12.2.3 Matrix Duplicate (DUP)

- the sample must be reprocessed and reanalyzed unless the sample concentration is <5 times the Reporting Limit, then the ± Reporting Limit rule applies;
- if the reanalysis is within the control limits, the second value is reported;
- if the reanalysis is still outside of the control limits, a CAR must be written and then approved by your Section Manager or Unit Leader.

12.2.4 Matrix Spike (MS)

- the sample must then be reprocessed and reanalyzed unless the sample concentration exceeds the spike concentration by a factor of 4 times;
- the original spike results must be entered onto the spreadsheet with the "S" code even though the control limits were exceeded;
- the reanalysis result must be entered onto the spreadsheet using the "T" code regardless of whether it is within the control limits. There is no need to write a corrective action if both the "S" and "T" codes were entered into LIMS.

13.0 HEALTH AND SAFETY

As always, general laboratory safety practices should always be followed. Waste samples should be handled with care due to the uncertainty of the properties and contents involved. Refer to the specific MSDS for the hazardous properties of any chemical or reagent involved in this procedure.

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INORGANIC ANALYSIS PROTOCOL
Analysis of Metals by Inductively Coupled Argon Plasma (ICAP) (Simultaneous Operation)

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REVISION NUMBER: 02

1.0 PURPOSE

To outline the guidelines for determining metal concentrations by Inductively Coupled Argon Plasma (ICAP) Emission Spectrometry - Simultaneous Operation.

2.0 REFERENCE

This SOP was written using USEPA SW-846 "Test Methods for Evaluating Solid Waste", Method 6010 as a reference.

3.0 METHOD SUMMARY

ICP is a technique for the analysis of solubilized or digested samples for metal concentrations using atomic emission spectrometry. All matrices including water, TCLP extracts, wastes, soils, sludges and sediments require digestion prior to analysis. The digestion procedures are listed in separate SOPs. The most common digestion procedures are SW-846 Methods 3010 and 3050. The instrument (Simultaneous ICP) is capable of analyzing simultaneously 29 different elements on a sample.

3.1 Parameter List

Element	Wavelength (nm) ICAP 1100	Reporting Limit (ug/L)
Ag	328.0	30
Al	308.2	200
As	193.6	100
B	249.6	50
Ba	493.4	50
Be	313.0	5
[1]Ca	315.8	100
Cd	228.8	5
Co	228.6	20
Cr	267.7	20
Cu	324.7	20
Fe	259.9	30
K	766.4	2000
Li	670.7	100
Mg	279.0	200
Mn	257.6	10
Mo	202.0	100
[1]Na	589.5	200
Ni	231.6	20
P	214.9	200
Pb	220.3	50
Sb	206.8	100
Sc	261.4	*
Se	196.0	100
[1]Si	288.1	100
Sn	189.9	100
Sr	421.5	100
Ti	334.9	10
[1]Tl	377.5	500
V	292.4	10
Y	371.0	*
Zn	213.8	10

* Sc is used as an internal standard on ICP 61.
* Y is used as an internal standard on ICP 1100.

[1]Alternate Wavelengths are used for ICAP 61:

Ca 317.9	Si 251.6
Na 588.9	Tl 190.8

The reporting limits that are listed above are at a level where the ICP can accurately quantitate at a concentration five times that listed.

Actual instrument detection limits (IDL) and linear ranges are also determined quarterly. The IDLs are determined by analyzing a solution at a concentration of five times the reporting limit (listed above) seven times on three non-consecutive days. The IDL is then calculated as 3 times the average standard deviation of these readings. The actual IDLs are always below the reporting limits.

4.0 INTERFERENCES

Spectral, Physical and Chemical Interferences are the three main interferences that are commonly present on the ICP.

4.1 Spectral Interferences

Mainly caused by continuous background wavelength, stray light from a high concentration element or overlap of a spectral line from another element. The ICP can correct for the first two types of interferences by using background correction adjacent to the wavelength. Spectral overlap can be corrected by monitoring the interfering wavelength and computer correcting the results for the false concentration. The values used to correct are known as Inter-element Correction Factors or IEC's.

4.2 Physical Interferences

Usually associated with the sample uptake and nebulization processes. These interferences can usually be eliminated by using a peristaltic pump which assures a constant sample uptake rate. If a sample is extremely viscous or contains a very high dissolved solids concentration, a dilution of the sample may be required to assure a constant and smooth nebulization rate.

4.3 Chemical Interferences

Normally not significant on the ICP. These interferences include ionization effects and molecular compound formation. Chemical interferences are highly dependent on the sample matrix type and the element.

Most interferences can be corrected by ensuring a constant sample uptake rate and by using the correcting abilities of the computer. If severe interferences are suspected, an alternate method such as flame AA or graphite furnace AA can be used or to verify the ICP results.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests.

6.0 INSTRUMENTATION AND EQUIPMENT

The instruments used are the ICAP 1100 and the ICP 61 which are made by Thermo Jarrell Ash. The instruments are simultaneous ICP's which currently have 29 analytical wavelengths. Additional wavelengths may be added as required.

The instruments are operated via a HAST 386 computer system and Thermo Jarrell Ash software. They also come equipped with a peristaltic pump for sample uptake and an autosampler.

7.0 PREVENTIVE MAINTENANCE

The required preventive maintenance is listed in the preventive maintenance notebook which is kept at the instrument. All maintenance is recorded in this notebook along with the date and the signature of the analyst preforming the maintenance.

7.1 Daily Maintenance

Includes changing the pump winding for consistent sample uptake and a visible check of the waste container to make sure that it doesn't overflow.

7.2 Weekly Maintenance

Includes checking the air filters on the back of the instrument for excessive dust buildup, checking the tip of the torch for excessive buildup of material, and dusting the instrument.

7.3 Monthly Maintenance

Includes cleaning and checking the water recirculator for proper fluid level, cleaning the spray chamber and checking and replacing the printer ribbon as necessary.

The instruments are under a full service contract with the manufacturer for all major repairs.

8.0 STANDARDS AND QC SOLUTIONS

8.1 Stock Standards and QC solutions

All stock standards and QC solutions are purchased from an outside supplier in aqueous form. Two types of standards are used: single element and custom mixed standards. The suppliers that are currently used are Inorganic Ventures, and SPEX. Single element standards are available for most elements at a 1000 mg/L concentration.

The shelf life of all purchased solutions is one year from receipt.

8.2 Calibration Standards

Prepared with Milli-Q water that has been acidified to the same concentrations as the samples. All calibration standards are spiked with 10 ppm of internal standard. The calibration standards are prepared daily as follows:

8.2.1 Calibration Blank

Add approximately 500 mL of Milli-Q DI water to a 1000 mL Class A volumetric flask. Repipette 10 mL of concentrated nitric acid and 50 mL of concentrated hydrochloric acid into the flask. Bring up to volume with Milli-Q DI water and mix thoroughly.

8.2.2 Calibration Standard

Add approximately 50 mL of Milli-Q deionized water to a 100 mL Class A volumetric flask. Re-pipette 1 mL of nitric acid and 5 mL of hydrochloric acid into the flask. Using Eppendorf pipettes, add 1.0 mL of XWGC-9, 1.0 mL XWGC-10A, 1.0 mL XWGC-11 and 1.0 mL PLAL2-3X. Bring up to volume with Milli-Q water and mix thoroughly.

* See Appendix A for element concentrations.

8.3 QC Solutions

Prepared with Milli-Q water that has been acidified to the same concentration as the samples. All QC solutions are spiked with 10 ppm of internal standards. The QC solutions are prepared as required (at least weekly) as follows:

8.3.1 ICV/CCV: Initial and Continuing Calibration Verification

Prepared by adding approximately 250 mL of Milli-Q water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of hydrochloric acid. Add 5.0 mL WGC-CAL-1C, 5.0 mL WGC-CAL-1D, and 25 mL of Al (1000 ug/mL). Bring up to volume with Milli-Q water and mix thoroughly.

* See Appendix B for element concentrations.

8.3.2 CRI

Prepared by adding approximately 250 mL of Milli-Q water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of concentrated hydrochloric acid to the flask. Add 5.0 mL WGC-CRI-1, and 5.0 mL of WGC-CRI-2. Bring up to volume with Milli-Q water and mix thoroughly.

* See Appendix C for element concentrations.

8.3.3 50% and 20% Standards

Prepared by making a serial dilution of the stock calibration standards. These solutions are used to verify the linearity of the calibration curve.

8.3.4 ICSA: Interferant Check Standard

Prepared by adding 250 mL of Milli-Q deionized water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of concentrated hydrochloric acid. Add 50 mL of CLPP-ICS-A. Bring up to volume with Milli-Q water and mix the solution thoroughly.

8.3.5 ICSAB: Interferant Check Standard

Prepared by adding 250 mL of Milli-Q deionized water to a 500 mL Class A volumetric flask. Add 5 mL of concentrated nitric acid and 25 mL of concentrated hydrochloric acid. Add 50 mL of CLPP-ICS-A, 5.0 mL CLPP-ICS-B, 2.5 mL As (1000 ppm) and 2.5 mL Se (1000 ppm). Bring up to volume with Milli-Q water and mix thoroughly.

*See Appendix D for element concentrations for ICSA and ICSB Solutions.

9.0 PROCEDURE

9.1 Sample Preparation

The samples are digested according to the SOPs for the specific matrix type. The most commonly used digestion procedures are from EPA SW-846 Methods 3010 and 3050. The samples are checked for the proper preservation prior to digestion. The pH of the samples is checked using pH paper and the results (< or > 2) are recorded in the digestion notebook.

Prior to analysis all samples are spiked with 10 ppm of either Y or Sc (internal standard) depending on the instrument the samples will be analyzed on.

9.2 Instrument Set Up

Set up the instrument with the proper operating conditions as provided by Thermo Jarrell Ash. The instrument must be allowed to become thermally stable (usually requiring about an hour) prior to profiling and calibration.

Profile the instrument according to the manufacturer's recommended procedures

9.3 Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the linear range of the instrument.

The instrument is standardized using a calibration blank and calibration standard. The results are given in intensities.

9.4 Analytical Run

After the instrument is standardized, an analytical run is initiated. The first run of the day would proceed as follows:

STD	Reanalysis of calibration standard as a sample
ICV	Initial Calibration Verification
ICB	Initial Calibration Blank
CRI	Spiked Blank
ISA	Interferant Check Standard A
ISB	Interferant Check Standard B
20% STD	
50% STD	
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank
PB	Preparation Blank
LC1	Laboratory Control Sample
LC2	Laboratory Control Sample
Sample 1	
Sample 1	Duplicate (D)

Sample 1	Spike (S)
Sample 1	Serial Dilution (L)
Sample 2	
.	
.	
Sample 10	
CCV	Continuing Calibration Verification
CCB	Continuing Calibration Blank

* See Section 11.5 for acceptance criteria.

9.4.1 If the CCV and CCB results are acceptable, the run may continue without restandardization. If any of the post run QC is out of control, or close to being out of control, the instrument is restandardized before analyzing the next digestion. Any Samples with elements associated with an out of control CCV or CCB will be reanalyzed.

9.4.2 The CCV and CCB are run after every standardization, at a frequency of 10% during a run, and at the end of each analytical run.

9.4.3 The STD, 50% and 20% Standards are analyzed only at the beginning of each day. The CRI, ICSA and ICSB will be run at the beginning of the day and every 8 hours thereafter.

10.0 CALCULATIONS

The calculations for weight, volume, and dilution factors are performed by LIMS. The Sample ID is inputed for each sample. If a dilution is needed it is recorded on the run log and the dilution is put into LIMS via the data file.

Spreadsheets containing initial weight/volume and final volume along with % solids (if required) are updated in the LIMS system and this information is used to calculate results.

The sample results are stored in a data file on the HAST Computer. These files are then edited for any excess data or results, such as elements that were not required for a given sample. The edited file is then transferred directly to the LIMS system. This system helps to eliminate transcription errors, since data is not entered by hand.

10.1 Accuracy

10.1.1 ICV/CCV, LCS/LCSD % Recovery

$$\% R = \frac{\text{observed concentration}}{\text{actual concentration}} \times 100$$

10.1.2 Matrix Spike % Recovery

$$\% R = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

10.2 Precision

10.2.1 Matrix Dup. and LCS Dup. Relative Percent Difference (RPD)

$$RPD = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.0 QUALITY CONTROL

11.1 At least one Preparation Blank (PB) and at least two Laboratory Control Standards (LCS) will be included in each digestion batch of 20 samples. Regardless of the matrix being processed, the LCS and PB will be in an aqueous media. The Preparation Blanks are analyzed to determine if contaminants are being introduced into the sample via the Sample Preparation Procedures.

11.2 The Laboratory Control Samples are analyzed to determine both the precision and accuracy of the digestion process.

Precision will be measured by the reproducibility of both LCS results and will be calculated as Relative Percent Difference (RPD). Results must have a $\leq$ 20% RPD in order to be considered acceptable. If the LCS results are not within the control limits, all samples in the preparation set must be redigested and reanalyzed.

Statistical control limits are available and can be used per client request.

Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within +/- 20% of the known concentration. If either of the LCS results are outside the control limits, all samples in the preparation set must be redigested and reanalyzed.

* See Appendix E for element concentrations.

11.3 A duplicate sample will be prepared at a frequency of 5% (1 in 20 samples). RPD must be ±20%.

11.4 A spike sample will be prepared at a frequency of 5% (1 in 20 samples). Spike Recovery must be within 75 - 125%.

* See Appendix E for element concentrations.

11.5 A Serial Dilution (4X) will be prepared from the digestate at a frequency of 5% (1 in 20 samples). If the concentration is >10 times the IDL, results should agree within +/- 10% of the original results.

11.6 The acceptance limits for each solution are summarized below:

STD	±5% of the known concentration
ICV	±10% of the known concentration
ICB	+/- reporting limit
CRI	No limits have been established
50% and 20%	The linear regression curve for the high standard, 50% and 20% standards must have a correlation coefficient of ≥ 0.995.
ICSA	±20% of the known concentration
ICSB	±20% of the known concentration
CCV	±10% of the known concentration

CCB	+/- reporting limit
Matrix Dup.	If ≥ 5X reporting limit 20% RPD, if < 5X reporting limit ± reporting limit, if < reporting limit no control range
Matrix Spike	If sample concentration is ≤ 4X spike level 75-125%, if sample concentration is > 4X spike level no control range. If TCLP matrix spike is < 50%, Standard Addition must be performed.
Serial Dilution	If the analyte concentration is >10X the IDL, results should agree within +/- 10% of the original sample result.
LCS	80-120% of the known concentration
PB	± reporting limit

Currently, the acceptance criteria are at fixed control limits. Statistical limits are also produced by the QC department and are used when specified by the client or contract.

11.6 Metals Standards Traceability

Custom made and single element stock standard solution which are traceable to NIST or EPA are purchased. On receipt, each standard is recorded in a bound standards log book and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are documented in the log book.

12.0 CORRECTIVE ACTION

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out of control situation. The test must not be resumed

until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the section manager.

12.1 Listed below are steps to be taken when an out of control situation occurs. The analyst must:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a corrective action report form;
- document on the corrective action report that an in control has been achieved; and
- receive approval (signature) of the unit leader, QA personnel, or the section manager prior to the release of any analytical data associated with the problem.

12.2 Suggested Actions to specific out of control situations:

12.2.1 Calibration Curve

- reanalyze the standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

12.2.2 Initial Calibration Verification (ICV)

- repeat ICV to verify proper preparation;
- prepare new ICV from original stock;
- recalibrate with a new standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

12.2.3 Initial Calibration Blank (ICB)

- prepare new ICB to verify proper preparation;
- verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability;
- determine the source of contamination by process of elimination, carryover from a previous analysis or reagent contamination and correct the problem;
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze the ICB and any associated samples.

12.2.4 Laboratory Control Standards (LCS)

If the LCS is low:
- reanalyze the LCS to verify that it is out of control;
- determine the source of error within the preparation procedure, repeat the sample set, write a CAR.

If the LCS is high:
- reanalyze the LCS to verify that it is out of control;
- determine the source of error within the preparation procedure, repeat the sample set;
- determine if the high value is due to contamination;
- check for contamination of reagents, LCS stock solution, or in the preparation area;
- correct for contamination, reanalyze;
- Write a CAR.

12.2.5 Laboratory Control Standard Duplicate (LCSD)

- Must meet all requirements and control limits as the LCS in addition to limits set for precision.

- Precision: if precision is out of control, initiate the same actions specified for the LCS.

12.2.6 Preparation Blank (PB)

- reanalyze the PB to verify that it is beyond the detection limit;
- determine the source of contamination;
- determine if a high value is due to contamination;
- check for contamination of reagents or in the preparation area;
- correct for contamination, reanalyze the set;
- in the extreme case where all samples in the set are at least ten times greater than the PB, reanalysis will not be required; however, a corrective action report and approval will be necessary.

12.2.7 Matrix Duplicate (DUP)

- a CAR will be written and approved by the Unit Leader. Sample will be ticked with a "*."

12.2.8 Matrix Spike (MS)

- A CAR will be written and approved by the Unit Leader. Sample will be ticked with a "*."

12.2.9 Serial Dilution (L)

- prepare a new serial dilution to verify proper preparation;
- a car will be written and approved by the unit leader or section manager.

12.2.10 Continuing Calibration Verification (CCV)

- repeat the CCV to verify proper preparation;
- prepare new a CCV from the original stock;
- check for instrument base-line drift or a change in one or more of the reagents;
- check reagents/solutions and prepare fresh if necessary;
- recalibrate with a new standard curve and repeat all samples since the previous in control CCV;
- never dispose of any samples until you are sure that all QC are within the control limits.

12.2.11 Continuing Calibration Blank (CCB)

- prepare a new CCB to verify proper preparation;
- verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc., to achieve stability;
- determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct problem;
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze the CCB and any associated samples;
- never dispose of any samples until you are sure that all QC are within the control limits.

If any of the ICV, ICB, Spike Blank, 50% standard, 20% standard, CCV or CCB results are out of control for any element, the instrument is restandardized and the samples associated with the out of control elements are reanalyzed.

If the PB or LCS are out of control for any element, the samples are redigested. An exception is if the sample concentrations are $\geq$ 10X the PB contamination. In this case, the results are reported as is.

If any of the Matrix Duplicate or Matrix Spike results are out of control, the client is notified of the poor results via a case narrative that is sent with the data report.

Corrective Action Report (CAR) forms are available for poor PB, LCS, matrix dup and matrix spike problems. These forms are completed by the analyst performing the analysis. The forms are then reviewed and signed by the unit leader. The signed forms are kept on file in the QC department.

Additional, detailed information on corrective action procedures is listed in Appendix B.

13. HEALTH AND SAFETY

Material Safety Data Sheets are available for all reagents.

Acids should be handled with care.

The standards contain potentially harmful elements. Care should be taken to avoid contact with the stock solutions. Wash hands well if contacted.

The ICP torch puts out harmful ultraviolet radiation. The torch should never be looked at directly without proper eye protection.

APPENDIX A

Standard Stock Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	Calibration Std. Conc. (mg/L)
Spex	XWGC-9	B	1000	10
		Ba	2000	20
		Be	500	5
		Ca	10000	100
		K	10000	100
		Li	1000	10
		Mg	10000	100
		Na	10000	100
		Se	1000	10
		Sr	1000	10
Spex	XWGC-10A	As	1000	10
		Pb	1000	10
		Sb	200	2
		Si	1000	10
		Sn	1000	10
		Tl	2000	20
Spex	XWGC-11	Ag	200	2
		Cd	500	5
		Co	1000	10
		Cr	1000	10
		Cu	1000	10
		Fe	5000	50
		Mn	1000	10
		Mo	1000	10
		Ni	1000	10
		Ti	1000	10
		V	1000	10
		Zn	1000	10
Spex	PLAL2-3x	Al	10000	100

APPENDIX B

Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICV/CCV Conc. (mg/L)
Inorganic Ventures	WGC-CAL-1C	Ca	5000	50
		Mg	5000	50
		K	5000	50
		Na	5000	50
		Fe	2500	25
		Ba	1000	10
		Tl	1000	10
		As	500	5
		Cr	500	5
		Co	500	5
		Cu	500	5
		Pb	500	5
		Li	500	5
		Mn	500	5
		Ni	500	5
		Se	500	5
		Sr	500	5
		V	500	5
		Zn	500	5
		Be	250	2.5
		Cd	250	2.5
		Ag	100	1
Inorganic Ventures	WGC-CAL-1D	B	500	5
		Mo	500	5
		Si	500	5
		Sn	500	5
		Ti	500	5
		Sb	100	1
Inorganic Ventures	Aluminum	Al	1000	50

APPENDIX C

Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	CRI Conc. (mg/L)
Inorganic Ventures	WGC-CRI-1	Ca	200	2
		Mg	200	2
		K	200	2
		Na	200	2
		Al	40	0.4
		Ba	40	0.4
		Tl	40	0.4
		As	20	0.2
		Fe	20	0.2
		Li	20	0.2
		Se	20	0.2
		Sr	20	0.2
		Co	10	0.1
		Pb	10	0.1
		V	10	0.1
		Ni	8	0.08
		Cu	5	0.05
		Zn	4	0.04
		Mn	3	0.03
		Cr	2	0.02
		Ag	2	0.02
		Be	1	0.01
		Cd	1	0.01
Inorganic Ventures	WGC-CRI-2	B	20	0.2
		Mo	20	0.2
		Si	20	0.2
		Sn	20	0.2
		Ti	20	0.2
		Sb	12	0.12

APPENDIX D

Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICSA Conc. (mg/L)
Inorganic Ventures	CLPP-ICS-A	Al	5000	500
		Ca	5000	500
		Mg	5000	500
		Fe	2000	200

				ICSB Conc. (mg/L)
Inorganic Ventures	CLPP-ICS-A	Al	5000	500
		Ca	5000	500
		Mg	5000	500
		Fe	2000	200
Inorganic Ventures	CLPP-ICS-B	Ag	100	1
		Cd	100	1
		Ni	100	1
		Pb	100	1
		Zn	100	1
		Ba	50	0.5
		Be	50	0.5
		Co	50	0.5
		Cr	50	0.5
		Cu	50	0.5
		Mn	50	0.5
		V	50	0.5
Inorganic Ventures	Arsenic	As	1000	5
Inorganic Ventures	Selenium	Se	1000	5

APPENDIX E
Known Digested QC Values
(mg/L)

Element	LCS/Spike	TCLP Spike
Al	2	-
Sb	0.5	-
As	2	5
Ba	2	100
Be	0.05	-
B	1	-
Cd	0.05	1
Ca	10	-
Cr	0.2	5
Co	0.5	-
Cu	0.25	-
Fe	1	-
Pb	0.5	5
Li	0.5	-
Mg	10	-
Mn	0.5	-
Mo	1	-
Ni	0.5	-
K	10	-
Se	2	1
Si	5	-
Ag	0.05	1
Na	10	-
Sr	1	-
Tl	2	-
Sn	1	-
Ti	1	-
V	0.5	-
Zn	0.5	-
P	10	-

Control Limits
LCS: 80 - 120%
Spike: 75 - 125%
TCLP Spike: >50%

Non-responsive

Table 1-1

Volatile Organic Compound Analytical Results
Private Well Samples
Union, Illinois
June 1990
(Concentrations in μg/L)

Parameters	Sample Locations			
	Non-responsive			
Methylene chloride	12	8	18	7
1,1,1-Trichloroethane	5	ND	ND	4 J

J - Estimated value below detection limit.
ND - Analyte not detected above method detection limit.
* - Exact address not identified.

Table 1-2

Volatile Organic Compounds Analytical Results
Private Well Samples
Union, Illinois
December 1990
(Concentrations in $\mu g/L$)

Compound	Non-responsive								
Chloroform	ND	ND	ND	1	ND	ND	ND	ND	ND
1,1,1-Trichloroethane	9	ND	ND	4	ND	ND	ND	ND	ND
Carbon tetrachloride	ND	ND	ND	2	ND	ND	ND	ND	ND
Tetrachloroethene	ND	ND	ND	4	ND	ND	2	ND	ND
1,1-Dichloroethane	ND	ND	ND	ND	ND	ND	2	ND	ND

ND - Analyte not detected above method detection limit.

Table 1-3

Volatile Organic Compound Analytical Results
Private Well Samples
Union, Illinois
January 1991
(Concentrations in $\mu g/L$)

Parameter	Intersection of Olson Rd. and Jefferson Rd.	6106 Olson Rd.	7218 Northrop Rd.	So Good Barbeque Jefferson St.
1,1,1-Trichloroethane	ND	ND	ND	ND

ND - Analyte not detected above method detection limit.

Table 1-4

Volatile Organic Compound Analytical Results
Private Well Samples
Union, Illinois
September 1991
(Concentrations in μg/L)

Parameter	Sample Location Non-responsive
Methylene chloride	16 B
Acetone	4 JB
1,1,1-Trichloroethane	31
Trichloroethylene	8

B - Analyte detected in blank.
J - Estimated value below detection limit.

Table 1-5

Previous Private Well Sampling Summary
Techalloy Company, Inc.
Union, Illinois

Sampling Date	VOCs Analyzed	Analytical Method	Detection Limits	Sample Preserved with HCL
June 1990	HSL	SW-846 8240	5-10 ug/L	No
June 1990	HSL	SW-846 8240	5-10 ug/L	Yes
December 1990	DWC	EPA 524	5-10 ug/L	Yes
September 1991	HSL	SW-846 8240	5-10 ug/L	Yes

VOC - Volatile Organic Compound
HSL - Hazardous Substance List
DWC - Drinking Water Compounds

SECTION 2

PRIVATE WELL SAMPLING PROGRAM

2.1 SAMPLE LOCATIONS AND RATIONALE

Techalloy identified the private wells previously sampled by Techalloy in a notice published in the local paper and in an announcement during a city meeting in 1990. It was announced that if Union residents had a water well and wanted the well to be sampled, they should contact Techalloy. A total of 17 residents responded to the notification, and all 17 wells were sampled. A different number and combination of the 17 wells were sampled on four different occasions between June 1990 and September 1991.

Six of the 17 well samples had detections of VOCs. In 1993, a well survey was conducted using the Illinois State Water Survey Private Well Database, the Illinois State Geological Survey Database and the McHenry County Department of Health Well Construction Reports to determine locations of any additional private water wells downgradient of the Techalloy facility. According to these databases, no additional residential wells are located downgradient of the Techalloy facility in proximity to the known groundwater plume. State or county well records contained only one of the private wells included in the June 1990 to September 1991 sampling events. Only the well at 7218 Northrup Road, located southwest of Techalloy, was included in the well records surveyed.

In addition to the well record search, Techalloy published a notice in the 14 May 1993 issue of the local newspaper soliciting responses from private well owners. One response was received from a previously unidentified private well owner located downgradient (northwest) of Techalloy and in proximity of the groundwater plume. This well is located at Non-responsive [redacted] and has been included with the private wells to be sampled. If, in the future, it is discovered that any additional private wells are identified within the proximity of the groundwater plume, the wells will be evaluated and potentially included in the groundwater sampling program.

Based on the results of the previous sampling and the newly acquired information, all the previously identified private wells along Main, Park, Wayne, and Johnson Streets; So Good Bar-B-Que on east Johnson; and the newly identified well at Non-responsive will be sampled and analyzed. These 13 wells include the six wells where VOCs were previously detected. The list of private wells to be sampled is presented in Table 2-1. The well locations are presented in Figure 2-1. A summary of the private well sampling effort is presented in Table 2-2. A summary of private well sampling data quality objectives is presented in Table 2-3.

It is proposed that, based on the results of the upcoming sampling and analyses, only selected wells be sampled and analyzed on a semiannual basis. The wells closest to the groundwater plume with analytical results indicating detections of constituents of concern at or above the MCL should be sampled on a semiannual basis until the well is secured or until U.S. EPA approves the termination of sampling and analyses.

2.2 SAMPLE COLLECTION PROCEDURES

Prior to sample collection, the wells must be purged according to the following steps:

- The tap/spigot will be turned on to induce the well pump to begin operating.
- The well pump will be allowed to operate for at least 20 minutes.
- Three pH, specific conductance, and temperature readings will be obtained during purging using portable field instruments. Measurements will be recorded until all three parameters have stabilized ($\pm$ 0.25 pH units, $\pm$ 50 μmhos/cm, and $\pm$ 0.5° C) for three consecutive readings.

Residential well holding tanks will be evacuated to ensure that water pumped directly from the aquifer is sampled. Water samples will be obtained from outlets as close as possible to the pump, and will not be collected from leaky or faulty spigots. Aerators, strainers, and hose attachments on the tap must be removed prior to sampling. In addition, all samples will be collected prior to passing through any filters, water softening devices, or pressure tanks if at all possible. To avoid dislodging particles in the faucet, the stream flow will not

Table 2-1

Private Wells To Be Sampled and Analyzed
Techalloy, Union, Illinois

1. Non-responsive [redacted]
2.
3.
4.
5.
6.
7.
8.
9.

10. Non-responsive [redacted]
11.
12.
13. So Good Bar-B-Que
East Jefferson Street
Union, Illinois 60180
Gen. Mgr. Dick Moritz
(815) 923-2144

Non-responsive

Table 2-2

Summary of Private Well Sampling Effort
Techalloy Company, Inc.
Union, Illinois

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Field Blank			Matrix Spike/Matrix Spike Duplicate[1]			Matrix Total[2]
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Private well groundwater	pH, temperature, specific conductance	VOCs	13	1	13	1	1	1	1	1	1	1	1	1	15
		Metals-filtered	13	1	13	1	1	1	1	1	1	—	—	—	15
		Metals-unfiltered	13	1	13	1	1	1	1	1	1	—	—	—	15
		Nitrate	13	1	13	1	1	1	1	1	1	—	—	—	15
		Sulfate	13	1	13	1	1	1	1	1	1	—	—	—	15
		Cyanide	13	1	13	1	1	1	1	1	1	—	—	—	15
		TSS	13	1	13	1	1	1	1	1	1	—	—	—	15

Notes:

[1] Matrix spike/matrix spike duplicate (MS/MSD) samples are not additional samples, but are instead investigative samples on which MS/MSD analyses are performed. MS/MSD analyses are for organic samples only and require double the normal volume for aqueous VOC analysis. Duplicate/spike analyses will be performed for inorganic samples.

[2] The matrix total does not include trip blank samples, MS/MSDs and duplicate/spike samples. One trip blank sample will be shipped with every shipment container of aqueous VOA samples.

Table 2-3

Data Quality Objectives
Private Well Sampling
Techalloy Company, Inc.
Union, Illinois

Data Quality Objective	Task	Data Collection Activity	Data Type	Data Use	Analytical Level
Potential Receptors	Groundwater Sampling at Private Wells	Field parameter evaluation	pH, conductivity, temperature	SP	I
		Collect and analyze groundwater samples	VOCs	SC, EA	V
			Metals	SC, EA	IV
			Nitrate	SC, EA	V
			Sulfate	SC, EA	V
			Cyanide	SC, EA	IV
			TSS	SC, EA	V

Notes and Abbreviations:

Data Use Symbols:

SC - Site Characterization
EA - Engineering Evaluation
SP - Sampling Protocol

Analytical Levels:

I - Qualitative Screening with Field Instruments
II - Field Analysis with Field Instruments
III - Off-Site Analysis by Analytical Laboratory
IV - Analyses by Routine Methods
V - Analyses by Special Methods

be adjusted while sampling.

VOC samples will be collected directly into laboratory-prepared 40-mL sample bottles to avoid excessive agitation and loss of volatiles. Filtered and unfiltered metals samples will be collected for analysis. The unfiltered samples will be collected directly into laboratory-prepared sample bottles. The filtered metals samples will be collected in laboratory-prepared sample containers and then passed through polyethylene tubing with an in-line 0.45-micron filter via a peristaltic pump to the final laboratory prepared sample containers. The samples for cyanide, nitrate, and sulfate analyses will be collected directly into laboratory-prepared sample bottles. Table 2-4 lists the required sample containers, sample volumes, sample preservation requirements, and holding times associated with all parameters applicable to the private well sampling.

The time and date of sample collection, as well as the field test results for temperature, pH, and conductivity will be recorded. Any deviation from the sampling procedures listed above will be recorded. The name(s), mailing address, and telephone number of the resident owner(s)/operator(s) will be entered into the sampling logbook.

2.3 SAMPLE PRESERVATION AND SHIPMENT

Section 4.3.2 of the RCRA Groundwater Monitoring Technical Enforcement Guidance Document, September 1986, OSWER-9950.1 states that the owner/operator should refer to the specific preservation method in SW-846 that will be used for the constituent in the sample. The residential well water volatile organic analysis (VOA) methodology for the Techalloy RFI will be based on Testing Methods for Evaluating Solid Waste, Revision 0, November 1990, (SW-846), Method 8260. Section 6.0 of SW-846 Method 8260 refers sample preservation requirements to Subsection 4.1.2, Table 4-1. Table 4-1 recommends using sulfuric acid (H_2SO_4), hydrochloric acid (HCL), or solid sodium hydrogen sulfate ($NaHSO_4$) and cooling to 4°C to preserve VOA water samples. WESTON proposes to preserve all residential well water VOA samples with HCL to a pH<2. The use of this preservative is consistent with current U.S. EPA Region V Central Regional Laboratories (CRL) sampling

Table 2-4

Required Sample Containers, Volumes, Preservation, and Holding Times
Techalloy Company, Inc.
Union, Illinois

Material Type	Analysis	Number of Containers	Required Sample Volume	Sample Container Type	Sample Preservation	Sample Holding Time[1]
Private Well Groundwater	VOCs[1,2,3]	2	80 mL	40-mL glass vials	HCl to pH <2 and cool to 4°C	14 days for analysis
	Metals	1	1 Liter	1-liter polyethylene bottle.	Nitric acid to pH <2 and cool to 4°C	6 months (28 days for mercury)
	Cyanide	1	500 mL	500-mL plastic bottle	NaOH to pH > 12 and Cool to 4°C	14 days for extraction and analysis
	Nitrate and Sulfate	1	500 mL	500-mL plastic bottle	None	28 days for analysis

[1]All holding times are calculated from the date of sample collection.
[2]Duplicate analysis requires the sample to be collected at double the volume specified.
[3]MS/MSD for organics and spike/duplicate for metals and cyanide analyses require the sample to be collected at double the volume specified.
Note: One trip blank will accompany each aqueous VOC shipment container. Trip blanks will consist of two 40 mL vials.

protocols.

The samples will be kept in shipment containers with ice packs for preservation during the transport to the laboratory. All samples will be packed such that no movement or breakage of the samples will occur during transport. Shipment containers will be properly sealed and labeled according to the Department of Transportation (DOT) regulations. Custody seals will be placed across the shipment container and lid prior to shipment. Samples will be shipped to WESTON-Gulf Coast Laboratories in University Park, Illinois by an overnight courier.

2.4 CHAIN-OF-CUSTODY DOCUMENTATION

Section 6 of the RFI Quality Assurance Project Plan (QAPP) provides details of chain-of-custody documentation and procedures.

2.5 ANALYTICAL PROCEDURES

WESTON-Gulf Coast Laboratories, Inc. will perform all chemical analysis of the private well groundwater samples. If WESTON should assign an alternative laboratory to perform some or all of the chemical analyses, an addendum to the RFI QAPP will address the analytical requirements and specifications of the chosen laboratory.

This PWSP calls for analyzing for the standard parameters that U.S. SW-846 Method 8260 would provide. Although the Method 8260 parameters exclude certain Appendix IX parameters, the Method 8260 parameters include all parameters of concern at the Techalloy facility. All other classes of parameters (e.g., metals) are provided as a purely confirmatory measure. The Techalloy facility's extensive groundwater and soil sampling has confirmed the presence of chlorinated solvent and metal constituents. The manufacturing process at the Techalloy facility has been consistent throughout the duration of operation, and it is well known that potential constituents may be present in the soil and groundwater. Numerous RCRA closure and groundwater investigations have been completed at the facility and have

determined that the constituents of concern include chlorinated solvents and their derivatives, and metal constituents.

Among VOCs, the Contract Laboratory Program (CLP)-type analyses of SW-846 Method 8260 can detect all of the facility's likely volatile organics chemicals that may have impacted the environment. Prior investigations indicate that some of the groundwater contains TCE; 1,1,1-TCA; and PCE. The CLP-type analyses can detect those VOCs, plus any other degradation products. Tables 2-5 and 2-6 present the list of parameters for which all of the private well samples will be analyzed. In order to achieve the necessary data quality for the private well sample analyses, WESTON-Gulf Coast Laboratories, Inc. will utilize the analytical methodology of the U.S. EPA CLP for volatile organic and RCRA total metals analyses. The laboratory standard operating procedures (SOPs) are based on the following analytical protocols:

- Volatile Organics - U.S. EPA SW-846 Method 8260 using Gas Chromatography/Mass Spectrometry (GC/MS). Vinyl chloride by U.S. EPA SW-846 Method 8010.
- RCRA Total Metals - U.S. EPA CLP Document No. ILM02.1 using Inductively Coupled Argon Plasma (ICAP) and Graphite Furnace Atomic Absorption Spectrometry (GFAA).
- Cyanide - U.S. EPA CLP Document No. ILM02.1
- Nitrate - Nitrate by Lachat QuickChem AE based on EPA 600/4-79-020 Method 353.2 and Lachat Method 10-107-04-01-C.
- Sulfate - Turbidimetric Analysis of Sulfate based on EPA 600/04-79-020 Method 375.4; Standard Methods, 17th Ed. Method 4500-SO_4^{-2}.
- Total Suspended Solids - EPA 600/4-79-020 Method 160.2 and Standard Methods, 17th Edition, Method 2540D.

Appendix F-1 presents the SOPs for the non-CLP private well analytical methods.

The procedures for field measurements of pH, conductivity and temperature are described in the SOPs in Appendix B of the RFI QAPP.

Table 2-5

Volatile Organic Compounds
Laboratory Quantitation Limits
Method 8260
Techalloy Company, Inc.

Volatile Organic Compound	SW-846 Method	Detection Limits µg/L
Dichlorodifluoromethane	8260	0.5
Chloromethane	8260	0.5
Vinyl chloride	8010	0.8
Bromomethane	8260	0.5
Chloroethane	8260	0.5
Trichlorofluoromethane	8260	0.5
1,1-Dichloroethene	8260	0.5
Methylene chloride	8260	0.5
trans-1,2-Dichloroethene	8260	0.5
1,1-Dichloroethane	8260	0.5
cis-1,2-Dichloroethene	8260	0.5
2,2-Dichloropropane	8260	0.5
Chloroform	8260	0.5
Bromochloromethane	8260	0.5
1,1,1-Trichloroethane	8260	0.5
1,2-Dichloropropene	8260	0.5
1,1-Dichloropropene	8260	0.5
Carbon tetrachloride	8260	0.5
Benzene	8260	0.5
1,2-Dichloropropane	8260	0.5
Trichloroethene	8260	0.5
Dibromomethane	8260	0.5
Bromodichloromethane	8260	0.5
Toluene	8260	0.5
1,1,2-Trichloroethane	8260	0.5

Table 2-5 (Continued)

Volatile Organic Compounds
Laboratory Quantitation Limits
Method 8260
Techalloy Company, Inc.

Volatile Organic Compound	SW-846 Method	Detection Limits µg/L
1,3-Dichloropropane	8260	0.5
Dibromochloromethane	8260	0.5
Tetrachloroethene	8260	0.5
1,2-Dibromoethane	8260	0.5
Chlorobenzene	8260	0.5
1,1,1,2-Tetrachloroethane	8260	0.5
Ethylbenzene	8260	0.5
p-Xylene	8260	0.5
m-Xylene	8260	0.5
Bromoform	8260	0.5
o-Xylene	8260	0.5
Styrene	8260	0.5
1,1,2,2-Tetrachloroethane	8260	0.5
1,2,3-Trichloropropane	8260	0.5
Isopropylbenzene	8260	0.5
Bromobenzene	8260	0.5
2-Chlorotoluene	8260	0.5
n-Propylbenzene	8260	0.5
4-Chlorotoluene	8260	0.5
1,3,5-Trimethylbenzene	8260	0.5
tert-Butylbenzene	8260	0.5
1,2,4-Trimethylbenzene	8260	0.5
sec-Butylbenzene	8260	0.5
1,3-Dichlorobenzene	8260	0.5

Table 2-5 (Continued)

Volatile Organic Compounds
Laboratory Quantitation Limits
Method 8260
Techalloy Company, Inc.

Volatile Organic Compound	SW-846 Method	Detection Limits µg/L
p-Isopropyltoluene	8260	0.5
1,4-Dichlorobenzene	8260	0.5
1,2-Dichlorobenzene	8260	0.5
n-Butylbenzene	8260	0.5
1,2-Dibromo-3-chloropropane	8260	0.5
1,2,4-Trichlorobenzene	8260	0.5
Napthalene	8260	0.5
Hexachlorobutadiene	8260	0.5
1,2,3-Trichlorobenzene	8260	0.5

Table 2-6

Inorganic Methods and Reporting Limits Techalloy Company, Inc. Union, Illinois

Parameter	Method	CRDLs[1]	IDLs[2,3]
		Water (μg/L)	Water (μg/L)
Arsenic	ILM02.1	10	1.6
Barium	ILM02.1	200	6.5
Cadmium	ILM02.1	5	3.4
Chromium	ILM02.1	10	5.3
Copper	ILM02.1	25	5.0
Lead	ILM02.1	3	1.1
Mercury	ILM02.1	0.2	0.2
Nickel	ILM02.1	40	11.0
Cyanide	ILM02.1	10	---
		Reporting Limits (μg/L)	
Nitrate	EPA 353.2	100	
Sulfate	EPA 375.4	5,000	
TSS	EPA 160.2	5	

[1] Contract Required Detection Limit (CRDL) reporting limits are taken from U.S. EPA CLP Document Number ILM02.1.

[2] Metals results are reported to the CRDL, however, results are reported down to the Instrument Detection Limit (IDL) if the values are between the CRDL and the IDLs. Such values are flagged with a "B".

[3] IDLs are performed quarterly and are subject to change.

2.6 QUALITY ASSURANCE/QUALITY CONTROL

Section 4 of the RFI QAPP provides information regarding quality assurance/quality control procedures.

2.7 REPORTING

All actions pertaining to the PWSP will be summarized in monthly progress reports. Techalloy will submit copies of analytical results within 15 days of receipt. A discussion summarizing the field procedures, any modifications made to the approved sampling plan, and an evaluation of the laboratory data will be incorporated into the final RFI Report. A detailed discussion of data reduction, validation and reporting is presented in Section 10 of the RFI QAPP.

SECTION 3
SCHEDULE

3.1 SCHEDULE

The schedule and dictates stated in Sections VI.2. and VI.3. of the Administrative Order of Consent shall be followed.

APPENDIX F-1

STANDARD OPERATING PROCEDURES FOR NON-CLP ANALYTICAL METHODS

RELEASED
2019-007454
July 27, 2020 - TJW

ORGANIC ANALYSIS PROTOCOL
Gas Chromatography/Mass Spectrometry (GC/MS) Analysis of Volatile Organic Compounds by Method 8260

CONTROLLED DISTRIBUTION
COPY # : Uncontrolled
ISSUED TO : Tech Alloy QAP (VHS)
Full Signature Approvals Are Kept on File with WESTON®'s Analytics Division QA Standard Practice Records

REVISION NUMBER: 01

1.0 PURPOSE

To outline the guidelines for the analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS).

2.0 REFERENCES

This SOP was written using SW-846 Method 8260 as a reference.

3.0 METHOD SUMMARY

This method is used to determine volatile organic compounds in a variety of matrices. It is applicable to water, soil, sediment, sludge and waste drum samples.

This method can be used to quantify most volatile organic compounds that have a boiling point less than 200°F. It is also limited to those compounds that elute as sharp peaks from a capillary column. A listing of applicable compounds and their characteristic ions appears in Table 1.

A portion of sample, measured into a sample vessel, is purged with an inert gas. The volatile compounds are transferred to a trap, containing retarding materials.

The trap is then backflushed with the inert gas and rapidly heated to effectively transfer the compounds to the GC column. The GC oven is then temperature ramped to separate the compounds and introduce them to the source.

The mass filter separates the ions which are then detected by the analyzer. The data system then provides qualitative and quantitative information concerning the sample.

Those samples suspected of high background levels are screened by GC/FID prior to analysis. Initial dilutions are determined from the screen.

Instrument calibration occurs about every 12 hours, or prior to analysis. Instrument maintenance is performed as needed, on a daily, monthly or yearly basis.

4.0 INTERFERENCES

External interferences can be caused by contaminants from sample containers, preparative glassware and reagents, syringes and columns and manifest themselves as high background and/or discrete peaks. Some contaminants are also introduced through the sample vial seal and/or instrument sample connections. Proper glassware preparation, sample handling and instrument maintenance should eliminate these sources. A laboratory method blank is analyzed prior to any analysis to show absence of any contaminants. Reagent water sampled in the lab and carried through all field operations is also analyzed to show absence of contaminants from field sampling.

Carryover is also another source of contamination. Any time a high level sample is analyzed, the next sample in the batch is checked for carryover. If carryover is suspected, that sample is re-analyzed. The position is rinsed with methanol/water. If carryover is excessive and continues into the next samples, the batch is aborted/paused, the column and trap baked, and/or blanks analyzed until all contamination is absent. If further response is required (i.e. trap replacement) it is documented in the maintenance logbook. See maintenance section.

Internal interferences can be purged from the sample with the target compounds and appear as elevated baselines or distinct peaks. Internal interferences most often manifest themselves as low/high recoveries of surrogate/matrix spike compounds. Matrix interferences vary from sample to sample.

The volatile lab must be free of solvents. Common air-borne lab contaminants, dichloromethane, toluene, 2-butanone and acetone are allowed up to 5X the EQL. Occasionally, higher values for acetone and dichloromethane are allowed, but this is approved before analysis.

4.1 Estimated Quantitation Limits (EQL)

Estimated quantitation limits for each routinely analyzed compound appears in Table 2. EQL's are highly matrix dependent and will vary.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. Listed below are the holding times and the references that include container and preservation requirements for compliance with the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA).

Matrix	SW-846
All	14 days (VTS)

VTS = verified time of sampling

All samples received for volatile analysis are refrigerated upon receipt at 4 ± 2°C. See specific methods for preservation recommendations or requirements.

6.0 INSTRUMENTATION AND EQUIPMENT

6.1 GC/MS

A temperature programmable chromatograph interfaced with a mass-selective detector capable of scanning from 35 - 260 amu every second or less using 70 volts of electron energy in the electron ionization mode. The system is capable of producing an acceptable spectrum of bromofluorobenzene when 50 ng is injected on-column.

6.2 Data System

The analytical system is interfaced with a data system capable of continuous acquisition and storage of mass spectral data. The RTE 6/1000 Rev. E and UNIX Series 400 software is capable of plotting specific masses versus time or scan numbers (Extracted Ion Current Profile) and integration of that abundance. The systems also contain the most recent NBS library.

6.3 Current Hardware/Software

- 1 Hewlett-Packard 5995 GC/MS. Equipped with a capillary DB-624 column.

- 1 Hewlett-Packard 5890 GC interfaced with a 5970 MSD. Equipped with a DB-624 capillary column.

- 1 Hewlett-Packard 5890 GC interfaced with a 5971 MSD. Equipped with a stand alone UNIX Series 400 computer system and peripherals. Contains a DB-624 Capillary Column.

- 1 Tekmar LSC 2 in connection with 1 Tekmar ALS 10 station autosamplers for the 5995.

- 1 Tekmar LSC 2000 in connection with an ALS 2050 for the 5890/5970 system.

- 1 OI-Analytical 4460A sample concentrator, MPM-16 Multiple Purging Module (16 stations), MHC-16 Cryo-focusing Module for the 5890/5971 MSD.