UNCONTROLLED

WEST VALLEY NUCLEAR SERVICES., INC

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ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

> Analysis of Wastes and Water with ICP Spectrometry

Approved by:

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Part I

1.0 PURPOSE

> This method is used for the elemental analysis of wastes and water using an inductively coupled plasma spectrometer (ICP). Because of the large calibration range (4 to 5 orders of magnitude) and the simultaneous capabilities of the instrument, it is ideal for many types of samples.

2.0 SCOPE AND APPLICATION

2.1 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1,500 mg/L.

2.2 Total elements are determined after appropriate digescion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps must be taken to correct for potential interference effects.

> 2.3 Attachment A lists elements along with recommended wavelengths and required detection levels.

2.4 No detailed instrumental operating instructions are provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the ICP.

3.0 Discussion

3.1 The method describes a technique for the simultaneous or sequential multielement determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical

spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency Inductively Coupled Plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference. Background correction is not required in cases of line broadening or where a background correction measurement would actually degrade the analytical result.

3.2 DEFINITIONS

3.2.1 Dissolved - Those elements which will pass through a 0.45 μm membrane filter.

3.2.2 Suspended - Those elements which are retained by a 0.45 μm membrane filter.

3.2.3 Total - The concentration determined on an unfiltered sample following vigorous digestion.

3.2.4 Instrumental Detection Limits - The instrumental detection limits are determined by multiplying by three the average of the standard deviation obtained from the analysis of a standard solution at a concentration 3 to 5 times the instrument detection limits. This measurement will define the sensitivity for the ICP.

3.2.5 Sensitivity - The slope of the analytical curve, i.e., it is the relationship between emission intensity and concentration.

3.2.6 Instrument Check Standard - A multielement standard of known concentration prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 6.6.1.)

> 3.2.7 Interference Check Sample - A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors. (See 6.6.1).

> 3.2.8 Quality Control Sample - A solution obtained from an outside source having known concentration values to be used to verify the calibration standards. (See 6.6.2.)

3.2.9 Linear Dynamic Range - The concentration range over which the analytical curve remains linear.

3.2.10 Reagent Blank - A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 6.5.2.)

3.2.11 Calibration Blank - A volume of deionized, distilled water acidified with HNO_3. (See 6.5.1.)

3.2.12 Method of Standard Addition - The standard addition technique involves the use of the unknown and the unknown-plus-a-known amount of standard by adding known amounts of standard to one or more aliquots of the processed sample solution.

3.2.13 Independent Standard - A standard composed of the analytes from a different source than those used in the standards used for the initial calibration.

3.3 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

3.3.1 In the collection of samples it is important to perform sample preservation to prevent loss of analyte.

3.2.1.1 Aqueous samples collected for the determination of dissolved elements must be filtered through a 0.45 μm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Use the first 50 to 100 mL to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1 + 1) HNO_3 to a pH of two or less. Normally, 3 mL of (1 + 1) acid per litre should be sufficient to preserve the sample.

3.2.1.2 Solid samples collected for the determination of total metals should be mixed thoroughly to achieve homogeneity.

3.3 INTERFERENCES

3.3.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

3.3.2.1 Spectral interferences can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the

monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effect can usually be compensated by a background correction adjacent to the analyte line.

3.3.2.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump will lessen these interferences.

3.3.2.3 Chemical interferences are characteristic by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not prominent with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

4.0 REFERENCES

(1) Winge, R. K., Peterson, V. J., and Fassel, V. A., "Inductively Coupled Plasma-Atomic Emission Spectroscopy Prominent Lines," EPA-600/4-79-017.

(2) Winefordner, J. D., "Trace Analysis: Spectroscopic Methods for Elements," Chemical Analysis, Volume 46, pp. 41-42.

(3) Handbook for Analytical Quality Control in Water and Wastewater Laboratories, PEA600/4-79-019.

(4) Garbarino, J. R., and Taylor, H. E., "An Inductively Coupled Plasma Atomic Emission Spectrometric Method for Routine Water Quality Testing", Applied Spectroscopy 33, No. 3 (1979).

(5) "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020.

(6) Annual Book of ASTM Standards, Part 31.

(7) "Carcinogens - Working with Carcinogens", Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

(8) "OSHA Safety and Health Standards, General Industry", (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised January 1976).

(9) "Safety in Academic Chemistry Laboratories", American Chemical Society Publications, Committee on Chemical Safety, 3rd Edition, 1979.

(10) "Inductively Coupled Plasma-Atomic Emission Spectrometric Method of Trace Element Analysis of Water and Waste", Method 200.7 modified by CLP Inorganic Data/Protocol Review Committee, original method by Theodore D. Martin, EMSL/Cincinnati.

(11) "Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Aqueous and Solid Samples", modified from the Contract Laboratory Program, SOW No. 785, by P. C. Lindahl, Argonne National Laboratory.

Part II

5.0 EQUIPMENT

5.1 Inductively Coupled Plasma-Atomic Emission Spectrometer

5.1.1 Computer controlled Inductively Coupled Plasma Spectrometer with background correction.

5.1.2 Nitrogen gas supply (High Purity)

5.1.3 Argon gas supply, welding grade or better. (Liquid argon is preferred.)

5.1.4 Peristaltic Pump

5.1.5 Printer

5.1.6 Mass Flow Controller

5.1.7 Exhaust Duct

5.1.8 HF Resistant Torch Assembly

6.0 REAGENTS AND STANDARDS

NOTE: All standards, solutions and blanks are prepared according to ACP 8.1.

6.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Ultrapure acids are recommended and may be necessary for trace element analysis.

6.1.1 Hydrofluoric Acid - (48 - 51°) Reagent Grade

6.1.2 Hydrochloric Acid - Conc. (sp gr 1.19)

6.1.3 (1 + 1) HCl - Add 50 mL of hydrochloric acid to 50 mL H_2O (6.2)

6.1.4 Nitric Acid - Conc. (sp gr 1.41)

6.1.5 (1 + 1) HNO_3 - Add 50 mL of nitric acid to 50 mL H_2O (6.2)

6.2 Deionized, distilled water - Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards, and as dilution water. The purity of this water must be equivalent to ASTM Type II reagent water of Specification D 1193 (4.6).

6.3 Standard stock solutions may be purchased or prepared from ultra high purity grade chemicals or metals. (See ACP 8.1.) These will have a shelf life of one year.

> 6.4 Mixed calibration standard solutions - Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in acid-cleaned volumetric flashs. Add appropriate concentrations of acid(s) and dilute to 100 mL with deionized, distilled water. An acid concentration of 2 percent HNO_3 is recommended. Calibrated Eppendorf pipets should be used when preparing these solutions and care should be taken that the elements are compatable and stable. Transfer the mixed standard solutions to a teflon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging. The upper limit on the expiration date is the expiration date of the oldest stock solution used to prepare the calibration standard. Label the standard per ACP 7.1. Calibration standards must be initialy verified using a quality control sample.

> NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of deionized, distilled water and warm the flask until the solution clears. Cool and dilute to 100 mL with deionized, distilled water. For this acid matrix the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require the addition of HCl.

6.5 Two types of blanks are required for the analysis. The calibration blank (3.2.11) is used in establishing the analytical curve while the reagent blank (preparation blank, 3.2.10) is used to correct for possible contamination resulting from varying amount of the acids used in the sample processing.

6.5.1 The calibration blank is prepared by diluting 2 mL of (1 + 1) HNO_3 to 100 mL with deionized, distilled water. Prepare a sufficient quantity to be used to flush the system between standards and samples.

6.5.2 The reagent blank (or preparation blank) must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

6.6 In addition to the calibration standards, an instrument check standard (3.2.6) and a quality control sample (3.2.8) are also required for the analyses. These standards will have a shelf life of one year.

NOTE: Poor results on the quality control or instrument check standard may indicate either a bad calibration standard or quality control samples. This would indicate a possible incompatibility among analytes in the solution.

6.6.1 The instrument check standard for continuing calibration verification is prepared by the analyst by combining compatible elements at a concentration near the mid-point of their respective calibration curves. (See 9.3.1.3.)

6.6.2 The quality control sample for the initial calibration verification should be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier. (See 9.3.1.1.)

7.0 SAFETY

7.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard.* Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands throughly after handling.

7.2 An exhaust duct must be operating for the ICP.

7.3 Sample digestions are done in fume hoods using usual laboratory safety precautions (ACP 7.2).

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* See ACP 7.2, Laboratory Safety.

8.0 RECORDS

8.1 All measurement data and sample identification shall be recorded on the work sheet attachment A. The final result shall be recorded on the analytical request sheet (PRD 5.1).

9.0 CALIBRATION

9.1 Calibration Standards - Prepare mixed calibration standard solutions as in 6.4. If hydrofluoric acid is present, the standard must be prepared using plastic labware. For analysis, the HF resistant torch and nebulizer must be used.

9.1.2 Concentrations for the calibration solutions should be set at a level near the point of interest. A minimum concentration for the calibration standards would be approximately 1 ppm.

9.2 Instrument Operating Conditions - The analyst should follow the instructions provided by the manufacturer of the instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line. All measurements must be within the instrument linear range where correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results. General instrument calibration should include:

9.2.1 Set up instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 20 minutes of operation prior to calibration.

> An Example of Proper Operating Parameters

Output Power	1.1 KW
Reflected Power	<10.0 W
Argon Flow (Plasma)	16.0 ℓ min^{-1}
> Mass Flow Controller	0.7 ℓ min^{-1}

9.2.2 Initiate appropriate operating configurations of computer.

9.2.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures using a standard solution such as those described in section 6.4.

9.2.4 Begin the sample run flushing the system with distilled water between each sample. Analyze the instrument check standard (6.6.1) and the calibration blank (6.5.1) with each 20 samples.

9.3 Quality Assurance/Quality Control

9.3.1 Check the instrument standardization by analyzing appropriate quality control check standards as follows:

> 9.3.1.1 A quality control standard (6.6.3) must be used daily for the initial calibration verification. Analyses shall be conducted on an independent standard at a concentration other than that used for calibration, but within the calibration range. For ICP, the Initial Calibration Verification Solution(s) must be run at each wavelength used in the analysis of the sample. When measurements exceed the control limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. (See ACP 8.2.)

9.3.1.2 Analyze the calibration blank (6.5.1) at a frequency of 10 percent. The result should be within ±5 times the instrument detection (table 1). If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument.

> 9.3.1.3 For continuing calibration verification, analyze an appropriate instrument check standard (6.6.1) containing the elements of interest at a frequency of 10 percent. This check standard is used to determine instrument drift. If agreement is not within ±10 percent of the expected values for all elements of interest, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

> 9.3.1.4 The spiked sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology.[3] The spike is added before the digestion steps. At least one spiked sample

>[3] Sample spikes and blanks are only required for SPDES analysis.

analysis must be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) for each group of samples or for each 20 samples received, whichever is more frequent. Samples identified as field blanks cannot be used for spiked sample analysis. If the spike recovery is not within the limits of 75 to 125 percent, the data of all samples received associated with that spiked sample must be flagged with the letter "N" on attachments B and C. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such a case, the spike recovery should not be considered and the data shall be reported unflagged even if the percent recovery does not meet the 75 to 125 percent recovery criteria. In the instance where there is more than one spiked sample per matrix per group, if one spike sample recovery is not within contract criteria, flag all the samples of the same matrix in the group. Individual component percent recoveries (%R) are calculated as follows:

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

9.3.1.5 At least one duplicate sample must be analyzed from each group of samples of a similar matrix type (i.e., water, soil) for each batch of samples or for each 20 samples received, whichever is more frequent.(4) Samples identified as field blanks cannot be used for duplicate sample analysis. The Relative Percent Difference (RPD) for each component are calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

(4) Duplicate samples are only required for SPDES analysis.

Where,

RPD = Relative Percent Difference
D_1 = First Sample Value
D_2 = Second Sample Value (duplicate)

> The results of the duplicate sample analysis must be reported on attachment D only if they are outside the control limits. A control limit of ±20 percent for RPD shall be used for sample values greater than 5 times the Contract Required Detection Level (CRDL). A control limit of ± the CLP CRDL shall be used for sample values less than 5 times the CRDL (table 1), and this control limit (±CRDL) should be entered in the "Control Limit" column on attachment D. If one result is above the 5 x CRDL level and the other is below, use the ±CRDL criteria. If either sample value is less than the CRDL, the RPD is not calculated and is indicated as "NC" on attachment D.

> If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*" on attachments B and D.

10.0 PROCEDURE

> 10.1 Aqueous Samples

10.1.1 For the determination of elements in aqueous samples, shake sample and transfer 100 mL mixed sample to a 250 mL beaker. Add 2 mL of HNO_3 to the sample. Heat on a hot plate until the volume has been reduced to

Between 25 and 50 mL making certain the sample does not boil. After this treatment, cool sample and filter to remove insoluble material that could clog the nebulizer. Adjust the volume to 100 mL(1) with deionized distilled water. The sample is now ready for analysis.(2)

Concentrations so determined shall be reported as "total."

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(1) For trace analysis, the volume may be reduced to 20 mL.
(2) Samples may be spiked with Sc (5 ppm) as an internal standard, which the results can be ratioed against.

NOTE: In place of filtering, the sample after dilution and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

10.2 Solid Samples

10.2.1 For the determination of elements in solid samples, e.g., sediments, sludges, and soils, mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 gms) a 1.0 to 1.5 g portion of sample and transfer to a 100 mL beaker.

Add 10 mL of (1:1) nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker.

After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3 mL of 30 percent hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

Continue to add 30 percent H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30 percent H_2O_2.)

Next, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered beaker to the hot plate, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent, or centrifuge the sample). Dilute the filtrate (100 mL final volume) with deionized water. The sample is now ready for analysis.(2)

10.2.2 For the analysis of glass and melter feed, see ACM-MICRO-1901 and ACM-FUSION-3301.

10.3 Report Forms

10.3.1 All reports in the sample data package must be submitted in a legible form. The data report package for analyses of each sample must be complete before submission and shall include:

> 10.3.1 Tabulated results in mg/L or µg/L for aqueous samples or µg/g or percent for solid samples (identification and quantity) of specified chemical constituents by the specified analyses. The results for solid samples will be reported on a dry weight basis. If the duplicate sample analysis is not within the control limits, flag it with an asterisk (*). If the spike sample recovery is not within control limits, flag the data with the letter N. Note any sample problems in the comments section. Report results to two significant figures for values from 0 to 0.90 and three significant figures for results greater than 1.00. For rounding rules, follow the EPA Handbook of Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/4-79-019).

> 10.3.2 The spike sample recovery should be reported on attachment C Spike Sample Recovery and included in the data package.

> 10.3.3 The duplicate sample analysis results should be reported on attachment D and included in the data package.

11.0 CALCULATIONS

11.1 Reagent blanks (preparation blanks) should be analyzed for each batch and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

11.1.1 If the concentration of the blank is less than or equal to the instrument detection level, no correction of sample results is performed.

11.1.2 If the concentration of the blank is above the instrument detection level: For any group of samples associated with a particular blank, the concentration of the sample with the least concentrated analyte must be 10 times the blank concentration, or all samples associated with the blank and less than 10 times the blank concentration must be redigested and reanalyzed, with the exception of an identified aqueous-blank value and the fact that a blank concentration was observed documented in the comments section of the report.

11.2 If dilutions or concentrations were performed, the appropriate factor must be applied to sample values.

11.3 The concentration determined in the digest for solid samples are to be reported on the basis of the dry weight of the sample.

$$\text{Concentration (dry wt.) (mg/kg)} = \frac{C \times V}{W \times S} \qquad \text{OR} \qquad \% = \frac{C \times V}{W \times 10000}$$

Where,

C = Concentration (mg/L)
V = Final volume in millilitres after sample preparation
W = Weight in grams of sample
S = % Solids/100 (attachment 1)

12.0 ATTACHMENTS

12.1 Attachment A - "Elements Determined by Inductively Coupled Plasma Emission" (SPDES)

> 12.2 Attachment B - ICP Worksheet (SPDES)

> 12.3 Attachment C - Spike Recovery Worksheet

> 12.4 Attachment D - Duplicates Worksheet

> 12.5 Attachment E - "Percent Solids"

> 12.6 Deleted

ATTACHMENT A

TABLE 1: ELEMENTS DETERMINED BY INDUCTIVELY COUPLED PLASMA EMISSION (SPDES)

Element	Wavelength, nm	Required Detection Levels[1] (µg/L)
Aluminum	396.15	200
Arsenic	188.98	25
Barium	233.53	200
Cadmium	214.438	20
Chromium	267.72	50
Copper	324.75	25
Iron	259.94	100
Lead	220.35	100
Manganese	257.61	50
Nickel	231.60	40
Selenium	196.03	40
Silver	328.07	20
Zinc	213.86	20

[1] These RDL are the instrument detection limits obtained in pure water that must be met using the procedure. The detection limits for samples may be considerably higher depending on the sample matrix.

> ATTACHMENT B Page ___ of ___

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ICP WORKSHEET SPDES

SAMPLE NAME ____________________ LOG NUMBER ____________________
SAMPLE WEIGHT ____________________ SAMPLE MATRIX ____________________
SPECIAL INSTRUCTIONS ____

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* *

ELEMENT	CONC. ALIQUOT	CONC. SAMPLE	ELEMENT	CONC. ALIQUOT	CONC. SAMPLE
Ag			As		
Ba			Pb		
Cd			Cr		
Fe			Se		
Al			Cu		
Mn			Ni		
Zn					

ANALYST ____________________ DATE ____________________
APPROVED ____________________ DATE ____________________

> ATTACHMENT C Page ___ of ___
>

SPIKE SAMPLE RECOVERY

Lab Name _____ Lab Sample ID No. _____

Date _____ Units _____

Matrix _____

Compound	Control Limit %R	Spiked Sample Result (SSR)	Sample Result (SR)	Spiked Added (SA)	%R[1]
Metals:					
1. Aluminum	75 - 125				
2. Arsenic	75 - 125				
3. Barium	75 - 125				
4. Cadmium	75 - 125				
5. Chromium	75 - 125				
6. Copper	75 - 125				
7. Iron	75 - 125				
8. Lead	75 - 125				
9. Manganese	75 - 125				
10. Nickel	75 - 125				
11. Selenium	75 - 125				
12. Silver	75 - 125				
13. Zinc	75 - 125				
Other:					

[1] %R = [(SSR - SR)/SA] x 100
"N" = out of control
"NR" = Not required
Comments: _____

> ATTACHMENT D Page ___ of ___
>

DUPLICATES

Lab Name _____ Lab Sample ID No. _____

Date _____ Units _____

Matrix _____

Compound	Control Limit[1]	Sample (S)	Duplicate (D)	RPD[2]
Metals:				
1. Aluminum				
2. Arsenic				
3. Barium				
4. Cadmium				
5. Chromium				
6. Copper				
7. Iron				
8. Lead				
9. Manganese				
10. Nickel				
11. Selenium				
12. Silver				
13. Zinc				
Other: _____				

*Out of Control
[1]To be added at a later date.
[2]RPD = [|S - D|/((S + D)/2)] x 100
NC = Non calculated RPD due to value(s) less than CRDL.

> ATTACHMENT E

PERCENT (%) SOLIDS

1. Add a portion of the sample to a tared weighing dish. Weigh and record the weight.

2. Place weighing dish plus sample, with the cover tipped to allow for moisture escape, in a drying oven that is set at 103 to 105°C. Perform this task in a well ventilated area.

3. Dry the sample to constant weight. Cool the sample in a desicator with the weighing dish cover in place before each weighing. Record each weight. Do not analyze the dried sample.

4. Calculate and report data on a dry weight basis. Also report the percent solids for each sample.

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

FORM A

ICP WORKSHEET

DELETED