79.8	104.6	34.8	333.3
27	AM-15	78.5	109.7	34.3	314.7
28	AM-16	95.5	115.4	44.8	347.4
29	AM-17	98.5	135.5	38.3	318.8
30	AM-20	191.2	136.6	84.4	948.0
31	ACS-M (EN	97.0	195.2	71.2	0.0
32	AM-16	100.9	118.2	33.8	343.3
33	AM-20	198.0	128.6	95.6	954.3
34	WATER BLA	0.0	31.1	6.0	0.0
35	AM-8	9.7	60.8	0.5	0.0
36	AM-10	30.3	88.6	6.1	8.4
37	AM-14 #1	85.2	112.1	41.6	295.4
38	AM-14 #2	82.6	104.8	45.1	315.2
39	AM-18 #1	170.1	112.6	87.2	857.3
40	AM-18 #2	179.5	121.0	87.1	843.9
41	1703-3	19.8	3314.0	0.0	47.1
42	1703-4	15.4	3298.0	0.0	71.6
43	AM-16	92.6	98.2	44.8	397.0
44	AM-20	194.6	106.9	106.2	995.8
45	AM-21	190.5	119.2	89.1	933.8
46	1703-1A,	0.0	200.0	0.0	0.0
47	1703-2A,	0.0	480.9	0.0	0.0
48	1703-1B,	0.0	181.7	0.0	0.0
49	1703-2B,	0.0	187.5	0.0	0.0
50	1703-1C,	0.0	130.2	0.0	0.0
51	1703-2C,	0.0	135.9	0.0	0.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAYS 1 AND 2 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	AM-16	93.1	95.6	40.6	396.6
2	AM-20	190.6	128.3	103.0	952.4
3	WATER BLA	0.0	20.9	8.6	0.0
4	AM-8	6.3	55.9	3.9	0.0
5	AM-10	28.4	72.3	16.9	36.6
6	AM-14	83.3	84.0	40.0	367.6
7	AM-14	88.0	95.7	30.7	353.5
8	AM-18	176.2	124.0	82.4	853.4
9	AM-18	172.7	115.6	85.5	863.0
10	1703-1D	0.0	85.0	0.0	0.0
11	1703-2D	0.7	104.0	0.0	0.0
12	1703-3	0.0	78.6	0.0	0.0
13	1703-4	0.0	55.3	0.0	0.0
14	1703-3U	8.3	3298.0	0.0	134.0
15	1703-4U	20.3	3324.0	0.0	93.6
16	AM-16	95.4	90.6	41.6	410.0
17	AM-20	192.9	119.3	111.1	962.1

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1341.0	1364.0	689.3	1787.0
2	ACS-M	93.1	214.9	67.4	0.0
3	WATER BLA	0.0	62.9	10.6	0.0
4	AM-1	0.0	92.1	0.0	0.0
5	AM-4	0.0	87.8	0.0	0.0
6	AM-6	0.0	82.2	3.9	0.0
7	AM-8	12.1	79.8	3.1	0.0
8	AM-10	28.7	110.8	0.0	0.0
9	26, GAIN	1373.0	1471.0	919.2	1706.0
10	AM-12	42.8	133.5	6.7	0.0
11	AM-14	81.4	105.4	35.8	317.5
12	AM-16	88.7	112.1	29.5	366.5
13	AM-20	191.2	117.5	85.1	977.8
14	ACS-M	98.8	163.0	65.5	0.0
15	AM-16	94.1	92.6	32.4	404.2
16	AM-20	198.1	113.1	102.5	977.9
17	WATER BLA	0.0	30.4	10.9	0.0
18	AM-3	0.0	79.1	0.0	0.0
19	AM-6	0.0	68.5	0.0	0.0
20	AM-8	8.2	66.2	0.0	0.0
21	AM-10	34.0	87.8	8.7	8.2
22	26, GAIN	1425.0	1499.0	1041.0	1740.0
23	AM-12	43.7	98.5	19.2	40.9
24	AM-14	89.5	93.6	35.7	348.9
25	AM-18	176.5	119.2	75.8	859.7
26	AM-16	99.3	98.3	52.3	379.8
27	AM-20	196.3	119.8	89.8	981.2
28	WATER BLA	0.0	15.1	21.3	0.0
29	AM-8	12.1	71.6	13.1	0.0
30	AM-10	34.4	82.6	3.7	29.9
31	AM-14	86.7	99.8	40.4	333.8
32	AM-14	85.6	92.6	38.0	348.2
33	AM-18	183.2	110.2	81.8	881.4
34	AM-18	176.3	114.8	93.4	858.7
35	26, GAIN	1452.0	1453.0	1103.0	1718.0
36	1703-5	0.0	103.8	0.0	0.0
37	1703-6	0.0	117.6	0.0	0.0
38	1703-5U	16.3	3334.0	0.0	101.3
39	1703-6U	12.3	3341.0	0.0	96.3
40	AM-16	100.8	97.9	37.6	402.2
41	AM-20	202.6	133.9	90.3	954.6

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 2 CERTIFICATION AND DAY 4 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26, GAIN	1495.0	1524.0	1085.0	1807.0
2	ACS-M	91.0	164.2	66.9	0.0
3	AM-16	88.4	104.1	36.5	382.9
4	AM-20	192.7	123.0	90.0	973.7
5	AM-3	2.5	86.9	0.0	0.0
6	AM-6	2.9	64.2	1.5	0.0
7	AM-8	10.6	68.9	7.7	0.0
8	AM-10	26.6	103.0	5.4	0.0
9	AM-12	41.9	120.4	12.7	0.7
10	AM-14	86.5	98.5	36.2	348.1
11	AM-18	164.7	120.8	70.8	867.0
12	WATER BLA	0.0	30.8	13.1	0.0
13	26, GAIN	1541.0	1609.0	1199.0	1774.0
14	AM-8	4.3	79.0	4.4	0.0
15	AM-10	32.4	87.7	14.8	1.5
16	AM-14	87.9	107.4	41.9	301.2
17	AM-14	84.7	104.9	38.6	324.3
18	AM-18	169.0	125.7	79.2	840.5
19	AM-18	164.8	138.5	58.7	839.8
20	1703-7	0.0	128.7	0.0	0.0
21	1703-8	0.0	119.3	0.0	0.0
22	1703-7U	10.7	3326.0	0.0	129.2
23	1703-8U	15.8	3316.0	0.0	135.7
24	AM-16	93.9	130.5	40.8	318.6
25	AM-20	194.5	144.4	78.8	936.9
26	26, GAIN	1527.0	1603.0	1210.0	1758.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 3 CERTIFICATION AND DAY 5 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1594.0	1713.0	1388.0	1713.0
2	ACS-M	94.4	133.3	72.5	44.7
3	AM-16	88.5	58.0	56.4	471.2
4	AM-20	187.1	87.8	99.9	1032.0
5	26, GAIN	1630.0	1748.0	1380.0	1764.0
6	AM-3	0.0	27.9	0.0	0.0
7	AM-6	0.0	22.1	2.4	0.0
8	AM-8	5.4	24.4	2.4	33.8
9	AM-10	19.7	44.3	8.2	111.7
10	AM-12	34.0	60.4	16.4	126.3
11	AM-14	75.6	49.9	43.2	436.6
12	AM-18	169.3	65.0	110.8	927.5
13	WATER BLA	0.0	0.0	20.1	35.1
14	26	1623.0	1748.0	1551.0	1642.0
15	AM-8	3.8	20.3	0.5	55.7
16	AM-10	29.9	39.3	16.7	114.5
17	26	1580.0	1684.0	1450.0	1743.0
18	AM-14	75.6	51.4	51.5	421.8
19	AM-14	82.9	64.4	42.5	402.3
20	AM-18	174.7	76.5	100.9	928.6
21	AM-18	165.7	73.2	96.3	936.1
22	1703-9	0.0	86.8	0.0	0.0
23	1703-10	0.0	78.7	0.0	0.0
24	1703-9U	12.9	3328.0	0.0	52.1
25	1703-10U	15.2	3333.0	0.0	87.3
26	AM-16	91.6	39.2	59.9	497.2
27	AM-20	191.9	79.3	102.8	1046.0
28	26	1617.0	1764.0	1433.0	1666.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 4 CERTIFICATION AND DAY 6 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1294.0	1284.0	786.6	1400.0
2	ACS-M	99.1	139.2	83.2	33.8
3	AM-16	86.5	62.9	56.7	470.8
4	AM-20	199.4	88.1	114.8	1032.0
5	AM-3	0.0	31.4	0.0	0.0
6	AM-6	0.4	26.9	1.9	0.0
7	AM-9	17.7	28.4	4.1	22.5
8	AM-10	24.4	38.2	29.6	106.7
9	AM-12	34.4	51.3	30.1	132.6
10	AM-14	80.5	36.1	53.9	455.8
11	AM-18	171.9	65.0	103.5	954.1
12	WATER BLA	0.0	19.6	0.0	0.0
13	26	1274.0	1146.0	829.4	1322.0
14	AM-8	3.7	0.0	14.1	83.9
15	AM-10	29.0	40.7	15.0	117.5
16	AM-14	82.2	50.7	48.3	431.3
17	AM-14	79.4	46.3	41.4	448.1
18	AM-18	171.5	52.9	99.7	975.7
19	AM-18	173.7	69.2	108.8	937.8
20	1703-11	0.0	56.2	4.1	0.0
21	1703-12	0.0	61.1	0.0	0.0
22	1703-11U	15.6	3278.0	0.2	21.0
23	1703-12U	15.5	3280.0	0.0	40.9
24	AM-16	97.4	68.7	55.4	449.6
25	AM-20	195.6	98.2	119.4	1021.0
26	26	1326.0	1215.0	885.6	1364.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 7 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1685.0	1764.0	1331.0	1990.0
2	SCS-M	312.8	304.6	279.3	194.1
3	ACS-M	87.8	138.2	81.5	14.5
4	AM-16	89.7	69.4	44.3	445.6
5	AM-2C	184.7	101.6	92.7	1007.0
6	WATER BLA	0.0	77.0	0.0	0.0
7	AM-9	6.4	60.2	1.2	0.0
8	AM-10	27.2	59.9	16.0	64.2
9	AM-14	85.6	70.2	36.7	393.6
10	AM-14	79.6	80.1	43.2	365.4
11	AM-18	172.3	91.4	84.3	898.5
12	AM-18	174.7	104.2	83.7	887.5
13	26	1543.0	1544.0	1167.0	1909.0
14	SCS-M	319.9	315.1	307.7	158.2
15	1703-13	0.0	94.6	0.0	0.0
16	1703-14	0.0	91.4	0.0	0.0
17	1703-13U	15.6	3303.0	0.0	91.0
18	1703-14U	16.2	3318.0	0.0	75.9
19	AM-16	94.6	77.4	49.5	430.2
20	AM-20	191.6	107.8	100.6	993.0
21	QA-26, LA	1458.0	1469.0	1126.0	1788.0
22	QA 26, SM	1590.0	1624.0	1492.0	1924.0
23	SCS-M	318.1	287.7	252.4	289.0
24	RABBIT FE	ERR	ERR	0.0	0.0

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 8 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1437.0	1547.0	762.0	1969.0
2	SCS-M	323.1	352.4	235.2	161.7
3	AM-16	96.7	128.1	39.4	311.6
4	AM-20	188.4	150.1	70.4	913.0
5	WATER BLA	0.0	132.8	0.0	0.0
6	AM-9	7.0	111.9	3.3	0.0
7	AM-10	31.5	108.5	4.7	0.0
8	AM-14	87.7	119.4	38.3	271.2
9	AM-14	80.8	115.8	34.7	297.2
10	ACS-M	93.5	196.7	70.8	0.0
11	AM-18	171.8	134.9	57.4	840.4
12	AM-18	170.5	129.3	74.0	823.6
13	26 QA	1463.0	1549.0	838.1	1879.0
14	SCS-M	316.8	338.3	277.4	148.2
15	1703-15	1.8	129.2	0.0	0.0
16	1703-16	0.0	140.0	0.0	0.0
17	1703-15U	17.8	3298.0	0.0	152.3
18	1703-16U	22.4	3301.0	0.0	167.0
19	AM-16	93.7	131.5	39.6	321.7
20	AM-20	192.5	158.1	74.0	909.9
21	26 QA	1476.0	1622.0	875.7	1952.0
22	SCS-M	167.1	214.6	155.6	244.6
23	SCS-M	322.5	323.9	250.3	204.9

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 9 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1354.0	1396.0	619.1	1687.0
2	SCS-M	356.7	356.8	248.4	122.7
3	ACS-M	91.8	217.8	64.4	0.0
4	AM-16	91.9	139.0	32.0	306.7
5	AM-20	194.9	167.7	52.7	891.6
6	WATER BLA	1.0	131.8	0.0	0.0
7	AM-9	9.8	108.4	1.4	0.0
8	AM-10	23.8	119.7	9.5	0.0
9	AM-14	75.0	119.8	37.2	282.6
10	AM-14	76.9	124.5	30.7	285.2
11	AM-18	167.8	147.6	68.0	794.9
12	AM-18	177.0	155.2	60.3	786.3
13	26, GAIN	1358.0	1423.0	708.6	1801.0
14	SCS-M	331.1	328.1	284.3	165.9
15	1703-17	0.0	144.9	0.0	0.0
16	1703-18	0.0	141.9	0.0	0.0
17	1703-17U	11.4	3315.0	0.0	150.1
18	1703-18U	15.3	3309.0	0.0	171.7
19	AM-16	90.2	106.4	41.1	374.7
20	AM-20	189.4	144.8	70.4	943.9
21	26	1371.0	1451.0	767.4	1709.0
22	SCS-M	334.6	318.0	271.9	193.1

TITLE: X-MET 840 FIELD CONCENTRATIONS, WATER, ALL ELEMENTS.
DAY 10 EQUIVALENCY.

	SAMPLE	Cu ppm	As ppm	Hg ppm	Pb ppm
1	26	1512.0	1637.0	555.3	1949.0
2	SCS-M	333.7	357.5	243.3	131.9
3	ACS-M	95.7	223.0	60.4	0.0
4	AM-16	96.3	127.9	38.0	318.6
5	AM-20	195.2	161.0	66.3	888.6
6	WATER BLA	0.0	136.0	0.0	0.0
7	AM-9	9.3	111.7	13.9	0.0
8	AM-10	31.2	140.6	12.1	0.0
9	AM-14	82.3	152.1	23.6	220.1
10	AM-14	89.2	165.1	15.0	206.5
11	AM-18	166.5	171.1	49.5	746.4
12	AM-18	173.3	138.7	62.0	801.3
13	26, GAIN	1475.0	1603.0	562.0	1843.0
14	SCS-M	343.9	347.2	245.7	139.8
15	1703-19	0.0	154.3	0.0	0.0
16	1703-20	0.0	134.9	0.0	0.0
17	1703-19U	18.1	3340.0	0.0	164.8
18	1703-20U	12.3	3331.0	0.0	198.9
19	AM-16	94.7	112.9	39.1	357.9
20	AM-20	183.3	156.5	78.1	890.5
21	26	1508.0	1596.0	714.9	1749.0
22	SCS-M	338.6	309.3	284.2	166.8
23	TENT-1	61.9	75.7	134.3	0.0
24	TENT-2	56.0	89.9	94.7	0.0

TABLE: COMPILATION OF FIELD NET INTENSITIES, SOIL, ALL ELEMENTS.

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
1ST PART PRE-CERTIFICATION.

	SAMPLE	Fe NET INT	CU CONC	Cu NET INT	AS CONC	As NET INT	HG CONC	Hg NET INT	PB CONC	Pb NET INT	BS NET INT
1	26	573.1		80.7		137.0		21.9		87.6	673.6
2	SCS-M	596.1		-20.4		61.8		0.1		-17.5	727.6
3	SOIL BL	541.2	0.0	-40.3	0.0	41.6	0.0	-5.0	0.0	-33.4	736.0
4	SOIL BL	543.1	0.0	-40.7	0.0	38.8	0.0	-2.9	0.0	-32.2	736.8
5	SM-1	532.4	9.4	-39.4	4.5	42.5	10.9	-4.4	22.5	-32.8	742.3
6	SM-2	516.3	9.4	-40.4	4.5	42.7	10.9	-3.5	22.5	-33.7	742.6
7	SM-4	543.0	18.8	-40.2	9.0	41.7	21.8	-4.9	45.0	-30.6	746.7
8	SM-5	507.0	18.8	-40.4	9.0	41.1	21.8	-3.1	45.0	-31.6	750.2
9	SM-6	570.4	25.0	-37.0	12.0	42.6	29.0	-4.9	60.0	-30.6	736.4
10	SM-7	508.9	25.0	-41.4	12.0	41.4	29.0	-4.0	60.0	-29.2	741.9
11	SM-8	488.4	50.0	-39.0	24.0	43.6	58.0	-4.6	120.0	-28.7	735.7
12	SM-9	536.2	50.0	-35.8	24.0	40.6	58.0	-3.4	120.0	-25.3	732.0
13	SM-10	473.4	100.0	-36.2	48.0	43.9	116.0	-2.8	240.0	-20.9	737.1
14	SM-11	515.8	100.0	-35.7	48.0	44.2	116.0	-2.1	240.0	-22.4	731.3
15	SM-12	490.1	125.0	-34.4	60.0	45.3	145.0	-2.0	300.0	-20.0	734.1
16	SM-13	513.3	125.0	-34.0	60.0	44.1	145.0	-2.9	300.0	-18.3	737.7
17	SM-14	517.2	250.0	-22.6	120.0	51.4	290.0	-0.4	600.0	-5.9	727.2
18	SOIL TE	505.3		-38.0		81.2		-9.1		-26.4	725.3
19	SM-15	522.8	250.0	-23.4	120.0	50.5	290.0	0.0	600.0	-6.3	726.1
20	SM-16	612.4	312.0	-18.3	150.0	49.5	362.0	1.0	750.0	1.6	715.1
21	SM-17	519.1	312.0	-20.5	150.0	50.5	362.0	1.4	750.0	2.5	723.7
22	SM-20	586.8	625.0	0.0	300.0	56.2	725.0	11.9	1500.0	31.7	710.2
23	SM-21	496.9	625.0	1.2	300.0	62.4	725.0	10.5	1500.0	29.5	712.3
24	SOIL BL	540.7		-41.0		36.8		-5.0		-28.2	734.9
25	SM-1	532.1		-40.0		38.3		-4.2		-29.4	739.7
26	SM-4	536.6		-40.2		37.9		-4.2		-27.8	741.9
27	PIT-1	845.8		-17.4		27.3		-2.1		-13.0	805.2
28	PIT-2	2008.0		14.7		-5.8		-2.3		18.7	725.5
29	PIT-3	467.2		-45.1		40.2		-5.2		-31.1	772.5
30	SM-6	580.8		-37.2		36.0		-3.2		-25.8	733.1
31	SM-8	487.4		-40.0		39.5		-3.3		-25.5	732.5
32	SM-10	470.2		-36.7		43.3		-1.0		-21.6	734.7

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
2ND PART PRE-CERTIFICATION.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	RMA 2-1	498.8	-49.2	40.5	-6.1	-31.9	802.4
2	SM-12	496.5	-35.1	41.4	-1.1	-17.5	733.4
3	SM-14	518.4	-23.9	49.2	1.1	-5.2	725.7
4	SM-16	615.3	-17.2	49.7	1.6	0.5	718.1
5	RMA 2-1	638.2	-41.3	35.6	-4.2	-27.8	763.5
6	RMA 2-1	344.6	-50.8	40.9	-6.0	-32.4	791.5
7	SM-20	586.1	1.1	59.3	8.5	30.8	708.4
8	SCS-M	599.4	-20.3	58.0	1.1	-14.2	724.3
9	SM-16	620.9	-16.6	45.5	2.9	3.3	715.4
10	SM-20	591.9	3.7	58.4	8.2	31.1	704.0
11	SOIL BL	542.1	-40.9	36.3	-4.2	-29.0	733.6
12	SM-8	481.5	-40.3	42.1	-3.5	-27.0	734.4
13	SM-10	469.9	-36.2	42.6	-1.7	-20.4	729.9
14	SM-14	518.1	-23.2	47.4	0.6	-3.1	721.9
15	SM-14	519.4	-23.7	46.2	0.1	-2.2	723.6
16	SM-18	490.2	-7.8	56.9	6.0	18.0	715.9
17	SM-18	494.9	-7.2	54.1	5.7	21.5	716.2

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 1 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SILO-1	532.3	-27.9	265.5	-27.6	-9.4	705.3
2	SILO-2	546.2	-28.4	247.6	-30.8	-9.3	717.2
3	PIT-1	712.9	-21.5	33.6	-2.8	-19.9	839.2
4	PIT-2	810.0	-20.3	32.6	-3.9	-14.5	832.3
5	2-18-1	391.1	-56.2	43.7	-6.6	-35.4	864.5
6	2-18-2	409.1	-53.7	42.6	-5.2	-35.0	830.8

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 2 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SW-16	616.8	-17.2	45.6	0.2	5.5	715.0
2	SW-20	586.4	2.1	56.1	9.5	33.2	704.9
3	SOIL BL	540.1	-42.1	36.6	-3.2	-30.0	734.8
4	SW-8	480.7	-40.6	40.1	-3.3	-26.0	735.8
5	SW-10	466.2	-35.7	43.0	-2.3	-19.4	732.2
6	2-8-A,	515.1	-40.8	33.8	-2.7	-27.1	711.0
7	2-8-B,	695.3	-27.6	30.5	-1.5	-6.5	682.0
8	SW-14	520.2	-23.2	44.3	0.8	-0.5	720.6
9	SW-14	518.5	-23.0	46.9	1.0	-3.8	722.4
10	SW-18	488.6	-6.9	55.7	7.2	19.0	716.1
11	SW-18	492.5	-8.4	54.8	7.1	20.6	713.9
12	SILO-3	577.4	-29.4	225.8	-21.5	-10.3	711.1
13	SILO-4	527.5	-27.5	243.1	-25.1	-10.5	715.9
14	PIT-3	801.9	-14.2	26.6	-1.8	-10.7	787.2
15	PIT-4	833.6	-10.5	27.7	-0.5	-15.1	780.4
16	2-18-3	484.5	-49.9	35.5	-5.7	-27.7	824.6
17	2-18-4	480.4	-49.9	35.9	-4.7	-28.9	828.5
18	2-8-1	843.7	-17.3	23.6	1.9	11.4	663.2
19	2-8-2	768.4	-15.0	28.0	1.7	11.1	672.3
20	SW-16	614.0	-18.5	45.7	2.5	4.2	713.1
21	SW-20	587.6	2.5	55.7	9.5	32.6	706.9

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 1 CERTIFICATION AND DAY 3 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	686.2	113.9	190.3	42.0	122.3	647.1
2	SCS-M	595.5	-20.7	61.4	0.4	-17.3	725.0
3	SOIL BL	538.9	-40.1	41.1	-3.2	-34.1	735.9
4	SM-1	533.9	-40.6	42.6	-3.1	-34.5	744.6
5	SM-4	543.3	-38.7	43.4	-4.4	-32.3	740.9
6	SM-6	585.6	-36.7	45.8	-4.0	-33.8	736.9
7	SM-8	487.9	-38.3	45.1	-4.7	-29.2	733.1
8	SM-10	471.6	-36.2	47.9	-2.9	-24.8	736.8
9	SM-12	493.9	-32.9	46.7	-3.6	-20.4	732.1
10	SM-14	513.6	-21.6	52.7	-0.7	-6.8	719.5
11	SM-16	617.9	-17.5	50.7	1.9	0.1	718.4
12	SM-20	587.2	4.8	62.7	7.2	28.0	711.5
13	SCS-M	595.4	-18.8	58.6	0.4	-15.6	725.5
14	SM-16	622.4	-16.6	51.4	0.4	0.0	715.3
15	SM-20	589.2	3.1	61.2	7.1	30.1	709.5
16	SOIL BL	542.9	-40.4	40.7	-4.5	-32.5	736.8
17	SM-3	576.6	-37.0	40.1	-3.8	-30.9	728.4
18	SM-6	588.2	-36.7	42.7	-3.2	-31.2	731.9
19	SM-8	492.0	-39.5	43.8	-3.4	-28.8	734.3
20	SM-10	470.5	-36.3	47.0	-1.8	-24.2	733.8
21	SM-12	488.1	-32.3	45.5	-2.2	-20.1	728.8
22	SM-14	517.3	-23.3	47.8	0.1	-3.6	718.8
23	SM-18	489.3	-5.8	56.4	6.1	19.2	716.1
24	SM-14	514.0	-22.9	49.1	0.8	-4.9	721.9
25	SM-16	611.8	-17.3	47.1	1.2	3.1	712.5
26	SM-20	584.2	2.7	60.7	6.5	30.4	710.5
27	SOIL BL	534.2	-40.9	39.7	-5.2	-31.1	737.9
28	SM-8	484.9	-39.3	41.3	-3.1	-27.3	735.8
29	SM-10	462.5	-36.7	43.0	-2.2	-21.2	734.2
30	SM-14	513.0	-22.3	47.1	-0.1	-3.2	719.7
31	SM-14	518.8	-23.3	49.0	0.1	-3.6	722.8
32	SM-18	490.8	-7.5	56.9	7.5	19.5	716.2
33	SM-18	490.7	-7.2	54.1	7.6	21.6	712.4
34	SILO-5	529.3	-27.8	236.8	-24.9	-8.2	698.3
35	SILO-6	530.0	-30.7	230.7	-25.9	-12.7	709.1
36	PIT-5	710.5	-16.2	33.3	-3.2	-19.1	758.7
37	PIT-6	723.6	-15.0	34.3	-0.5	-20.8	749.8
38	2-18-5	379.1	-50.0	44.1	-6.2	-34.3	801.5
39	2-18-6	375.3	-50.6	39.4	-5.0	-31.9	810.2
40	2-8-3	746.3	-15.7	31.1	1.0	4.7	669.4
41	2-8-4	767.3	-18.5	28.7	0.3	5.0	668.1
42	SM-16	614.7	-17.4	46.4	1.0	4.3	711.1
43	SM-20	593.2	1.1	57.5	10.4	31.4	705.9

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 4 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	SM-16	623.2	-18.2	47.9	0.3	4.4	719.6
2	SM-20	587.7	1.9	61.4	9.3	27.8	713.2
3	SOIL BL	540.5	-41.4	38.8	-5.7	-30.2	742.0
4	SM-8	487.9	-40.5	41.0	-4.2	-26.0	740.6
5	SM-10	464.1	-36.3	43.9	-1.6	-21.9	739.7
6	26	635.4	99.8	169.7	45.0	104.9	659.9
7	SM-14	514.5	-25.3	48.9	1.4	-5.2	726.5
8	SM-14	514.7	-23.7	48.5	0.6	-4.1	724.4
9	SM-18	491.2	-7.7	56.3	8.6	18.1	719.1
10	SM-18	496.7	-8.1	56.5	5.7	20.6	718.6
11	SILO-7	513.1	-29.6	240.6	-27.2	-8.4	713.8
12	SILO-8	479.7	-30.8	243.1	-26.0	-11.6	717.0
13	PIT-7	733.9	-14.5	32.6	-2.6	-18.3	769.5
14	PIT 8	785.1	-13.8	31.2	-2.1	-17.4	766.5
15	2-18-7	384.6	-50.0	43.3	-4.7	-35.7	800.2
16	2-18-8	372.2	-48.6	42.4	-4.5	-34.1	794.4
17	2-8-5	775.2	-19.9	29.6	0.5	2.9	680.5
18	2-8-6	736.8	-22.3	32.1	-0.7	0.3	690.5
19	26	558.9	76.9	133.1	33.8	86.8	673.8
20	SM-16	618.9	-18.5	49.0	0.4	2.5	718.4
21	SM-20	588.5	3.9	59.5	9.0	30.0	708.7
22	SCS-M	596.2	-20.8	58.2	1.1	-15.0	727.8

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 2 CERTIFICATION AND DAY 5 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	597.7	96.0	160.6	26.6	104.6	665.2
2	SCS-M	598.8	-19.0	63.8	-0.2	-19.1	733.0
3	SM-16	613.9	-17.2	50.3	0.3	1.2	720.2
4	SM-20	595.3	2.5	61.9	6.4	29.1	714.3
5	SM-3	581.5	-37.5	43.8	-4.2	-33.5	740.1
6	SM-6	586.1	-36.9	42.2	-5.7	-30.1	742.7
7	SM-8	490.7	-39.4	45.2	-4.4	-30.0	743.7
8	SM-10	464.1	-36.4	49.5	-2.3	-25.9	747.2
9	SM-12	494.1	-33.8	48.9	-1.7	-23.4	738.4
10	SM-14	518.7	-22.8	51.4	-0.5	-6.0	728.9
11	SM-18	489.3	-8.6	58.7	5.7	16.9	727.0
12	SOIL BL	543.8	-41.8	40.6	-4.1	-33.5	744.1
13	26, CAI	612.3	97.6	163.8	37.4	100.1	661.7
14	SM-8	490.3	-37.8	42.5	-4.0	-28.3	741.6
15	SM-10	463.7	-36.7	46.1	-1.9	-24.6	743.0
16	SM-14	512.9	-24.6	50.6	1.7	-7.0	726.5
17	SM-14	515.0	-22.8	51.5	-0.2	-6.7	728.2
18	SM-18	491.2	-7.8	59.4	6.7	15.8	722.2
19	SM-18	493.5	-8.2	59.6	3.5	17.6	724.0
20	SILO-9	589.8	-25.6	246.9	-30.7	-7.8	715.3
21	SILO-10	548.1	-27.3	228.6	-27.3	-10.4	714.8
22	26, CAI	613.0	97.1	165.0	38.2	101.9	662.2
23	PIT-9	774.2	-15.3	32.8	-2.0	-19.3	777.0
24	PIT-10	729.6	-17.5	33.1	-2.2	-20.2	775.6
25	2-18-9	413.8	-49.0	42.1	-5.6	-33.2	812.4
26	2-18-10	357.4	-51.4	42.0	-5.2	-33.9	807.6
27	2-8-7	781.1	-17.4	30.6	1.2	5.8	669.7
28	2-8-8	787.0	-18.5	28.3	-0.5	7.1	674.9
29	SM-16	621.7	-16.5	48.4	0.6	3.7	719.0
30	SM-20	599.4	2.7	59.9	8.5	32.1	713.2

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 3 CERTIFICATION AND DAY 6 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	556.0	84.8	136.8	22.7	90.4	665.2
2	SCS-M	598.4	-21.7	58.5	0.8	-15.5	728.2
3	SM-16	619.0	-18.2	50.7	0.8	0.6	721.4
4	SM-20	589.4	0.6	58.9	12.0	28.6	715.0
5	26, GAI	557.5	83.7	135.4	24.6	87.8	667.6
6	SM-3	576.1	-39.1	38.8	-3.0	-29.9	734.1
7	SM-6	578.8	-38.7	38.0	-3.3	-26.7	740.7
8	SM-8	487.0	-39.6	42.6	-4.4	-27.8	743.1
9	SM-10	468.5	-37.4	45.4	-0.4	-23.7	741.5
10	SM-12	495.2	-34.3	42.9	-0.6	-18.5	736.6
11	SM-14	514.6	-23.8	48.5	0.7	-4.4	723.8
12	SM-18	488.1	-7.9	57.4	7.3	19.4	723.9
13	SOIL BL	536.8	-42.6	36.9	-3.9	-30.4	741.0
14	26, GAI	590.7	89.4	141.4	25.7	90.1	672.7
15	SM-8	482.3	-38.1	40.1	-5.4	-24.6	741.5
16	SM-10	463.0	-37.2	44.7	-2.4	-22.3	740.6
17	SM-14	514.8	-23.6	48.8	1.0	-5.0	725.5
18	SM-14	517.6	-22.9	47.6	1.3	-4.4	725.1
19	SM-18	492.1	-7.5	55.8	6.5	18.9	724.5
20	SM-18	494.2	-8.0	56.5	5.0	20.0	720.9
21	SILO-11	543.8	-28.7	239.5	-25.9	-11.2	720.6
22	SILO-12	569.8	-29.1	257.5	-26.5	-9.1	720.4
23	PIT-11	783.5	-11.9	30.1	-1.6	-15.1	773.0
24	PIT-12	797.4	-7.4	28.2	-1.7	-13.8	769.1
25	2-18-11	380.0	-50.9	40.0	-5.2	-31.9	803.2
26	2-18-12	395.1	-51.6	40.8	-5.8	-32.2	812.4
27	2-8-9	767.1	-19.4	28.7	0.7	3.1	677.2
28	2-8-10	756.9	-18.2	30.1	-0.1	5.2	675.1
29	SM-16	616.9	-19.2	47.0	1.3	2.9	721.0
30	SM-20	587.5	-0.5	56.6	11.5	30.6	709.9

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 4 CERTIFICATION AND DAY 7 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	549.7	83.9	138.3	34.8	86.1	665.4
2	SCS-M	599.1	-20.5	56.4	0.5	-11.9	725.1
3	SM-16	619.1	-18.3	47.0	1.9	3.6	719.0
4	SM-20	590.1	0.5	56.1	11.5	30.9	709.1
5	SM-3	573.2	-39.6	36.6	-4.8	-26.8	736.0
6	SM-6	584.7	-38.8	36.4	-2.9	-26.3	739.1
7	SM-8	492.5	-39.7	40.0	-5.3	-24.3	740.0
8	SM-10	469.0	-36.9	43.9	-0.7	-22.2	742.7
9	SM-12	497.5	-35.5	43.8	-1.0	-18.5	739.5
10	SM-14	518.1	-24.2	46.6	1.5	-3.0	726.9
11	SM-18	494.0	-8.0	54.5	7.6	21.0	722.8
12	SOIL BL	544.4	-41.0	34.9	-5.0	-27.4	737.7
13	26	543.9	74.8	127.1	26.8	79.3	672.9
14	SM-8	490.9	-39.1	38.5	-4.7	-23.9	741.0
15	SM-10	467.5	-36.0	41.7	-1.7	-19.6	742.2
16	SM-14	516.3	-25.6	46.0	2.9	-2.6	727.3
17	SM-14	519.8	-23.3	45.5	0.6	-1.6	723.2
18	SM-18	492.3	-8.1	55.6	7.1	20.6	724.0
19	SM-18	495.2	-8.3	53.7	6.6	21.9	724.5
20	SILO-13	545.8	-27.6	238.9	-25.1	-5.9	704.2
21	SILO-14	560.6	-28.4	240.2	-24.5	-10.4	710.0
22	PIT-13	760.2	-15.4	31.9	-2.5	-18.5	772.1
23	PIT-14	725.6	-15.0	32.8	-2.7	-17.8	772.1
24	2-18-13	382.7	-50.3	41.2	-4.6	-33.3	800.8
25	2-18-14	375.0	-51.3	38.4	-5.3	-29.8	795.5
26	2-8-11	769.3	-18.6	28.2	1.9	6.1	667.0
27	2-18-12	750.3	-19.7	27.7	0.0	4.6	679.9
28	SM-16	616.6	-19.0	47.8	1.3	3.7	722.1
29	SM-20	592.7	0.2	55.1	12.1	31.1	713.5

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 8 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	713.5	112.0	182.6	53.2	114.1	647.6
2	SCS-N	593.6	-20.9	60.9	-0.2	-16.4	730.4
3	SM-16	613.0	-17.8	51.5	1.9	-0.4	721.4
4	SM-20	588.3	3.3	62.3	9.5	28.5	709.2
5	SOIL BL	537.6	-40.5	39.7	-3.6	-32.4	739.7
6	SM-8	483.5	-39.0	45.5	-3.3	-30.9	743.7
7	SM-10	462.4	-35.5	47.3	-2.1	-24.6	738.6
8	SM-14	511.0	-23.2	50.3	2.1	-7.6	722.0
9	SM-14	512.9	-21.9	50.1	-0.2	-4.9	722.4
10	SM-18	489.9	-8.2	56.9	6.1	17.8	721.6
11	SM-18	491.0	-7.9	56.5	5.9	19.5	718.8
12	26	782.3	122.4	210.0	64.7	125.7	639.4
13	SCS-N	595.7	-21.0	59.6	0.5	-15.6	726.6
14	SILO-15	517.7	-27.5	227.9	-26.7	-12.7	698.5
15	SILO-16	551.2	-27.1	259.9	-30.9	-8.6	705.8
16	PIT-15	769.0	-13.7	32.8	-1.7	-18.6	762.7
17	PIT-16	752.0	-14.8	31.9	-0.6	-18.7	769.3
18	2-18-15	409.0	-49.8	43.2	-4.9	-35.3	818.2
19	2-18-16	417.8	-50.8	41.7	-6.5	-33.7	807.8
20	2-8-13	875.2	-15.4	27.0	0.2	5.3	664.9
21	2-8-14	748.1	-17.3	32.0	-0.3	5.1	671.7
22	SM-16	617.2	-19.3	50.2	1.3	0.1	718.8
23	SM-20	587.3	0.9	60.5	8.5	30.0	709.5
24	RABBIT	17.1	-134.9	-38.6	-10.1	35.8	1593.0

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 9 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	598.6	94.5	161.1	3.9	110.2	671.8
2	SCS-N	594.5	-19.6	68.3	-2.4	-21.9	741.9
3	SM-16	610.8	-16.4	59.4	-1.9	-5.3	725.7
4	SM-20	589.0	4.2	67.2	2.9	27.0	713.4
5	SOIL BL	539.6	-41.1	47.9	-5.0	-39.2	749.9
6	SM-8	489.0	-37.2	49.3	-5.5	-33.5	741.9
7	SM-10	466.2	-34.3	52.5	-3.1	-28.6	744.5
8	SM-14	511.1	-22.9	57.1	-1.5	-11.0	730.1
9	SM-14	513.4	-21.8	58.3	-0.2	-1[illegible].5	726.3
10	SM-18	489.3	-5.4	63.4	1.8	16.0	721.2
11	SM-18	490.9	-6.7	63.0	2.9	14.5	726.6
12	26, GAI	616.1	94.9	164.0	12.6	104.3	665.4
13	SCS-N	597.2	-18.9	65.8	-0.9	-21.0	733.0
14	SILO-17	540.8	-26.0	239.2	-34.3	-7.1	724.6
15	SILO-18	561.0	-24.5	255.3	-34.7	-11.6	715.1
16	PIT-17	754.9	-13.5	37.9	-2.1	-22.7	783.1
17	PIT-18	755.5	-14.4	37.9	-0.1	-23.5	775.5
18	2-18-17	393.3	-49.5	47.3	-5.6	-38.8	809.6
19	2-18-18	367.1	-47.7	47.4	-4.9	-38.9	805.1
20	2-8-15	773.9	-16.8	34.9	-1.0	-0.7	676.7
21	2-8-16	803.3	-15.9	34.5	0.4	2.3	671.4
22	SM-16	616.5	-18.8	51.5	-0.2	-0.2	723.2
23	SM-20	591.0	3.0	64.3	7.0	27.4	712.3

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 10 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	550.9	81.7	135.6	1.2	90.7	678.1
2	SCS-N	596.9	-19.7	66.5	-2.0	-21.5	733.0
3	SN-16	614.8	-15.5	57.0	-1.8	-3.8	724.3
4	SN-20	587.8	3.5	65.4	2.6	28.2	713.3
5	SOIL BL	535.5	-40.0	46.2	-4.6	-37.9	740.9
6	SN-8	492.2	-38.0	49.5	-5.1	-34.1	743.7
7	SN-10	468.7	-34.9	51.5	-2.8	-27.7	742.6
8	SN-14	514.8	-21.2	53.4	-0.5	-8.4	723.3
9	SN-14	514.9	-23.0	55.0	0.0	-9.4	721.9
10	SN-16	487.0	-6.5	64.2	2.7	14.7	720.7
11	SN-18	489.4	-6.8	63.7	2.2	16.2	721.[illegible]
12	26, GAI	584.0	88.8	150.1	10.7	92.7	670.9
13	SCS-N	596.9	-18.5	62.6	0.5	-18.3	728.3
14	SILO-19	548.7	-24.9	244.3	-35.4	-7.7	710.9
15	SILO-20	537.5	-27.6	246.8	-34.3	-10.8	718.6
16	PIT-19	729.8	-12.6	36.9	-1.0	-23.9	770.3
17	PIT-20	762.7	-13.7	37.7	-1.6	-22.8	769.1
18	2-18-19	363.9	-50.0	48.2	-5.8	-39.5	799.6
19	2-18-20	368.1	-49.1	50.0	-5.8	-40.6	810.8
20	2-8-17	707.8	-20.8	37.7	-0.4	-5.4	685.6
21	2-8-18	885.2	-15.4	31.6	0.4	2.0	669.4
22	SN-16	619.5	-15.7	53.1	-2.5	1.0	716.5
23	SN-20	588.1	2.1	60.7	5.5	30.3	709.2

TITLE: X-MET 840 FIELD NET INTENSITIES, WET SOIL, ALL ELEMENTS.
DAY 11 EQUIVALENCY.

	SAMPLE	Fe NET INT	Cu NET INT	As NET INT	Hg NET INT	Pb NET INT	BS NET INT
1	26	578.5	89.0	152.3	-3.6	97.0	672.0
2	SCS-N	599.7	-18.5	67.4	-1.4	-22.4	735.1
3	SN-16	624.0	-15.6	56.6	-0.8	-4.1	723.5
4	SN-20	588.9	4.1	66.3	2.8	27.4	713.7
5	SOIL BL	537.0	-38.7	46.8	-3.9	-38.3	739.4
6	SN-8	486.9	-38.7	48.3	-3.7	-33.7	743.3
7	SN-10	466.4	-35.4	54.2	-3.6	-29.4	741.2
8	SN-14	518.6	-21.1	57.0	-2.1	10.0	723.0
9	SN-14	514.5	-22.5	53.5	0.0	-8.8	723.7
10	SN-18	488.7	-6.3	43.4	3.1	13.9	724.3
11	SN-16	489.1	-5.4	62.3	2.7	15.1	721.5
12	26, GAI	595.3	93.0	149.6	5.5	91.3	669.3
13	SCS-N	594.8	-20.0	64.7	-1.6	-19.2	734.1
14	2-8-19	735.0	-17.3	36.0	-0.3	2.5	677.5
15	2-8-20	738.9	-19.4	37.1	-0.3	-3.0	681.7
16	SILO-21	520.8	-24.9	243.6	-34.5	-8.4	710.6
17	SILO-22	565.7	-24.5	250.8	-39.0	-5.7	710.0
18	PIT-21	800.1	-13.7	38.4	-2.9	-23.8	792.8
19	PIT-22	758.7	-12.3	38.5	-1.6	-24.3	778.4
20	2-18-21	393.6	-49.5	48.5	-4.8	-40.3	808.8
21	2-18-22	342.7	-48.5	50.2	-5.3	-41.0	788.6
22	SN-16	614.4	-16.4	55.2	0.2	-3.4	722.7
23	SN-20	586.9	3.[illegible]	65.2	6.1	27.4	709.5

APPENDIX H

Laboratory Analyses of Surrogate and Actual Field Samples

Table H-1

Analysis of Surrogate and Actual Field Samples Using Laboratory Based Instrumentation

Element: Copper Analytical Method: Inductively Coupled Plasma

		Water Samples		Soil Samples				
Sample Analysis Day	Replicate Number	LWEM*	1703	LSEM	Silo	Pit	2-18	2-8
1	1	120	1	160	36	210	8	18
1	2	123	1	120	15	210	7	18
2	1	106	1	120	17	220	6	17
2	2	108	1	110	17	230	7	19
3	1	100	1	100	14	200	8	12
3	2	97	1	110	15	3400	6	14
4	1	102	1	120	20	220	7	17
4	2	99	1	130	17	220	6	17
5	1	110	0	120	16	200	5	14
5	2	110	0	79	35	190	6	12
6	1	110	1	120	18	210	5	16
6	2	110	1	110	19	210	6	14
7	1	100	1	120	16	230	6	16
7	2	100	1	110	19	210	6	14
8	1	110	1	120	19	250	7	21
8	2	110	1	120	22	220	6	17
9	1	110	1	130	24	230	7	15
9	2	110	1	100	15	230	7	18
10	1	100	1	110	21	240	8	17
10	2	100	1	150	20	240	7	32
AVG		107	0.9	118	19.8	219	6.6	16.9
SD		7	0.3	17	6.0	15	0.9	4.2
RSD		7	33	15	30	7	14	25

*Spiked equivalency samples prepared using RMA soil and ORNL groundwater. Concentration in soil 100 μg/g for all elements. Concentration in water 50 μg/mL for arsenic, 100 μg/mL for others.

Table H-2

Analysis of Surrogate and Actual Field Samples Using
Laboratory Based Instrumentation

Element: Arsenic Analytical Method: Graphic Furnace Atomic Absorption

		Water Samples		Soil Samples				
Sample Analysis Day	Replicate Number	LWEM*	1703	LSEM	Silo	Pit	2-18	2-8
1	1	50	4800	69	1840	3	1.2	1.8
1	2	48	4950	92	1700	2.7	1.2	2.6
2	1	50	5000	67	1820	3	1	2.5
2	2	53	5050	92	1760	2.7	1.2	2.5
3	1	50	5000	69	1950	3.2	1.3	2.6
3	2	49	4650	84	1700	2.7	1.4	2.7
4	1	50	5100	71	2070	3	1.2	3
4	2	48	5100	84	2040	3	1.2	2.4
5	1	50	4900	78	1880	3.4	1.3	3.2
5	2	46	5050	84	1620	2.7	1.4	2.8
6	1	48	5420	65	1810	3.2	1.1	3.3
6	2	49	5250	90	1720	3	1.2	2.8
7	1	49	5150	74	1840	3.2	1.3	3
7	2	54	5300	83	1890	2.8	1.3	3
8	1	48	5000	74	1870	3.4	1.1	3.2
8	2	50	4700	95	1600	2.8	1.5	2.8
9	1	48	4600	66	1960	3.2	1.1	3.4
9	2	47	4900	83	1630	3.1	1.1	2.9
10	1	54	5300	69	1720	3.2	1.5	2.8
10	2	46	5250	82	1800	3.1	1	3.1
AVG		49	5025	79	1811	3	1	3
SD		2	227	10	133	0	0	0
RSD		5	5	12	7	7	12	13

*Spiked equivalency samples prepared using RMA soil and ORNL groundwater. Concentration in soil 100 μg/g for all elements. Concentration in water 50 μg/mL for arsenic, 100 μg/mL for others.

Table H-3

Analysis of Surrogate and Actual Field Samples Using
Laboratory Based Instrumentation

Element: Mercury Analytical Method: Cold Vapor Atomic Absorption

		Water Samples		Soil Samples				
Sample Analysis Day	Replicate Number	LWEM*	1703	LSEM	Silo	Pit	2-18	2-8
1	1	81	0	34	0	0	0	0
1	2	77	0	25	0	0	0	1
2	1	88	0	54	0	0	0	0
2	2	93	0	48	0	0	0	1
3	1	103	0	46	0	0	0	0
3	2	108	0	44	1	0	0	0
4	1	106	0	48	0	0	0	0
4	2	101	0	31	0	0	0	0
5	1	105	0	34	0	0	0	1
5	2	104	0	39	0	0	0	1
6	1	116	0	38	1	0	0	0
6	2	113	0	56	0	0	0	1
7	1	115	0	38	0	0	0	1
7	2	108	0	39	0	0	0	0
8	1	109	0	45	0	0	0	0
8	2	110	0	44	0	0	0	0
9	1	93	0	40	0	0	0	1
9	2	106	0	420	0	0	0	1
10	1	110	0	37	0	0	0	0
10	2	100	0	37	0	0	0	1
AVG		102	0	41	0	0	0	0
SD		11	0	7	0	0	0	1
RSD		11		18				

*Spiked equivalency samples prepared using RMA soil and ORNL groundwater. Concentration in soil 100 $\mu g/g$ for all elements. Concentration in water 50 $\mu g/mL$ for arsenic, 100 $\mu g/mL$ for others.

Table H-4

Analysis of Surrogate and Actual Field Samples Using Laboratory Based Instrumentation

Element: Lead Analytical Method: Inductively Coupled Plasma

		Water Samples		Soil Samples				
Sample Analysis Day	Replicate Number	LWEM*	1703	LSEM	Silo	Pit	2-18	2-8
1	1	91	23	160	77	180	13	1010
1	2	94	19	110	47	200	11	640
2	1	99	22	100	70	190	10	560
2	2	100	22	91	120	200	10	770
3	1	100	22	110	160	230	11	790
3	2	100	22	120	75	240	10	680
4	1	103	22	120	1000	190	10	810
4	2	99	22	130	95	200	6	500
5	1	110	22	130	53	190	9	800
5	2	100	22	89	150	400	10	660
6	1	110	22	120	84	180	10	930
6	2	110	22	120	72	320	9	710
7	1	110	23	110	53	190	8	840
7	2	100	22	100	2400	210	8	780
8	1	110	24	120	58	200	10	860
8	2	110	24	120	86	280	10	800
9	1	100	22	130	50	211	11	600
9	2	100	23	99	110	240	11	950
10	1	110	23	96	69	220	11	820
10	2	110	23	130	58	190	11	940
AVG		103	22	115	244	223	10	773
SD		6	1	17	548	54	2	136
RSD		6	5	15	224	24	20	18

*Spiked equivalency samples prepared using RMA soil and ORNL groundwater. Concentration in soil 100 $\mu g/g$ for all elements. Concentration in water 50 $\mu g/mL$ for arsenic, 100 $\mu g/mL$ for others.

APPENDIX I

Statistical Evaluation of Equivalency Testing for Selected Element/Sample Combinations

Statistical Evaluation of Equivalency Testing for Selected Element/Sample Combinations

I. ARSENIC

1. Outliers: There were no outliers outside the "Mean $\pm$ 4*(Std Dev)". However, for Sample = LWEM, Method = CSI, Day = 10, and Replicate = 2 the concentration (ug/ml) = 6 is identified as an outlier by the one-sided Dixon's Test [pp 167, 2]. This data point will be closely examined for any following statistical test. In addition, the Shapiro and Wilk's W-statistics [pp 177, 2] were calculated for each case to check if the normal assumption is appropriate. The probabilities of the W-statistic are given in Table I.1. These probabilities should be greater than 0.05. Only the data for Sample = LWEN, and Method = LAB have a low probability (i.e., 0.033). This case has a small range (e.g., 54 - 46 = 8) but the values are shewed towards the lower values. However, for this data set, the significant Shapiro and Wilk's test should not affect other statistical test. A summary table of the data is as follows:

Table I.1

Summary Statistics for Arsenic Measurements

Sample	Method*	N	Mean	St. Dev.	Min	Max	W-statistic
LWEM μg/mL	CSI	20	44	14	6	66	0.116
	LAB	20	49	2	46	54	0.033
	USA	20	53	9	35	69	0.701
1703 μg/mL	CSI	22	3314	18	3278	3341	0.338
	LAB	20	5025	227	4600	5450	0.830
	USA	20	3607	944	3607	6769	0.121
LSEM μg/g	CSI	20	96	20	51	137	0.863
	LAB	20	79	10	65	95	0.130
	USA	20	93	25	52	144	0.572
SILO μg/g	CSI	22	2517	124	2307	2783	0.674
	LAB	20	1811	133	1600	2070	0.735
	USA	22	3302	277	2811	3808	0.708

* CSI refers to the XRF manufactuer's multivariate calibration scheme. USA refers tot he USATHAMA single variate calibration scheme. LAB refers to the reference analytical method.

2. <u>Equality of Replicate Variance</u>: The least-squares fit of $Log(S_{ij}) = a \log(M_{ij}) + b$ indicated two cases with significant slope. These cases are given in Table I.2 with the estimated slopes. For both cases, the slopes are negative indicating that variance decreases with increasing concentration which is usually opposite of what is expected. A closer examination of the data in Fig. I.1 and Fig. I.2 show that one value in each case is at an extreme and the remaining values are in a cluster. The extreme values have a high leverage (or influence) on the slope of each line. If these values are removed, the slopes for both this cases are not significantly different than zero at the 5% significance level. Therefore, the inference from this analysis is that the assumption of equality of variance can be made for all the data sets.

Table I.2

Equality of Variance Analysis for Two Special Cases

Sample	Method	Slope	P-Value	Slope[a]	P-Value
LWEM µg/mL	CSI	-7.25	0.026	-12.41	0.104
SILO µg/g	LAB	-91.7	0.023	-108.6	0.221

[a]High leverage point removed.

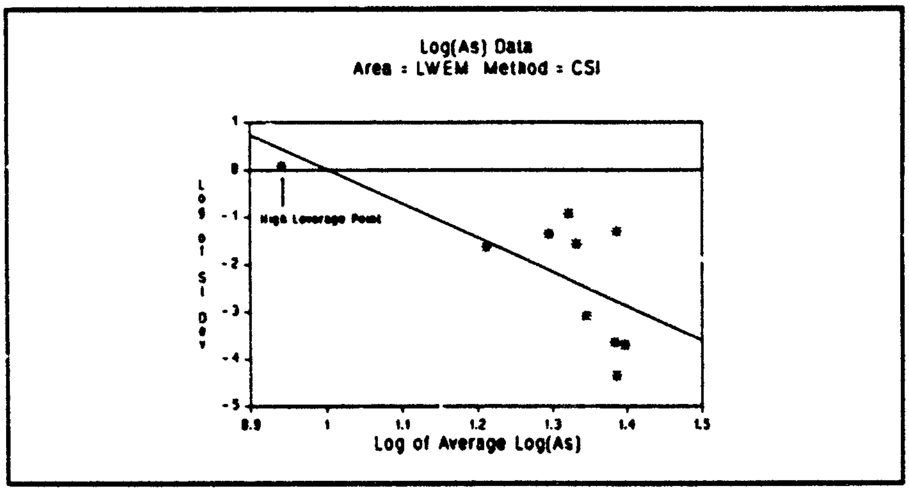

Fig. I.1 Equality of variance test for Sample LWEM and Method=CSI.

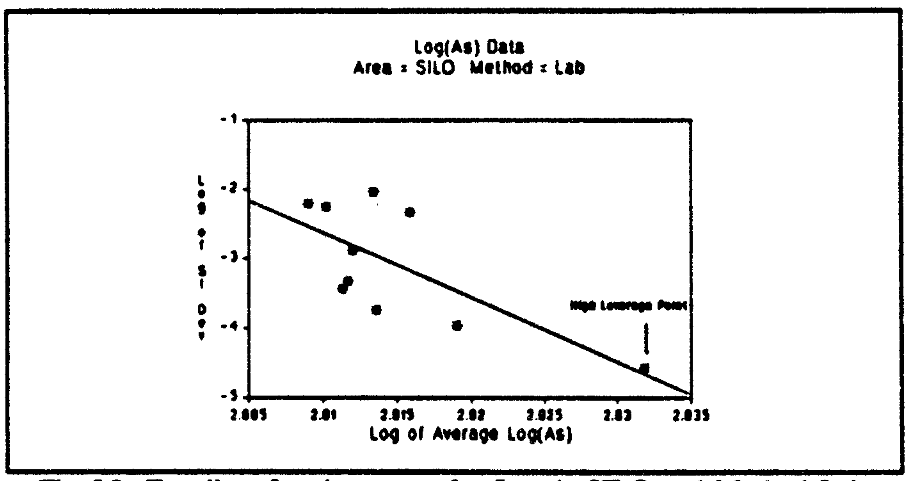

Fig. I.2 Equality of variance test for Sample SILO and Method Lab.

3. Variance Ratio - 95% Confidence Interval: The 95% confidence intervals of the ratio of the variance for the proposed method (i.e., CSI and USA) to the variance of the reference method (i.e., LAB) are given in Table I.3. These results show that there are significant differences between the proposed method and accepted method for all methods used on aqueous samples (e.g., LWEM and 1703). For the soil samples (e.g., LSEM and SILO), only Method = USA for Sample = LWEN has 95% confidence intervals excluding 1 (e.g., [1.08, 4.01]). The aqueous sample results depend on the level of concentration. The variance of the proposed method is larger than that of reference metnod for low concentration samples (e.g., LWEM) while the variance of the proposed method is smaller than that of the referencece method for high concentration samples (e.g., 1703).

Table I.3

The 95% Confidence Intervals of the Proposed-Method Variances to Accepted-Method Variances

Sample	Method	Lower Limit	Ratio	Upper Limit	Proposed Variance	Accepted Variance	Num Df[a]	Dem Df[a]
LWEM	CSI	4.03160	14.9846	55.6947	97.4	6.5	10	10
LWEM	USA	4.97120	18.4769	68.6749	120.1	6.5	10	10
1703	CSI	0.00128	0.0047	0.0165	97.1	20750.0	11	10
1703	USA	0.00182	0.0068	0.0251	140.3	20750.0	10	10
LSEM	CSI	0.59275	2.2031	8.1886	351.4	159.5	10	10
LSEM	USA	1.07940	4.0119	14.9114	639.9	159.5	10	10
SILO	CSI	0.26892	0.9856	3.4748	17395.2	17650.0	11	10
SILO	USA	0.52125	1.9374	7.2008	34194.8	17650.0	10	10

[a]Degrees of freedom

4. Bias Test by ANOVA: According to the EPA Equivalency Petition, the bias test for the proposed methods can only be performed on those cases that have equivalent precisions (see, part 3). However, the ANOVA test is fairly robust to deviations in the assumptions of equivalent variances, and normality. Therefore, all methods were examined by the ANOVA procedure. For cases with 11 days, only the first 10 days were used. The ANOVA results are summarized in Table I.4 by listing the probability values of the F-test for each source of variation. A probability value of less than 0.05 (i.e., 5% significance level) indicates a significant difference among the levels of the sources of variation. A significant METHOD x DAY interaction indicates that the results varied as a function the different results of the day on which the determination were made. If the interaction source is not significant, a pooled error term is used to test the METHOD effect. In this analysis, the significant or non-significant METHOD effect did not change from Table I.4 by using the pooled error term. The only non-significant METHOD effect was for Sample = LWEM with USA vs LAB. All the other cases would fail the EPA equivalency test based on the ANOVA bias analysis. For Sample = LWEM and Method = USA vs LAB, this case would fail because the variances are not equivalent. The data are illustrated in Fig. I.2-I.5 by box plots which support the equivalent petition results.

Table I.4

Probability Values for the Sources of Variation
in the ANOVA Table

Method	Source of Variation	LWEM µg/mL Low	1703 µg/mL High	LSEM µg/g Low	SILO µg/g High
CSI vs Lab	Method	0.0321	0.0001	0.0029	0.0001
	Day	0.0121	0.0035	0.3856	0.7074
	Method X Day	0.0157	0.0045	0.6292	0.4428
USA vs Lab	Method	0.1854	0.0001	0.0361	0.0001
	Day	0.9499	0.0001	0.5704	0.1402
	Method X Day	0.9785	0.0001	0.7133	0.0070

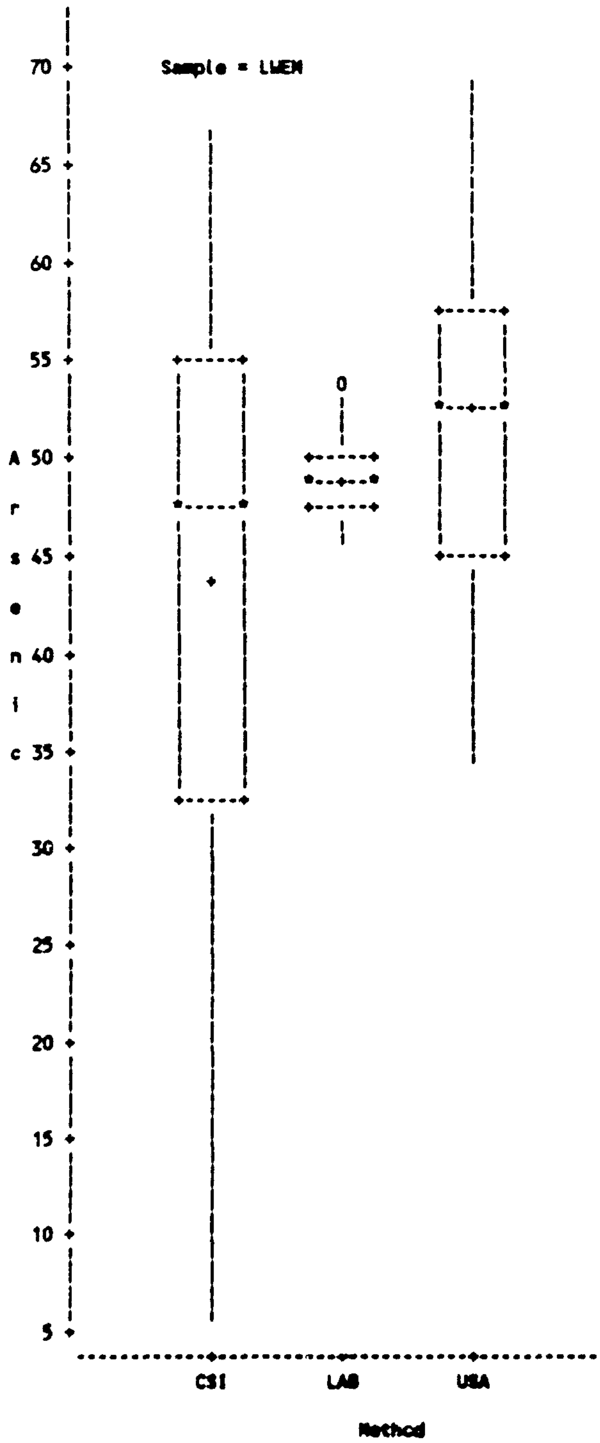

Fig. 1.3 Arsenic measurements (µg/mL) for Sample LWEM.

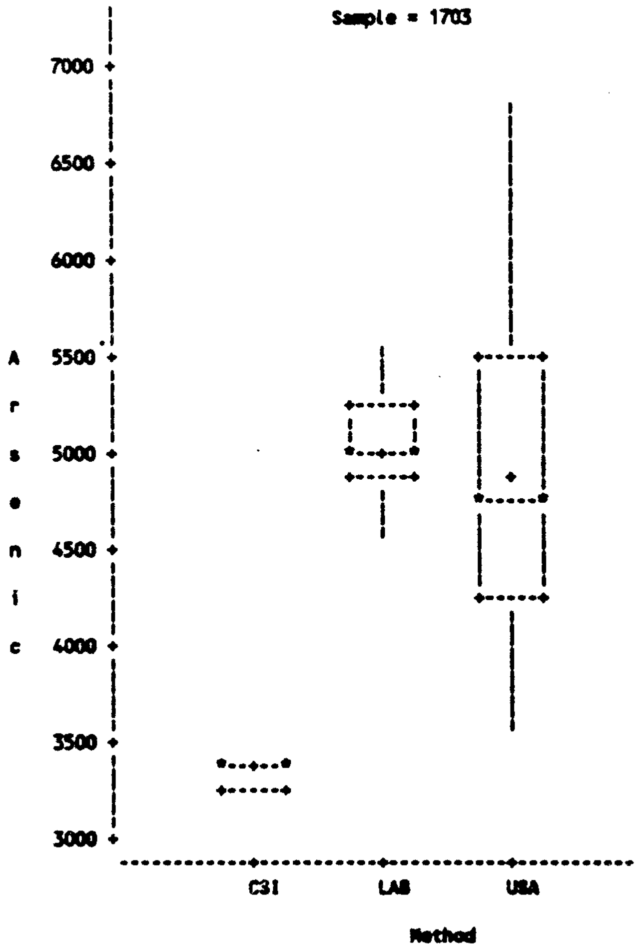

Fig. I.4 Arsenic measurements (µg/mL) for sample 1703.

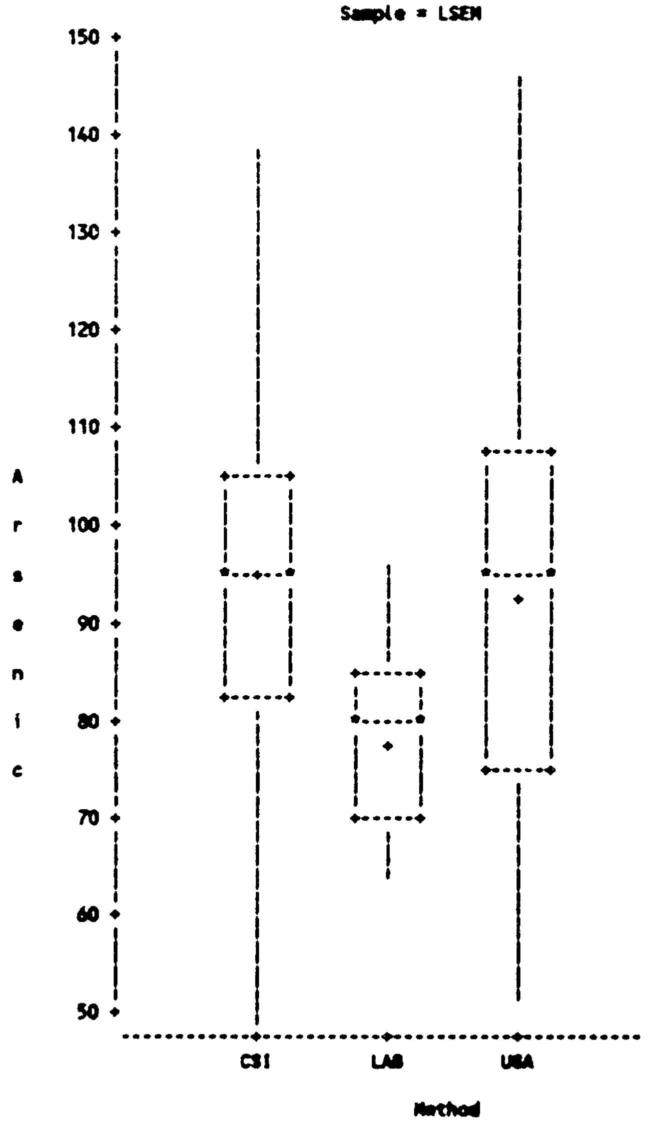

Fig. 1.5 Arsenic measurements (µg/g) for Sample LSEM.

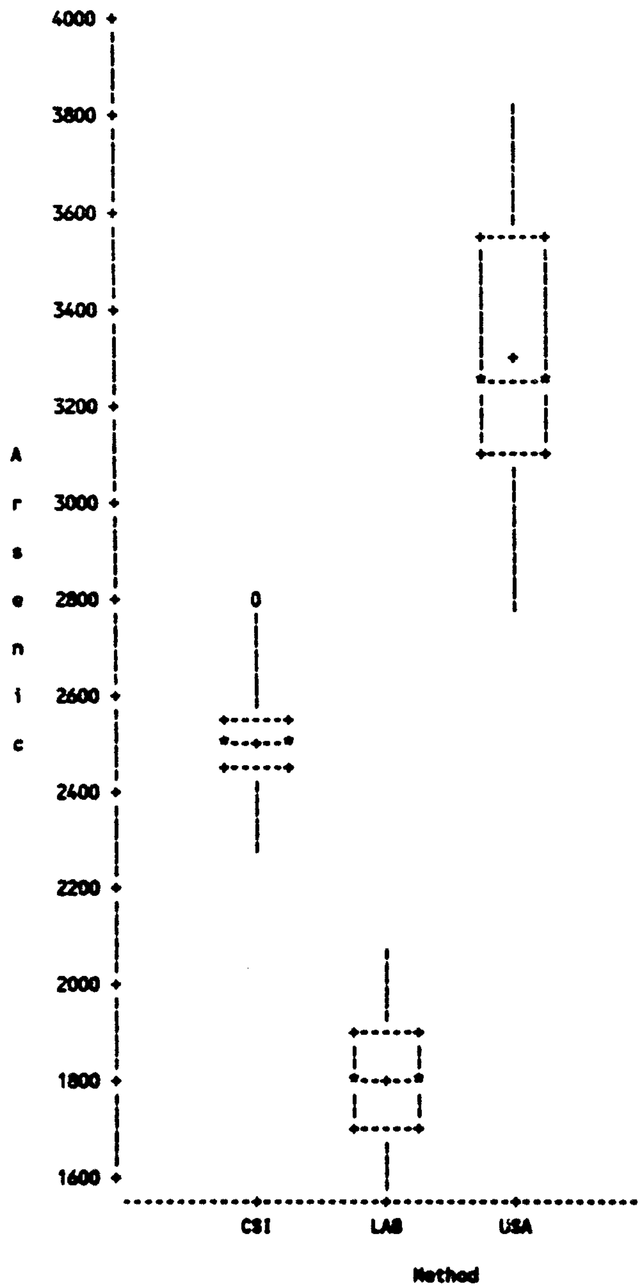

Fig. 1.6 Arsenic measurements (µg/g) for Sample SILO.

II. COPPER

1. Outliers: There were no outliers outside the "Mean ± 4*(Std Dev)" range. The Shapiro and Wilk's W-statistics [pp 177, 2] were calculated for each case to check if the normal assumption is appropriate. The probabilities of the W-statistic are given in Table II.1. These probabilities should be greater than 0.05. The data for Sample = LWEM, and Method = LAB have a low probability (i.e., 0.012), thus the values are skewed towards the lower values. A summary table of the data is as follows:

Table II.1

Summary Statistics for Copper Measurements

Sample	Method	N	Mean	St. Dev.	Min	Max	W-statistic
LWEM µg/mL	CSI	20	96	4	85	104	0.211
	LAB	20	107	7	97	123	0.012
	USA	20	78	3	72	84	0.179

2. Equality of Replicate Variance: The least-squares fit of $\text{Log}(S_{ij}) = a \log(M_{ij}) + b$ to the logarithms of the copper concentrations did not indicate any significant slopes. Therefore, the inference from this analysis is that the assumption of equality of variance can be made for copper concentrations for Sample = LWEM and all methods.

3. Variance Ratio - 95% Confidence Interval: The 95% confidence intervals of the ratio of the variance for the proposed methods (i.e., CSI and USA) to the variance of the accepted methods (i.e., LAB) are given in Table II.2. These results show that there are significant differences between the variances of the proposed method and reference method for all methods used for Sample = LWEM.

Table II.2

The 95% Confidence Intervals of the Proposed-Method Variances to Accepted-Method Variances

Sample	Method	Lower Limit	Ratio	Upper Limit	Proposed Variance	Accepted Variance	Num Df	Dem Df
LWEM	CSI	4.72	10.12	37.63	16.2	1.6	10	10
LWEM	USA	1.35	5.00	18.58	8.0	1.6	10	10

4. Bias Test by ANOVA: According to the EPA Equivalency Petition, the bias test for the proposed methods can only be performed on those cases that have equivalent precisions (see, part 3). However, the ANOVA test is fairly robust to deviations in the assumptions of equivalent variances, and normality. Therefore, all methods were examined by the ANOVA procedure. The ANOVA results are summarized

in Table IV.3 by listing the probability values of the F-test for each source of variation. A probability value of less than 0.05 (i.e., 5% significance level) indicates a significant difference among the levels of the sources of variation. A significar: METHOD x DAY interaction indicates that the results varied as a function the different results of the day on which the determination were made. If the interaction source is not significant, a pooled error term is used to test the METHOD effect. For the cooper measurements at sample LWEM, all sources of variations were significant at the 5% significant level for both proposed methods. Therefore, the proposed methods for measuring cooper would fail the equivalent petition criteria. The data are illustrated in Fig.II.1 by a box plot which support the equivalent petition results.

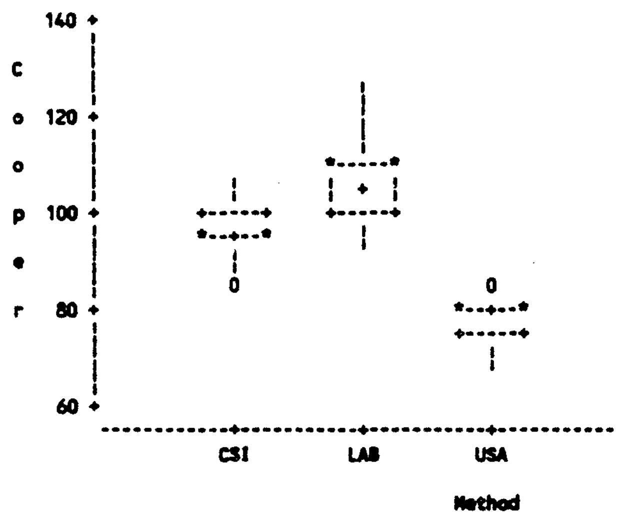

Fig. II.1 Copper measurements (µg/mL) for Sample LWEM.

III. LEAD

1. Outliers: There were no outliers outside the "Mean ± 4*(Std Dev)". The Shapiro and Wilk's W-statistics [pp 177, 2] were calculated for each case to check if the normal assumption is appropriate. The probabilities of the W-statistic are given in Table III.1 These probabilities should be greater than 0.05. The data for sample = LWEM, and Method = LAB have a low probability (i.e., 0.001). The values are skewed towards the lower values. A summary table of the data is as follows:

Table III.1

Summary Statistics for Lead Measurements

Sample	Method	N	Mean	St. Dev.	Min	Max	W-statistic
LWEM μg/mL	CSI	20	111	31	66	192	0.140
	LAB	20	103	6	91	110	0.001
	USA	20	107	14	72	134	0.552
2-8 μg/g	CSI	20	798	117	532	1018	0.450
	LAB	20	773	136	500	1010	0.822
	USA	20	852	71	710	964	0.592

2. Equality of Replicate Variance: The least-squares fit of $\log(S_{ij}) = a \log(M_{ij}) + b$ indicated two cases with significant slope for Sample = 2-8, Methods= CSI and USA. These cases are given in Table III.2 with the estimated slopes. For both cases, the slopes are negative, indicating that variance decreases with increasing concentration. This is the opposite of what is typicalloy encountered. The transformation suggested by the equivalency petition is $Y = [\log(Pb)]^{1-a}$, which means the logarithm of the lead concentrations for methods CSI and USA would be raised to 47.4 power and 93.9 power, respectively. A plot of $\log(S_{ij})$ vs $\log(M_{ij})$ in Fig. II.1 (a) and Fig. II.1 (b) show no unusual leverage points. A closer examination of the data was made by plotting the standard deviation of Pb concentration and the average Pb concentration for each day. Both the standard deviations vs. days and averages vs days are on the same plot in Fig. II.1 (c) and Fig. II.1 (b). The left-hand y-axis is the scale for the standard deviations and the right-hand y-axis is the scale for the averages. Figures II.1 (a) and II.1 (b) show that the averages are very high for day = 1 and standard deviations are low. The reverse is true for days 9 and 10 with low averages and high standard deviations. These results indicated the significant equality of variance analysis were due to a significant "Day" effect. Therefore, transformation were not applied to the Pb data for methods CSI and USA. In addition, a comparison of the ranges of the standard deviations for the replicate samples were made for the three methods. This comparison showed that the standard deviation range for Method = Lab (e.g., St. Dev. range = 219) was larger than the standard deviation ranges for Method = CSI and Method = USA (e.g., St. Dev. ranges = 144 and 115, respectively).

Both the average value and the standard deviation appear to depend upon the day on which the samples were analyzed. However, we believe that this is a fortuitous consequence of the fact that the largest standard deviation falls on the last day of testing, and the smallest standard deviation occurs on the first day.

Table III.2

Equality of Variance Analysis for Sample = 2-8 and Methods = CSI and USA

Sample	Method	Slope	P-Value
2-8 $\mu g/g$	CSI	-46.4	0.001
2-8 ug/g	USA	-92.9	0.022

3. Variance Ratio - 95% Confidence Interval: The 95% confidence intervals of the ratio of the variance for the proposed method (i.e., CSI and USA) to the variance of the reference method (i.e., LAB) are given in Table III.3. These results show that there are significant differences between the variances of the proposed method and reference methods for all methods used for the = LWEM and 2-8 samples. The proposed method variance is larger than the reference method variance method for aqueous samples (e.g., LWEM) while the proposed method variance is smaller than the reference method variance for soil samples (e.g., 2-8).

Table III.3

The 95% Confidence Intervals of the Proposed-Method Variances to Accepted-Method Variances

Sample	Method	Lower Limit	Ratio	Upper Limit	Proposed Variance	Accepted Variance	Num Df	Dem Df
LWEM	CSI	8.18	30.39	112.95	343.4	11.3	10	10
LWEM	USA	6.09	22.62	84.07	255.6	11.3	10	10
2-8	CSI	0.05	0.18	0.67	4516.1	25065.0	10	10
2-8	USA	0.03	0.12	0.44	2958.7	25065.0	10	10

4. Bias Test by ANOVA: According to the EPA Equivalency Petition, the bias test for the proposed methods can only be performed on those cases that have equivalent precisions (see, part 3). However, the ANOVA test is fairly robust to deviations in the assumptions of equivalent variances, and normality. Therefore, all methods were examined by the ANOVA procedure. The ANOVA results are summarized in Table IV.3 by listing the probability values of the F-test for each source of variation. A probability value of less than 0.05 (i.e., 5% significance level) indicates a significant difference among the levels of the sources of variation. A significant METHOD x DAY interaction indicates that the results varied as a function the different results of the day on which the determinations were made. If the interaction source is not significant, a pooled error term is used to test the METHOD effect. For the lead measurements at sample LWEM, all sources of variations were significant at the 5% significant level for the CSI proposed method. However, the USA proposed method shows no significant differences from the Lab results and would be accepted by the equivalency petition criteria. The lead results for sample 2-8 in Table IV.3 show no significant results for the Day effect and Method X Day interaction. The ANOVA results from pooling the interaction effect with the Day effect shows that there is no significant

Method effect for the CSI method (i.e. P-value = 0.5389) but a significant effect for the USA method (i.e., P-value = 0.0322) Therefore, only CSI proposed method at sample 2-8 would pass the equivalent petition criteria for accuracy but not for precision. The data are illustrated in Fig. III.2 and Fig. III.3 by box plots.

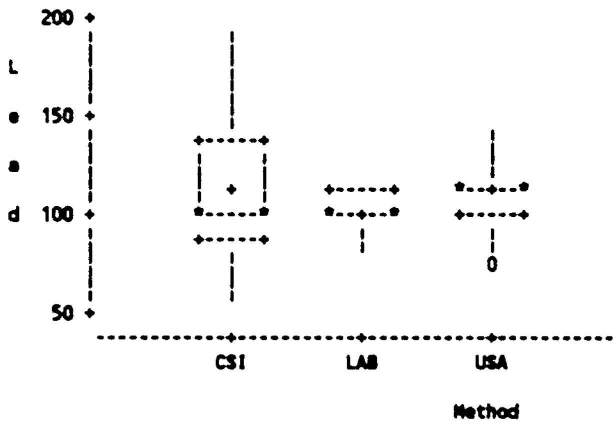

Fig. III.1 Lead measurements (µg/mL) for Sample LWEN.

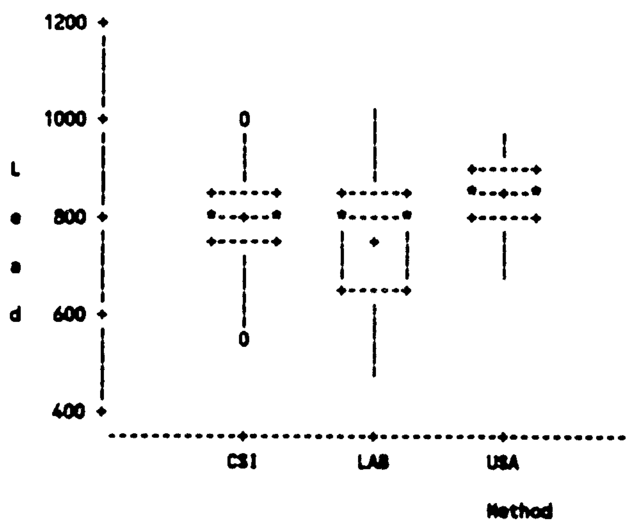

Fig. III.2 Lead measurements (µg/g) for Sample 2-8.

IV. MERCURY

1. Outliers: There were no outliers outside the "Mean ± 4*(Std Dev)". The Shapiro and Wilk's W-statistics [pp 177, 2] were calculated for each case to check if the normal assumption is appropriate. The probabilities of the W-statistic are given in Table IV.1. These probabilities should be greater than 0.05. The data for Sample = LWEM and Method = LAB and for Sample = 2-8, and Method = LAB have a low probability (i.e., 0.040 and 0.0001, respectively). The cause of this low probability for Sample = 2-8 is that no mercury was measured (e.g., all values are either 0 or 1). The mercury values for the x-ray methods must be due to interferences which are causing false positive readings. The mercury data for Sample = 2-8 will not be used for other parts of the equivalency petition procedure. A summary table of the data is as follows:

Table IV.1

Summary Statistics for Lead Measurements

Sample	Method	N	Mean	St. Dev.	Min	Max	W-statistic
LWEM µg/mL	CSI	20	84	5	74	95	0.776
	LAB	20	102	11	77	116	0.040
	USA	20	79	5	70	89	0.616
2-8 µg/g	CSI	20	194	20	163	232	0.571
	LAB	20	0.5	0.5	0	1	0.0001
	USA	20	313	60	222	437	0.582

2. Equality of Replicate Variance: The least-squares fit of $Log(S_{ij}) = a \log(M_{ij}) + b$ to the logarithms of the mercury concentrations did not indicate any significant slopes. Therefore, the inference from this analysis is that the assumption of equality of variance can be made for mercury concentrations for Sample = LWEM and all methods.

3. Variance Ratio - 95% Confidence Interval: The 95% confidence intervals of the ratio of the variance for the proposed method (i.e., CSI and USA) to the variance of the reference method (i.e., LAB) are given in Table IV.2. These results show that there are no significant differences between the proposed method and accepted method for all methods used for Sample = LWEM.

Table IV.2

The 95% Confidence Intervals of the Proposed-Method Variances to Reference Method Variances

Sample	Method	Lower Limit	Ratio	Upper Limit	Proposed Variance	Accepted Variance	Num Df	Den Df
LWEM	CSI	0.46	1.72	6.39	36.1	21.0	10	10
LWEM	USA	0.28	1.05	3.91	22.1	21.0	10	10

4. <u>Bias Test by ANOVA</u>: According to the EPA Equivalency Petition, the bias test for the proposed methods can only be performed on those cases that have equivalent precisions (see, part 3). However, the ANOVA test is fairly robust to deviations in the assumptions of equivalent variances, and normality. Therefore, all methods will be examined by the ANOVA procedure. The ANOVA results are summarized in Table IV.3 by listing the probability values of the F-test for each source of variation. A probability value of less than 0.05 (i.e., 5% significance level) indicates a significant difference among the levels of the sources of variation. A significant METHOD x DAY interaction indicates that the results varied as a function the different results of the day on which the determinations were made. If the interaction source is not significant, a pooled error term is used to test the METHOD effect. For the mercury measurements for sample LWEM, all sources of variations are significant at the 5% significant level for both proposed methods. Therefore, the proposed methods for measuring lead would fail the equivalent petition criteria. The data are illustrated in Fig. IV.1 by a box plot which support the equivalent petition results.

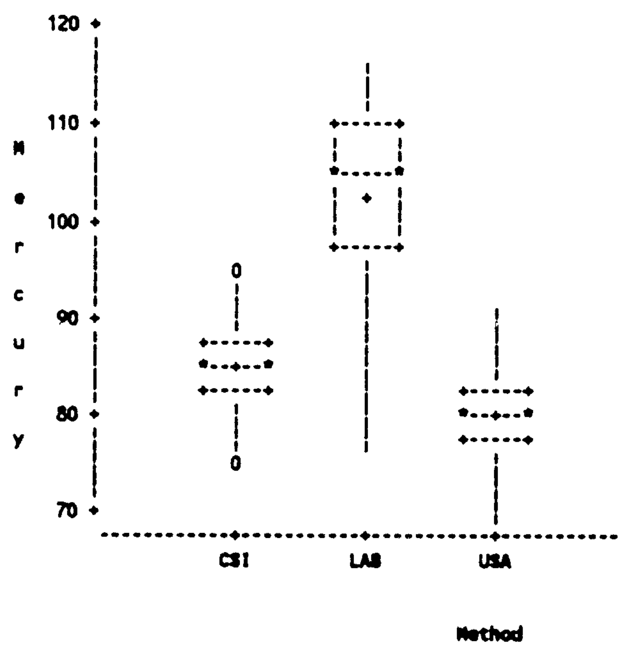

Fig. IV.1. Mercury measurements (µg/mL) for sample LWEM.

Table IV.3

Probability Values for the Sources of Variation in the ANOVA Table

Method	Source of Variation	LWEM μg/mL Copper	LWEM μg/mL Lead	2-8 μg/g Lead	LWEM μg/mL Mercury
CSI vs Lab	Method	0.0001	0.0942	0.5173	0.0001
	Day	0.0001	0.0009	0.4935	0.0012
	Method X Day	0.0003	0.0034	0.2536	0.0115
USA vs Lab	Method	0.0001	0.3232	0.0456	0.0001
	Day	0.0001	0.3852	0.7384	0.0004
	Method X Day	0.0001	0.7327	0.7253	0.0008

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