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TECHNICAL MEMORANDUM

TO: Shanna Schmitt, Minnesota Pollution Control Agency
Amy Hadiaris, Minnesota Pollution Control Agency
Tom Barounis, U.S. Environmental Protection Agency

FROM: Shane Waterman, Wenck Associates, Inc.
Matt Bowers, Wenck Associates, Inc.

Copy: Jay Biedny, Ramsey County
Joe Otte, Wenck Associates, Inc.

DATE: June 10, 2014

SUBJECT: QAPP Addendum No. 1
UFP-Quality Assurance Project Plan
TCAAP Redevelopment #2 Site

Purpose

The purpose of this correspondence is to provide sufficient detail to add total cyanide (EPA Method 9012B) to the list of contaminants of concern (COC) to the above-referenced QAPP. In addition, a discussion has been provided to clarify and request a modification to the QA/QC evaluation of ISM data collected under the QAPP. The discussion focuses on the appropriate statistical tool to evaluate the ISM replicates collected in specific decision units, i.e. using a 95% UCL calculation instead of the relative percent difference (RPD) as described in the current QAPP.

Method 9012B (Total Cyanide)

Cyanide hot spot sampling will be required as documented in the report titled "*Additional Investigation Work Plan, Building 501, Former Twin Cities Army Ammunition Plant (TCAAP), Arden Hills, Minnesota*" (Wenck, April 2014). The MPCA approved the work plan in a letter dated May 16, 2014. Revised QAPP worksheets and all appropriate QAPP appendices have been included with this submittal in Attachments 1 through 4.

ISM Samples - Relative Percent Difference (RPD) Calculation

Wenck has performed an initial assessment of the ISM bulk samples from two TCAAP decision units and has identified a potential issue with the evaluation process documented in the QAPP. ISM sample evaluation is discussed in QAPP Worksheet #14. An updated Worksheet #14 has been included with this submittal.

Due to our initial review of the ISM data collected to date we feel that calculating RPD values is not the appropriate statistical tool to evaluate ISM data. RPDs are relevant to laboratory analysis because laboratory and field duplicates are the same physical sample and RPD measures the consistency of the laboratory extraction and analytical method. RPD can also be used to evaluate matrix effects. With ISM replicates, we are not analyzing true field duplicates, the samples are not an aliquot of the same physical sample, they are different samples collected within a Decision Unit (DU) and as such, some variability not related to laboratory or sampling methodology would be expected. During the evaluation of the first set of ISM triplicate samples RPD values were found to be generally <35% for metals (expected due to the fact that the metals values were in the background range in all three samples) but >35% for PAH samples. The high PAH RPD values are somewhat expected due to the known issue pertaining to matrix-related interference. Highly organic soils tend to attribute to the matrix interference phenomenon.

The statistical analysis of the ISM data is intended to test if the ISM sampling protocol accurately reflects average contaminant concentrations in a DU. The replicates are not a test of the laboratory's method, but more a test of the conceptual site model (CSM). Differences we see in the ISM replicates show that contaminant concentrations can, and do, vary across a DU; however, when collected over the entire DU, replicate samples provide a separate estimate of the mean concentration. For the DUs chosen for replicate sampling during this project the samples from that DU will be combined to derive a 95% UCL.

Wenck proposes to evaluate future ISM samples through calculation of the 95% UCL. Wenck will use the Student's-*t* method for calculating the 95% UCL for this project. Once calculated using the ISM parent, duplicate and triplicate samples the 95% UCL concentration of these results will then be evaluated against the approved cleanup criteria. If the 95% UCL concentration is below the cleanup criteria the result will be considered acceptable and the DU ready for residential redevelopment. If the 95% UCL concentration exceeds the cleanup criteria, the decision will be to remediate the DU through excavation and offsite removal.

LIST OF ATTACHMENTS:

Attachment 1	Pace SOP – Total Cyanide using Micro-Distillation and SmartChem (SOP Number S-GB-I-064 rev.02)
Attachment 2	Updated QAPP Worksheets (Rev. 2)
Attachment 3	Updated QAPP Appendix F - Analytical Data Validation Form (Rev. 2)
Attachment 4	Updated QAPP Appendix G - Data Qualification Procedures and Data Qualifier Definitions (Rev. 2)

Attachment 1

Pace SOP – Total Cyanide using Micro-Distillation and SmartChem (SOP Number S-GB-I-064 rev.02)

Pace Analytical Services, Inc.
1241 Bellevue Street Suite 9
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STANDARD OPERATING PROCEDURE

Total Cyanide using Micro-Distillation and SmartChem

Reference Method: SW846 9012B and EPA 335.4

SOP NUMBER:	S-GB-I-064-REV.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-064-Rev.01

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 01/20/14

Kate Grams, Laboratory Quality Manager — Date: 1/16/14

Chad Rusch, Department Manager — Date: 1/16/14

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

This is **COPY#** **5** Distributed on **5/27/14** by **KEG** and is ____ **CONTROLLED or** **X** **UNCONTROLLED**

Table of Contents

1. Purpose/Identification of the Method

This Standard Operating Procedure (SOP) describes the analyses of Total Cyanide using Micro-Distillation and Analyzed by SmartChem Discrete analyzer using methods SW846 9012B and EPA Method 335.4.

2. Summary of Method

2.1 By means of a passive miniature distillation device, MICRO DIST, cyanide in the sample is released by digesting and acidifying cyanide complexes, and converting them to hydrocyanic acid (HCN). The cyanide ion is trapped in a 1.0 M sodium hydroxide absorbing solution, which is diluted to 0.25M solution during distillation. By means of discrete analysis, the 0.25M Noah distillate is converted to cyanogens chloride by reaction with chloramines-T, pyridine and barbituric acid to give a red-colored complex. The absorbance of this complex is measured at 570 nm by measuring the peak area resulting from the sample. The peak area is proportional to the concentration of the cyanide in the sample.

2.2 All samples must be distilled before analysis.

3. Scope and Application

3.1 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical and distillation methods.

3.2 Parameters: This SOP is applicable to the analysis of Cyanide in drinking, ground, surface, and saline waters, domestic and industrial wastes, soils, and TCLP, SPLP, ASTM extracts.

3.3 This SOP utilizes the SmartChem Discrete analyzer.

4. Applicable Matrices

4.1 Drinking, ground, surface, and saline waters

4.2 Domestic and industrial wastes, soils.

4.3 TCLP, SPLP, ASTM extracts

5. Limits of Detection and Quantitation

5.1 For aqueous samples the current Pace Reporting Limit is 0.02 mg/L. The current MDL is listed in the LIMs and is available by request from the Quality Department.

5.2 For solid samples the current Pace Reporting Limit is 0.6 mg/Kg. The current MDL is listed in the LIMs and is available by request from the Quality Department.

6. Interferences

6.1 Most non-volatile interferences are eliminated or minimized by the distillation procedure. Some of the known interferences are aldehyde, nitrate-nitrite, and oxidizing agents, such as chlorine, thiosulfate, and sulfide.

6.2 Oxidizing agents such as chlorine decompose most cyanide. Test a drop of the sample with potassium iodine-starch paper (KI-starch paper) at time of collection; a blue color indicates need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color changes; then add an additional 0.06 g ascorbic acid per liter of sample.

6.3 False positive results may be obtained by samples that contain Nitrate/or nitrite. Sulfamic acid is added to all samples prior to distillation to eliminate this interference.

6.4 Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. If a drop of sample on lead acetate paper indicated the presence of sulfide, treat the stabilized sample (pH >12) with bismuth nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled.

7. Sample Collection, Preservation, Shipment and Storage

Table 1: Sample Collection, Preservation and Handling

Matrix	Prep Method	Container(s)	Preservation Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SW-846 9012B EPA 335.4	250 ml plastic containers	pH >12; NaOH 14 days	On ice 4+/- 2°C	≤6°C
TCLP, SPLP, ASTM	SW-846 9012B EPA 335.4	glass or plastic containers (when solid) Leachate is filtered into a 250 ml plastic container.	pH >12; NaOH 14 days to extract, additional 14 days to analyze (Total 28 days from collection)	On ice 4+/- 2°C	≤6°C
Solid	SW-846 9012B	glass or plastic containers	None 14 days	On ice 4+/- 2°C	≤6°C

8. Definitions

8.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

8.2 Reagent Base Line (RBL) – Absorbance created when the diluent and method reagents are added to the cuvette. The diluent is a matrix match of the sample matrix.

8.3 Water Base Line (WBL) – Absorbance created when reagent grade water is added to the cuvette. The WBL is used to check the quality of the cuvette, condition of the filters, and lamp. If the value is too high or too low the cuvette will be rejected and will not be used in the analysis.

9. Equipment and Supplies

Table 2: EQUIPMENT

Equipment	Manufacturer	Model(s)/Catalog number •
SmartChem	SmartChem	SmartChem
Micro-Distillation	Lachat	Micro Distillation
Top loading Analytical Balance	Mettler	BA310P
1000 µL Pipette	Eppendorf	05-402-76 (Fisher Cat#)
500 µL Pipette	Eppendorf	05-402-72 (Fisher Cat#)
250 µL Pipette	Eppendorf	05-402-69 (Fisher Cat#)
Repipetors (0.5 - 5.0mL)	Barnstead/Thermolyne	13-687-33 (Fisher Cat#)
Vented Hood	Hamilton	
Spoonula (Lab Spoon)	FisherBrand or equivalent	14-375-10 (Fisher Cat#)
Gloves – Heat Resistant		
Cyanide Manifold	Lachat	10204-00-1-A
Interference filter – 570 nm	Lachat	

- Or equivalent

Table 3: Supplies

Supplies	Manufacturer	Catalog number •
Distillation tubes	Lachat	A17001
Sample Cups 4mL	Westco	927-0018-01
Volumetric Flasks	Fisher	Various
Parafilm	Fisher	
Teflon Chip	Fisher	
• Or equivalent		

10. Reagents and Standards

Table 4: REAGENTS

Reagent	Alias	Concentration	Directions found in:
Potassium Cyanide	Stock Standard	Neat	
Potassium Cyanide	Second Source Stock Standard	Neat	
Potassium Hydroxide		Neat	
Sodium Hydroxide		Neat	
Noah – 0.25 N	Carrier Solution	0.25 N	Table 6
Deionized Water			
Potassium Phosphate, Monobasic, (KH_2PO_4)		Neat	
Sodium dihydrogenphosphate Buffer	Buffer Solution	1 M	Table 6
Chloramine-T hydrate		Neat	
Chloramine-T Reagent	Color Reagent		Table 6
Barbituric Acid		Neat	
Pyridine		Neat	
Hydrochloric Acid		12 M	
Pyridine-Barbituric Acid Reagent	Pyridine-Barbituric Acid Reagent		Table 6
p-dimethylaminobenzalrhodanine	Indicating solution		Table 6
Standard Silver Nitrate Solution		0.0192 N	
Magnesium Chloride Hexahydrate		Neat	
Sulfuric Acid		Concentrated	
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Table 6
Nitrate Bismuth		Neat	
Lead Acetate Paper		Neat	
Sulfamic Acid		Neat	
Teflon chips		Neat	

Table 5: STANDARDS

Standard	Concentration	Direction found in:	Alias
Potassium Cyanide	1000 mg/L	Table 7	Stock Standard
Potassium Cyanide Must be different Lot # than Stock	1000 mg/L	Table 7	Second Source Stock Standard
Level 1 Calibration Standard	0.40 mg/L	Table 7	Cal Std. 1
Level 2 Calibration Standard	0.20 mg/L	Table 7	Cal Std. 2
Level 3 Calibration Standard	0.10 mg/L	Table 7	Cal Std. 3
Level 4 Calibration Standard	0.05 mg/L	Table 7	Cal Std. 4
Level 5 Calibration Standard and RLVS Standard	0.02 mg/L	Table 7	Cal Std. 5
Level 6 Calibration Standard	0.00 mg/L	Table 7	Cal Std. 6
Initial Calibration Verification – 1	0.10 mg/L	Table 7	ICV – 1
Initial Calibration Verification – 2	0.05 mg/L	Table 7	ICV – 2
Continuing Calibration Verification	0.20 mg/L	Table 7	CCV
Working Standard (1)	10 mg/L	Table 7	Working Standard (1)
Secondary Working Standard (1)	100 mg/L	Table 7	Secondary Working Standard (1)
Secondary Working Standard (2)	1.2 mg/L	Table 7	Secondary Working Standard (2)
Matrix Spike/Matrix Spike Duplicate – Liquid	0.10 mg/L	Table 7	MS/MSD-Liquid
Matrix Spike/Matrix Spike Duplicate – Solid	0.10 mg/L	Table 7	MS/MSD-Solid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Liquid	0.10 mg/L	Table 7	LCS/LCSD-Liquid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Solid	0.10 mg/L	Table 7	LCS/LCSD-Solid

Table 6: PREPARATION OF REAGENTS

Standard	Alias	Final Concentration	Directions	Final Volume
NaOH – 0.25 N	Carrier Solution	0.25 N	Weigh 10 g of neat NaOH into ~ 500 ml DI water in 1 L plastic container. Dilute to volume. Prepare fresh daily.	1000 ml
Sodium dihydrogenphosphate Buffer	Buffer Solution	1.0 M	Weigh 69 g of neat NaH_2PO_4 into ~ 500 ml DI water in 1 L volumetric flask. Add 2.0 mL of concentrated Probe Rinse Solution. Dilute to volume. Shelf life = 3 months.	500 ml
Chloramine-T Reagent	Color Reagent		Weigh 0.4 g of neat Chloramine-T hydrate into ~ 50 ml DI water in 100 ml volumetric flask. Dilute to volume. **PREPARE DAILY!**	100 ml
Pyridine-Barbituric Acid Reagent	Pyridine-Barbituric Acid Reagent		**MAKE UNDER HOOD!!!!!!!** Weigh 7.5 g barbituric acid into a 500 mL beaker Add 100 ml DI. Add 37.5 ml pyridine, mix to dissolve Add 7.5 ml conc. HCL. Transfer to 500 mL volumetric flask and dilute to volume. Shelf life = 3 months.	500 ml
p-dimethylaminobenzalrhodanine	Indicator Solution		Weigh 20 mg of neat p-dimethylaminobenzalrhodanine into a 100 ml volumetric flask. Dilute into 100 ml acetone. Shelf life = daily/when used.	100 ml
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Weigh 110.8 g DI water in beaker. Add 32.2 g Magnesium Chloride Hexahydrate ($MgCl_2.6H_2O$). Slowly add 139 g concentrated H_2SO_4. Transfer to automatic pipette container. Cool. Shelf life = 1 year.	200 g

Table 7: PREPARATION OF STANDARDS

Standard	Alias	Final Concentration	Directions	Final Volume
Potassium Cyanide	Stock Standard.	1000 mg/L	POISON!!!!! Weigh 2.51 g of neat KCN and 2 g of neat KOH into ~900 ml DI H2O in 1 L volumetric flask. Dilute to volume. Shelf life = 3 months. Standardize against AgNO3. If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Potassium Cyanide	Second Source Stock Standard	1000 mg/L	POISON!!!!! Weigh 2.51 g of neat second source KCN and 2 g of neat KOH into ~900 ml DI H2O in 1 L volumetric flask. Dilute to volume. Shelf life = 3 months. Standardize against AgNO3. If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Working Standard (1)	Working Standard (1)	100 mg/L	Pipette 10 ml stock standard into 100 ml volumetric flask. Dilute to volume with DI H2O. PREPARE DAILY!	100 ml
Working Standard (2)	Working Standard (2)	1.2 mg/L	Pipette 1.2 ml Working Standard (1) into 100 ml volumetric flask. Dilute to volume with DI H2O. PREPARE DAILY!	100 ml
Secondary Working Standard (1)	Secondary Working Standard (1)	10 mg/L	Pipette 1 mL Potassium Cyanide (second source stock standard) into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. PREPARE DAILY!	100 ml
Calibration standard – All other calibration points are diluted off of this standard by the instrument.	Cal. Std.	0.40 mg/L	Pipette 2.0 mls of Working Standard (2) into 6 ml distillation tube. Dilute to volume with DI H2O. Prepare daily and distill.	6 ml
CRDL Standard	CRDL	0.020 mg/L	Pipette 0.25 mls of calibration standard into 5 ml volumetric flask. Dilute to volume with 0.25N NaoH. Prepare daily.	5 ml
Calibration Blank	Cal. 0	0.000 mg/L	Use 0.25N NaOH only.	100 ml
Continuing Calibration Verification	CCV – 1	0.20 mg/L	Pipette 1.0 ml of Working Standard (2) solution into 6 ml distillation tube. Dilute to volume with DI H2O. Prepare daily and distill.	6 ml
Initial Calibration Verification –1	ICV – A	0.10 mg/L	Pipette 1.0 ml of Secondary Working Standard (2) solution into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	100 ml
Initial Calibration Verification –2	ICV – B	0.050 mg/L	Pipette 0.50 mls of Secondary Working Standard (1) solution into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	100 ml
Matrix Spike/Matrix Spike Duplicate – Liquid	MS/MSD - Liquid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into distillation tubes. Dilute to volume with sample. Prepare daily.	6 ml
Matrix Spike/Matrix Spike Duplicate – Solid	MS/MSD-Solid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into sample tubes. Add solid sample. Dilute to volume with DI H2O. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Liquid	LCS/LCSD-Liquid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into sample tubes. Dilute to volume with DI H2O. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Solid	LCS/LCSD-Solid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into sample tubes. Dilute to volume with DI H2O. Prepare daily.	6 ml

11. Calibration and Standardization

11.1 **Daily Calibration** – The SmartChem must be calibrated each time it is set up and at least every 24 hours that samples are analyzed. Calibration requires analysis of a minimum of 5 standards plus a blank due to the linear regression calibration used. The working range is from the current MDL to the highest standard in the calibration curve.

11.2 **Calibration Verification** – Each calibration must be verified by analyzing an Initial Calibration Verification (ICVA/ICVB) and Initial Calibration Blank (ICB). A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) must also be analyzed after each set of 10 samples.

11.3 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit to demonstrate acceptable recovery at the reporting limit.

11.4 **Acceptance Criteria** – The correlation coefficient of the response for elements requiring multiple levels must be ≥ 0.995. The results of the ICV/CCV checks must agree within ± 10%. The results of the ICB/CCBs must be <RL.

11.5 Follow steps in section 12 to run calibration curve.

11.6 The SmartChem can automatically prepare diluted standards and samples, as required, to bring high concentration standards and samples into the working range of the method. See the SmartChem Operation Manual Chapter 3 for instructions on how to configure the SmartChem Parameter Method File.

12. Procedure

NOTE: Document all sample volumes, standards and reagents used in the distillation on the prep log.

12.1 Distillation Procedure - This section of the SOP details the steps in distilling the samples before analysis. Samples must be checked for sulfides before distilling. If sulfides are present and the samples treated with bismuth nitrate, the entire calibration curve must be similarly treated and distilled. Refer to section 6.4 for procedure. In the following procedure, **D** and **M** refer to the marks on the collector tube. **D** means "distillation" end, and **M** means "measuring" end.

12.1.1 Set the controller to 120°C, allow the heater to warm up. The operating temperature of the distillation block needs to be verified annually.

12.1.2 Label tubes with the **M** end up, place as many properly labeled collector tubes as you have samples into the collector tube rack.

12.1.3 For liquid samples, place a drop of sample on lead acetate paper. If the paper darkens this indicates the presence of sulfide. Treat the stabilized sample (pH >12) with bismuth Nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled. Place 6 ml of sample into each properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.

NOTE: If samples contain and are treated for sulfide, the entire calibration curve must also be similarly treated and distilled along with the samples.

12.1.4 For soil samples, weigh out ~0.20 g of soil or sludge to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water with a calibrated automatic pipette into each properly labeled sample tube. Add ~ 0.1 g sulfamic acid.

12.1.5 For liquid samples, prepare one method blank by adding 6 mls of DI water to properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.

12.1.6 For soil samples, prepare one method blank by weighing out 0.20 g of Teflon chips to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water with a calibrated automatic pipette into a properly labeled sample tube. Add ~ 0.1 g sulfamic acid.

12.1.7 For liquid samples, prepare one LCS (and one LCSD if needed/requested) by pipetting 6.0 ml of deionized water, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.

12.1.8 For soil samples, prepare one LCS (and one LCSD if needed/requested) by weighing out 0.20 g of Teflon chips to the nearest 100th on a calibrated balance. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 ml of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.

12.1.9 For liquid samples, select one parent sample and prepare one MS/MSD pair per 10 samples by adding 6.0 ml of sample, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.

12.1.10 For soil samples, select one parent sample and prepare one MS/MSD pair by weighing out 0.20 g - 0.50 g of sample to the nearest 100th on a calibrated balance. The sample weight is dependent upon the sample matrix. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 ml of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.

12.1.11 Using a calibrated pipette, add 0.75 ml of 7.11 M sulfuric acid / 0.79 M magnesium chloride solution to the sample tube using the supplied automatic pipette.

12.1.12 Immediately push the **D** end of a Cyanide-1 collector tube over the open end of each sample tube to start the seal.

12.1.13 Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing, the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.

12.1.14 The pressing motion should be a smooth constant pressure, which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.

12.1.15 Put on heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.

12.1.16 Set the timer for 30 minutes.

12.1.17 When 30 minutes is up, remove the first tube, use heat-resistant gloves, from the block and immediately pull its sample tube using a downward twisting motion as opposed to a sideways motion. You must remove the sample tube within 4 seconds of removing it for the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution into the 5 gallon waste container.

12.1.18 Invert each collector tube and place it into the collector tube rack, now with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.

12.1.19 Hold collector tube horizontally and rinse its walls with the distillate in order to homogenize it. Roll tube in order to collect all droplets from the walls of the tube. Return the tube to an upright position so the **D** end is up.

12.1.20 With the **D** end up, break the collector tube in half by pulling the **D** end hard toward yourself to break it, the twisting and tearing off the **D** end. Discard the **D** end.

12.1.21 In the **M** end of the tube dilute to the 6.0 ml mark with DI water. This results in the original sample volume, but now in 0.25 M NaOH. Pour in culture tube.

12.1.22 In the event that a distillation needs filtering prior to analysis, the batch QC must also go through the same filtration process. Document in the prep-log.

12.2 Basic System Operation (Analytical) - This portion of the SOP is designed to allow the user to set up and run a method. For a more detailed explanation of the many other options, the user should refer to the SmartChem Software Reference Manual. It is assumed that a method program has already been created for the chemistries you wish to run.

12.3 If not already on, turn on computer, printer, monitor, and the SmartChem instrument. The SmartChem on/off toggle switch is located on the left side of the instrument.

12.4 Log into the PC and Network. Click twice on SmartChem New and enter the username: *SmartChem New WESTCO* and password: *JOE*.

12.5 Build Sequence in LIMSLink of batched samples:

12.5.1 Go into LIMSLink and click on the "*Running Man*".

12.5.2 Select the *Prep* method and click *Start New* followed by *OK*.

12.5.3 In the first two lines enter ICVA and ICVB followed ICB in the third line and CRDL in the fourth.

12.5.4 Click on Get Samples and enter the Batch, the Instrument ID (40WTA9) and click OK.

12.5.5 Repeat the last step for each batch of samples in the sequence.

12.6 SmartChem Sequence Loading and Analysis:

12.6.1 SmartChem Start-up procedure.

12.6.2 Go to Sample Entry

12.6.3 Double click on the desired Method to be run. For cyanide the method is CN-CN 335.4 Cyanide 20 to 400ug/L.

12.6.4 Click on Import from file button

12.6.5 Load Sequence file (create Run Plan) - open the SmartChem folder, scroll all the way to the right, and double click on *SmartChem*.

12.6.6 Save – Click on the red disk to save with the format *CN073112 40WTA9 WETA/EPA335.4 CCR*. 073112 is the month, day, year of the analysis. 40WTA9 is the instrument. WETA is the QUEUE and CCR is an example of analyst initials. Spaces must be used where they are. Click OK.

12.6.7 Save External File by clicking *YES* and Save the run plan as *CN073112*.

12.6.8 Go to System Monitor.

12.6.9 Select and Click on the Run Plan that was created.

12.6.10 Verify that the selected Run Plan is correct.

12.6.11 Load Samples, Standards, Controls, Diluent, and Empty Cups as displayed in the System Monitor.

12.6.12 Check the probe rinse, DI water, and Cleaning Solution bottles.

NOTE: When filling these solutions, pour slowly to minimize foaming. If filling is required while a run is in progress, do not lift siphon tube above the liquid surface. Insert a long stem funnel through the open port in the reservoir cap and fill the container slowly with the respective solution to minimize foaming.

12.6.13 Click Start in System Monitor to display user selectable options before beginning the analysis.

12.6.14 If a Wash Cuvette and/or WBL is required before the start of the analysis, then click on the appropriate button. It is recommended that a Wash Cuvette and WBL be run at the start of each day.

NOTE: It is recommended that a new WBL be run after a Wash Cuvette operation is performed and/or after the removal and/or replacement of the same or a new cuvette into the reaction tray.

12.6.15 Click the Start Button to begin analysis. A box will open. Make sure the Calibration and RBL options are selected for a run that is using a new calibration and click OK.

NOTE: Any daily or periodic maintenance must be recorded in the instrument daily logbook.

12.7 SHUTDOWN:

12.7.1 Click on EXIT icon

12.7.2 Turn off computer, printer and switch on surge suppresser.

13. Quality Control

13.1 Refer to the most current version of SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 Initial Calibration Verification (ICV)

13.2.1 The ICVA/ICVB must be analyzed before samples.

13.2.2 The ICVA/ICVB are not distilled.

13.2.3 Concentration must be within ± 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

13.2.4 If the ICVA/ICVB is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.2.5 The lot number of the Potassium Cyanide Standard used to make the ICVA/ICVB must be different from that of the calibration curve standards.

13.3 Continuing Calibration Verification (CCV)

13.3.1 The CCV is analyzed after every 10 samples.

13.3.2 The CCV is distilled with the batch.

13.3.3 Concentration must be within ± 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails, recalibrate and reanalyze all samples back to the last acceptable CCV.

13.3.4 If the CCV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.3.5 The lot number of the Potassium Cyanide Standard used to make the CCV must be different from that of the ICV.

13.4 Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

13.4.1 The ICB must be analyzed after the ICVA/ICVB and before samples. The CCB must be analyzed after the CCV and before samples.

13.4.2 The ICB and CCB are not distilled with the batch.

13.4.3 The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, correct the problem, and the calibration re-verified. If the reset of the I/CCB fails, recalibrate and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.4.4 If the sample concentration is greater than ten times the concentration in the ICB or CCB, the samples do not need to be reanalyzed.

13.5 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.6 **Method Blank (MB)**

13.6.1 A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples. The MB must be distilled with batch.

13.6.2 The absolute value must be < PRL.

13.6.2.1 When measurements are above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.6.2.2 If the analyte is detected in the method blank, method blank criteria is evaluated and flagged with an appropriate qualifier to the MDL when MDL reporting is required: qualify with an appropriate qualifier sample results less than 10 times the absolute value detected in the blank. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample-by-sample basis. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

13.7 **Laboratory Control Sample (LCS)**

13.7.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed if there is insufficient sample volume to perform a matrix spike/matrix spike duplicate or if the client requests one. The LCS must be distilled with batch.

13.7.2 Concentration must be within $\pm$ 10% of the true value for waters. For soils, the concentration must be within calculated in –house limits or default limits of $\pm$ 20%.

13.7.2.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.2.2 If no errors are found and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still out side the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.7.2.3 If sufficient sample volume is not available, report the sample data with an appropriate (L) qualifier flag on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.7.3 When an LCSD is performed, the precision between the LCS and LCSD must be =/< 20% RPD.

13.7.3.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.3.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.8 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

13.8.1 One pair per up to 10 environmental samples or 10% frequency whichever is more frequent. The MS/MSD must be distilled with batch.

13.8.2 Both QC samples must be calculated for accuracy and precision.

13.8.3 Concentration must be within $\pm$ 10% of the true value for waters. For soils, the concentration must be within calculated in house limits or default limits of $\pm$ 20%.

13.8.3.1 If the four times the concentration of the spike is less than the level of the parent, accuracy need not be calculated.

13.8.3.2 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.3.3 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.8.4 The precision between the MS and MSD must be =/< 20% RPD.

13.8.4.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.8.5 The parent sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD.

13.8.6 The lot number of the Potassium Cyanide used to make the CCV is different for the MS/MSD.

13.9 The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date. The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, investigate and correct the problem, verify the calibration, and reanalyze all analytical samples analyzed.

13.10 When preparation of a sample exceeds 14 days past the time of collection, notify the project manager before proceeding. If a sample is run past 14 days after collection, flag the result with an appropriate data qualifier.

13.11 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with an appropriate data qualifier.

13.12 See attachments Table 8 and Table 9 for a summary of QC.

14 Data Analysis and Calculations

14.1 The instrument provides calculated sample results in ug/L. Calculations are only necessary if a dilution was used.

14.1.1 Liquid Calculation

Raw Data Value (ug/L) x Dilution Factor / 1000 = Cyanide (mg/L)

14.1.2 Soil Calculation

$$\frac{\text{Raw Data Value (ug/L) X 0.006 L X Dilution Factor}}{(0.20 \text{ \% solids in decimal form x } 1000)} = \text{Cyanide (mg/Kg)}$$

15 Data Assessment and Acceptance Criteria for QC Measures

15.1 See Table 9: Data Assessment

16 Corrective Action for Out-of-Control Data

16.1 See Table 9: Data Assessment

17 Contingencies for Handling Out-of-Control or Unacceptable Data.

17.1 See Table 9: Data Assessment

18 Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s (most current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ* (most current revision or replacement), for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

18.1.5 Linear Calibration Range (LCR): The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by +/- 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

18.1.5.1 Criteria: All standards must be within +/- 10% of the true value with the exception of the highest standard analyzed over the calibration range that should be outside the 10% rule

18.1.6 Pace Analytical Services, Inc – Green Bay Laboratory will not use any data over the highest calibration standard used. All samples will be diluted and reanalyzed that are over the calibration range.

19 Method Modifications

19.1 Method modifications for EPA Method 9012B and EPA 335.4 are as follows

19.1.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

19.1.2 All major modification to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

19.1.3 Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.

19.1.4 EPA Method 9012B is an aqueous method, the laboratory has modified the method to accommodate solid matrices.

19.1.5 EPA Method 9012B and EPA 335.4 are described for macro glassware, the laboratory has chosen to use micro distillation equipment in place of the macro. All sample, reagent and standard volumes have been reduced accordingly

19.2 If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the "M5" data qualifier.

20 Instrument/Equipment Maintenance

20.1 See Section 12.

21 Troubleshooting

21.1 See SmartChem Operating Manual.

22 Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23 Waste Management

23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

24 Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

24.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25 References

25.1 Pace Quality Assurance manual (most current revision or replacement).

25.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 SmartChem 200 Method 280-400E (Rev: April 2007)

25.4 *Micro Dist Method Cyanide-1,* Lachat Instruments, October 12, 2000

25.5 SW846 9012B, Revision 2.0, November 2004.

25.6 EPA 600 R-93-100, Revised August 1993, Method 335.4-1

25.7 Standard Methods for the Examination of Water and Wastewater, 18th Edition, p. 4-18, Methods 4500-CN^- A, B, C, and D (1992)

26 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

26.1 Table 8: **QC SUMMARY**

26.2 Table 9: **DATA ASSESSMENT/CORRECT ACTION**

26.3 Attachment I: Flowchart

27 Revisions

Revision Number	Reason for Change	Date
S-GB-I-064.Rev.0	First Issue.	19Jan2010
S-GB-I-064-Rev.01	Throughout Document: Updated to new format in SOP: S-GB-Q-017 Preparation of SOPs (most current revision or replacement). Updated SOP References throughout document.	25Sept2012
S-GB-I-064-Rev.02	Throughout Document: Updated to new format in SOP: S-GB-Q-017 *Preparation of SOPs* (most current revision or replacement). Updated SOP References throughout document. Table 7: Updated standard expiration dates. Section 11.1: Updated calibration curve fit to linear regression. Sections 11.2, 13.2 and 13.4: Added language for high/low ICVs (ICVA/ICVB). 12.1.10: Updated Soil weight language. Throughout document: Changed references to sea sand to Teflon chips, updated method references to EPA 9012B.	16Jan2014

Table 8: QC SUMMARY

Analytical Method ⇨ Quality Control Measure ⇩	**EPA 335.4 SW846 9012B** **Frequency**	**Acceptance Criteria**
Method Blank	• One per batch of samples, up to 20 environmental samples, whichever is more frequent. • Must be distilled with batch.	• Project Specific or • Less than the RL (LOWEST STANDARD IN CURVE)
Laboratory Control Spike and Duplicate	• One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent. • An LCSD is required if MS/MSD is not performed or if requested by the client. • Must be distilled with batch.	• Project Specific or • For water 90 –110% with 20% RPD • For solids 80 –120% with 20% RPD
Matrix Spike / Matrix Spike Duplicate	• One pair per batch of samples, up to 10 environmental samples, whichever is more frequent. • Must be distilled with batch.	• Project Specific or • For water 90 –110% with 20% RPD • For solids 80 –120% with 20% RPD
Initial Calibration	• Minimum of 5 standards plus blank. Must be performed every time before samples are analyzed.	• Correlation Coefficient of 0.995
CRDL	• Analyzed after Initial Calibration, but before ICV	• 60-140%
Calibration Verification (ICV/CCV)	• ICV – analyzed after calibration but before samples. **The ICV is not distilled .** • CCV – analyzed after every 10 samples. **The CCV is distilled with batch.**	• Project specific or • Recovery between 90 – 110%
Calibration Blank (ICB/CCB)	• ICB – analyzed after ICV. **The ICB is not distilled with batch.** • CCB – analyzed after every CCV pair. • **The CCB is not distilled with batch.**	• Project specific or • Less than RL (LOWEST STANDARD IN CURVE)
Leach Blank	• The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date.	• Project specific or • Less than RL (LOWEST STANDARD IN CURVE)

Table 9: DATA ASSESSMENT/CORRECTIVE ACTION

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**If these conditions are not achieved ⇒**
Method Blank and Leach Blanks	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
RLVS standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used. If the MS or MSD fail because the concentration of the spike is less than 25% of the concentration of the parent, use appropriate flag for the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, use appropriate flag for the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate precision data qualifier. Generate a Non-Conformance Memo.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

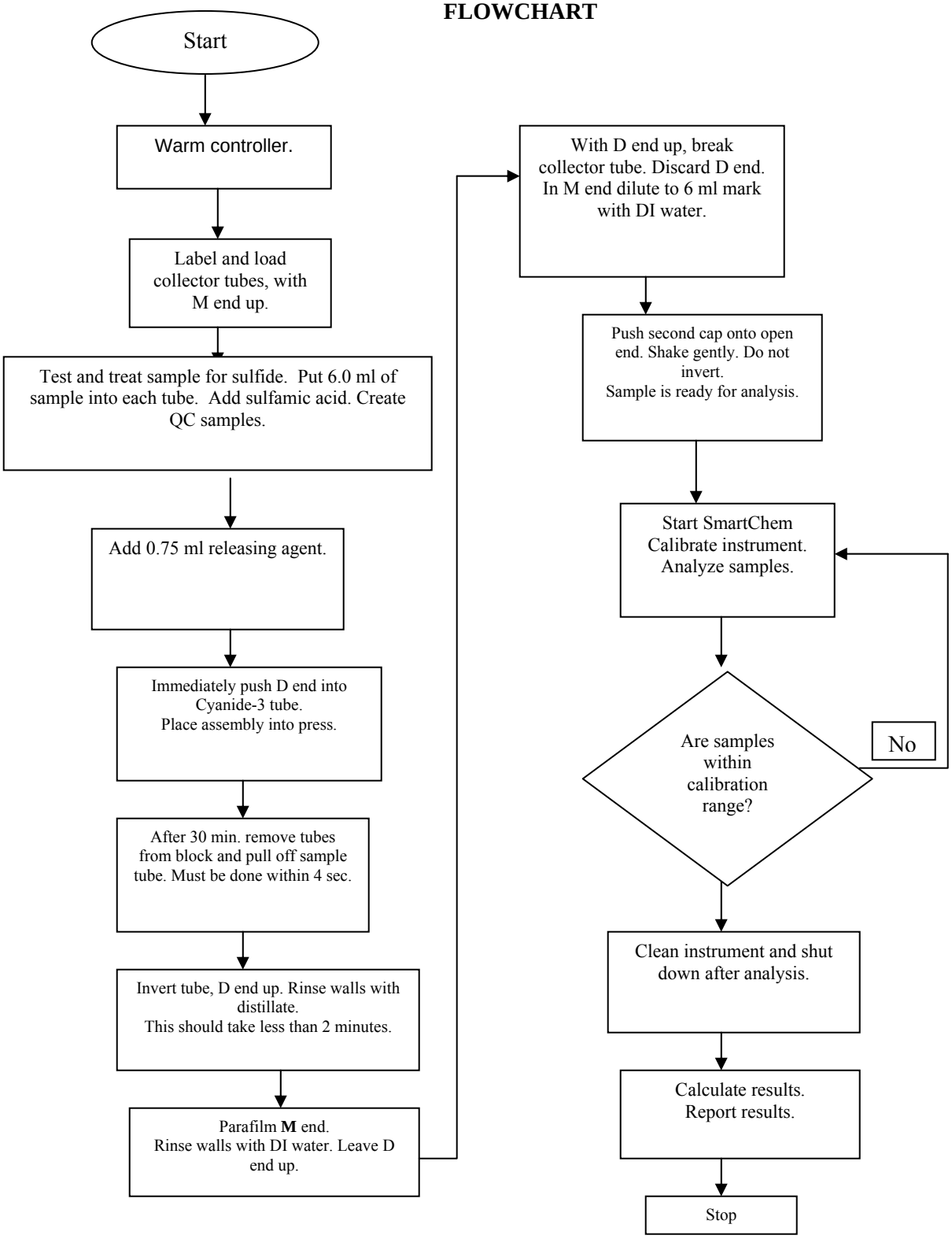
7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB do not have to be reanalyzed.

Table 10: Method Parameters

Type	End Point
Direction	Up
Decimals	2
Model	Linear
Filter 1	570 nm
Sample Blanking	No
Calibration Code	CYN4

Example of Extended Concentration Range 10 to 400 mg/L Without Sample Blanking					
Method Code: WCYN Range: 5-400 ug/L Cn Fluidics - Yes	Volume µL	Delay Time sec.	Read Time sec.	Rinse µL	Code
Sample	150				
Reagent 1 – Sodium Phosphate	63	36	0	0	CNSP
Reagent 2- Chloramine T	15	108	0	0	CNCL
Reagent 3 – Color Reagent	150	0	504	0	CNPY
Maximum Total Reaction Volume	670				
Minimum Sample and Reagent Volume	3				

Attachment I: Flowchart



Attachment 2

Updated QAPP Worksheets (Rev. 2)

QAPP Worksheet #12
Measurement Performance Criteria Table

Matrix	Soil
Analytical Group	Cyanide
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria[3]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
F-1	Cyanide: SW846: 9012B (S-GB-I-064-rev.2)	Precision	RPD ≤ 50% (or ± 4 x RL if sample or duplicate is < 5 x RL)	Field Duplicates	S & A
		Accuracy & Precision	Recovery 70-130%; RPD ≤ 35% If < 75% perform post distillation spike - see CLP decision tree; If [X] , <5 x RL use ± 2 x RL	MS/MSD[4] MD	S & A
		Accuracy	No detections exceeding the RL for the laboratory and < MDL for validation	Method Blank	A
		Accuracy	Recovery 85 – 115%, correlation coefficient > 0.995	Initial / Continuing Calibration	A
		Accuracy	Recovery 75-125% or established laboratory control limits with an LCS of similar matrix to client samples.	Laboratory Control Sample	A
		Precision	RPD ≤ 50% (or ± 4 x RL if sample or duplicate is < 5 x RL)	QA split sample[5]	S & A
		Sensitivity	70 – 130%	Low Level Standard (CRI)	A
		Field Completeness	100%	Data Completeness Check	S
		Analytical Completeness	100%	Data Completeness Check	A

Notes:

1) Reference number from QAPP Worksheet #21. Field SOPs are included in Appendix B.
2) Reference number from QAPP Worksheet #23. Laboratory SOPs are included in Appendix A.
3) The limits shown above are the data validation limits (which are also shown in Appendix G). Note that it is acceptable for the laboratory control limits (Worksheet #28) to be narrower or wider than the data validation limits listed above.
4) MS/MSDs will be performed on samples collected from the site. Note that the percent recovery criterion does not apply if the sample concentration exceeds four times the spike concentration.
5) QA split samples are collected at the discretion of the MPCA and/or USEPA for analysis by an independent laboratory. Ramsey County may also submit such samples to an independent laboratory at their discretion.

QAPP Worksheet #14
Summary of Project Tasks

Sampling Tasks:

Metals and PAH soil samples will be collected using Incremental Sampling Methodology (ISM) from each of the 22 Plexus ESA Sections. As mentioned above the basis for defining the ISM areas lying wholly or partially within the 427-acre redevelopment site to remain consistent with the 2004 Plexus ESA.

Plexus ESA Sections greater than approximately five (5) acres in size will be subdivided into roughly equally sized Decision Units (DUs). Plexus ESA Sections less than five (5) acres in size will be equal to one (1) DU (i.e., ISM area = DU). The following table presents a summary of Plexus ESA Sections and corresponding DUs subject to ISM:

Plexus ESA Sections ID(S)	ACRES	# OF DECISION UNITS (DU) WITHIN EACH Plexus ESA Sections	SIZE OF Decision UNITS (acres)
1001	12.9	NA	NA
1002	6.8	4	3.4
1003	13.9	NA	NA
1004	5.3	2	5.3
1005	26.6	10	5.3
1006	6.2	4	3.1
1007	36.7	14	5.2
1008	3.3	2	3.3
1009	3.3	2	3.3
1010	15.2	6	5.1
1011	10.2	4	5.1
1012	2.2	2	2.2
1013	19.1	NA	NA
1014	1.8	2	1.8
1015	8.9	4	4.5
1016	20.4	8	5.1
1017	3.6	2	3.6
3001, 3007, 3008	12.1	6	4.0
3002	5.0	2	5.0
4001	12.9	6	4.3
4002, 4003	15.9	8	4.0
4004, 4005	16.0	8	4.0
4006	17.1	8	4.3
4007 (minus the bldg. pad in this section)	14.17	10	0.64-4.43
4008	19.9	NA	NA
4009	61.2	24	5.1
	TOTAL	**138**	

Notes:
1) ISM Area = Plexus ESA Section
2) Documentation pertaining to the investigation and remediation of DUs 1001, 1003, 1013, building pad in 4007 and 4008 will be submitted under separate cover

For example, ISM Area 1002 is approximately 6.8 acres in size and will be divided into two approximately 3.4 acre DUs. As mentioned above if the ISM Area is less than five (5) acres in size it will not be subdivided and will be equal to a DU. Each DU will be divided into thirty (30) roughly equal polygons (see Figures 29 through 54 for DU grid layout). Thirty (30) incremental samples will be collected from each DU from 0-2 feet and composited to form one (1) bulk sample (≈7,500 g total). A second sample, using the same sampling method, will be collected from the 2-4 feet zone. Incremental samples will be collected randomly within a grid pattern. Triplicate samples will be collected from approximately 20% of the DUs (i.e., with two depth intervals at each sampling area, and 2 x 69 ISM sampling areas, there are 138 ISM samples; hence, approximately 28 of these will have triplicate sampling). These triplicate samples will be used to provide documentation of the reliability of the ISM sampling method in general and, in the DUs where triplicates are analyzed, will specifically document the reliability of the estimates of the mean concentration within the DU.

Evaluation of the ISM samples will be conducted through calculation of the 95% UCL. The parent ISM, duplicate and triplicate samples results will be compared to the 95% UCL concentration. If after calculating and evaluating the triplicate samples against the 95% UCL concentration all results are below the cleanup criteria the results will be considered acceptable and the DU ready for residential redevelopment. If after the same evaluation the triplicate samples one or more of the results exceeds the cleanup criteria, the appropriate decision will be to remediate the DU through excavation and offsite removal.

The ISM bulk samples will be sent to the laboratory for conditioning. The lab will perform the conditioning (i.e., physical breaking apart of adhered particles, removal of vegetation, gravel and debris), particle sizing and sub sampling activities. The lab-prepared subsamples will then be analyzed for metals and PAHs using standard analytical methodology.

PCB soil samples will be collected at former transformer locations or in areas where stained soils are observed indicating a surface release of oil. Such samples will be discrete samples (not ISM). Former transformer locations are shown on Figure 29 through 54.

Discrete VOC lab samples (soil) will be collected from incremental sample cores that reveal an elevated headspace result (as read by a photoionization detector equipped with a 10.6 eV lamp) greater than apparent background concentrations. Grab samples will be collected from the 0-2 foot and the 2-4 foot zones and screened using headspace baggie methodology. Grab samples for field screening will be collected after the increment is collected for the ISM bulk sample. If field-screening does not reveal elevated PID results above apparent background concentrations in any of the incremental sample cores within a given DU, then no VOC soil samples will be collected for laboratory analysis. Incremental sample cores with results exceeding 10 ppm headspace (at either depth interval), if any, will be sampled for discrete VOC sample analysis (not ISM).

Analysis Tasks:
PCBs: SW-846 Method 8082A
PAHs: Modified SW-846 Method 8270D – Selective Ion Monitoring (SIM)
Metals: SW-846 Method 6020A
Mercury: SW-846 Method 7471B
VOCs (soil): SW-846 Method 8260B

Quality Control Tasks:
The following QC samples will be analyzed for all methods: field duplicates (or ISM replicates), method blanks, laboratory control samples, matrix spikes (MS)/matrix spike duplicates (MSDs), and QA split samples (if necessary). Method 8260B and 8270D-SIM will also include: calibration (hardware tuning, internal standards, and initial/continuing calibration standards) and surrogates. Method 6020 will also include: calibration (hardware tuning, and initial/continuing calibration standards), calibration blanks (at 10% review) internal standards in each sample, interference check sample and other ICPMS interference checks , serial dilution, laboratory duplicate, and post digest spike if applicable. Method 7471B will also include: calibration and laboratory duplicates. Method 8082A will also include: calibration (initial/continuing calibration standards), surrogates, and Aroclor pattern checks.

Secondary Data:
Review and utilize the data identified in Worksheet #13.

Data Management Tasks:
Compile data into a summary table of analytical results (including corresponding Action Levels).

Documentation and Records:
Field data will be maintained in field books/field records and kept on file by Wenck. The database will be stored electronically on Wenck's network drive at the Maple Plain, Minnesota location.

Assessment/Audit Tasks:
A field audit will be conducted by the Wenck QA manager once at the beginning of the sampling activities. Laboratory audits (internal) will be conducted by the Pace Analytical Quality Manager (or designee) on an annual basis (minimum).

Data Review Tasks:
The laboratory will review all data prior to issuing an analytical report and verify analytical report completeness. Wenck will prepare field data verification and validation (100% of data). Diane Short will prepare analytical data validation reports (100% of data). Wenck will assess data usability as part of the Phase II Investigation Report. Data analysis (figures, tables, calculations, etc.) will be presented by Wenck in the Phase II Investigation Report.

QAPP Worksheet #15
Reference Limits and Evaluation Table (continued)

Matrix: Soil
Analytical Group: Metals
Concentration Level: Low

Method	SOP Number	Analytes	Action Level: Residential SRV[(1)] (mg/kg)	Action Level: Screening SLV[(1)] (mg/kg)	Project Reporting Limit Goal[(2)] (mg/kg)	Laboratory Reporting Limit[(3)] (mg/kg)
6020A	S-MN-I-492-rev.18	Antimony	12	NA	1.35	0.5
		Arsenic	9	NA	4.5	0.5
		Barium	1,100	NA	421	0.3
		Cadmium	25	NA	2.2	0.08
		Chromium[4]	87	NA	43.5	0.5
		Copper	100	NA	50	0.5
		Iron[5]	25,000	NA	12,500	50
		Lead	300	NA	150	0.1
		Manganese	3,600	NA	1,800	0.5
		Thallium	3	NA	1.5	0.1
		Vanadium[5]	50	NA	25	0.5

Matrix: Soil
Analytical Group: Mercury
Concentration Level: Low

Method	SOP Number	Analytes	Action Level: Residential SRV[(1)] (mg/kg)	Action Level: Screening SLV[(1)] (mg/kg)	Project Reporting Limit Goal[(2)] (mg/kg)	Laboratory Reporting Limit[(3)] (mg/kg)
7471B	S-MN-I-359-rev.18	Mercury	0.5	NA	0.25	0.02

Matrix: Soil
Analytical Group: Cyanide
Concentration Level: Low

Method	SOP Number	Analytes	Action Level: Residential SRV[(1)] (mg/kg)	Action Level: Screening SLV[(1)] (mg/kg)	Project Reporting Limit Goal[(2)] (mg/kg)	Laboratory Reporting Limit[(3)] (mg/kg)
9012B	S-GB-I-064-rev.2	Cyanide	60	NA	30	0.60

QAPP Worksheet #19
Analytical SOP Requirements Table (continued)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers(1 & 2)	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
Soil	Hg	Low	SW-846 Method 7471B Pace Analytical SOP# S-MN-I-359-rev.18	0.3 grams of soil for digestion	Bulks ISM Samples: 10 liter Teflon bags	</=6° C	28 days
Soil	VOCs	Low	EPA Method 5035 SW-846 Method 8260B Pace Analytical SOP# S-MN-O-521-rev.24	25 grams of soil	EnCore™ Sampler	LL: DI water, if not analyzed within 48 hours of collection sample must be frozen within 48 hours upon receipt at lab ML: Methanol Shipped: </=6° C	If not frozen upon receipt by the lab sample must be analyzed within 48 hours. If the sample is frozen, it must be analyzed within 14 days of collection.
Soil	Cyanide	Low	S-GB-I-064-rev.2	5 grams of soil	4 oz. glass jar or small plastic bottle	</=6° C	14 days
Soil	Moisture (%)	NA	Percent Moisture S-MN-I-367-rev.13	30 grams of soil	4 oz. glass jar or small plastic bottle	</= 6° C	NA

Notes:

1) The sample containers listed in the above table are appropriate for discrete soil samples. ISM bulk samples will be collected in the field and placed into new 10 liter Teflon bags.
2) Two extra containers/pairs of vials of soil will be collected at MS/MSD locations for PCB and VOC analysis. No extra jars are required at MS/MSD locations for metals and PAH analysis as samples will be collected from the ISM bulk sample.

QAPP Worksheet #20
Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical SOP Reference	No. of Sampling Locations (estimated)	No. of Field Duplicate Pairs[1]	No. of MS/ MSDs[2 & 3]	Total No. of Samples to Lab
Soil	PCBs	Low	SOP# S-MN-O-432-rev.21	TBD	TBD	TBD	TBD
Soil	PAHs	Low	SOP# S-MN-O-507-rev.20	138	N/A[4]	7	145
Soil	Metals	Low	SOP# S-MN-I-492-rev.18	138	N/A[4]	7	145
Soil	Cyanide	Low	SOP# S-GB-I-064-rev.2	TBD	TBD	TBD	TBD
Soil	Mercury	Low	SOP# S-MN-I-359-rev.18	138	N/A[4]	7	145
Soil	VOCs	Low	SOP# S-MN-O-521-rev.24	TBD	TBD	TBD	TBD

Notes:

1) Field duplicates will be collected at a minimum rate of 10% for each sampling event (see Note 4 regarding ISM replicate samples for PAHs and metals).
2) MS/MSD samples will be collected at a minimum rate of 1 per SDG (1 per group of up to 20 samples).
3) Two extra jars/pairs of vials of soil will be collected at MS/MSD locations for PCB, PAH, cyanide and VOC analysis (no extra jars are required at MS/MSD locations for metals analysis).
4) PAH and metals ISM bulk samples will be collected from within each of the 69 ISM sampling areas, from each of the two depth zones (0 to 2 feet and 2 to 4 feet). 20% of the ISM samples will be sampled in triplicate (approximately 28 ISM sample locations). The duplicate and triplicate samples will be considered blind field duplicates. These samples will be used to evaluate the reliability of the estimates of the mean concentration within that DU.

QAPP Worksheet #23
Analytical SOP References Table

Analytical SOP[(1)]	Revision Number and Date	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
SOP# S-MN-O-432	Rev.22 (2/21/13)	Definitive	PCBs	GC/ECD	Pace Analytical Minneapolis, MN	No
SOP# S-MN-O-507	Rev.20 (12/9/11)	Definitive	PAHs	GC/MS with Selective Ion Monitoring	Pace Analytical Minneapolis, MN	No
SOP# S-MN-O-540	Rev.10 (8/25/11)	Definitive	Extraction	Analytical Balance Sonicator	Pace Analytical Minneapolis, MN	No
SOP# S-MN-O-521	Rev.24 (12/4/12)	Definitive	VOCs	GC/MS	Pace Analytical Minneapolis, MN	No
SOP# S-MN-I-492	Rev. 18 (4/9/12)	Definitive	Metals	ICP/MS	Pace Analytical Minneapolis, MN	No
SOP# S-GB-I-064	Rev. 2 (1/20/14)	Definitive	Cyanide	SmartChem Analyzer	Pace Analytical Green Bay, WI	No
SOP# S-MN-I-359	Rev.18 (3/4/13)	Definitive	Mercury	CVAA Spectrophotometer	Pace Analytical Minneapolis, MN	No
SOP# S-MN-I-460	Rev.16 (4/4/13)	Definitive	Digestion	Analytical Balance	Pace Analytical Minneapolis, MN	No
SOP# S-MN-I-367	Rev.13 (1/10/13)	Definitive	Percent Moisture	Analytical Balance	Pace Analytical Minneapolis, MN	No
SOP# S-MN-L-147	Rev.0 (2/1/13)	Definitive	Sample Homogenization and Sub-Sampling	Spatulas, Analytical Balance, Sample Containers	Pace Analytical Minneapolis, MN	No
SOP# S-MN-O-495	Rev.12 (4/23/13)	Definitive	Extraction	Sonicator	Pace Analytical Minneapolis, MN	No

Notes:
1) Laboratory SOPs are included in Appendix A.II.

QAPP Worksheet #24
Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference[(1)]
GC/MS	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	Laboratory Analyst	SOP# S-MN-O-507-rev.20
	(Refer to the laboratory SOP, Section 11)	(Refer to the laboratory SOP, Section 11)	(Refer to the laboratory SOP, Section 11)	(Refer to the laboratory SOP, Section 11)	Laboratory Analyst	SOP# S-MN-O-521-rev.24
	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	Laboratory Analyst	SOP# S-MN-A-013-rev.11
GC/ECD	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	Laboratory Analyst	SOP# S-MN-O-432-rev.22
CVAA Spectro-photometer	(Refer to the laboratory SOP, Section 11)	(Refer to the laboratory SOP, Section 11)	(Refer to the laboratory SOP, Section 11)	(Refer to the laboratory SOP, Section 1	Laboratory Analyst	SOP# S-MN-I-359-rev.18
ICP/MS	(Refer to the laboratory SOP, Section 10	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	(Refer to the laboratory SOP, Section 10)	Laboratory Analyst	SOP# S-MN-I-492-rev.18
SmartChem Analyzer	(Refer to the laboratory SOP, Section 11	(Refer to the laboratory SOP, Section 11)	(Refer to the laboratory SOP, Section 13)	(Refer to the laboratory SOP, Section 16)	Laboratory Analyst	SOP# S-GB-I-064-rev.2

Notes:
1) Laboratory SOPs are included in Appendix A.II.

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Item	SOP Reference[1]
CVAA Spectrophotometer, GC/MS, GC/ECD, and ICP/MS	Preventative, Routine and Non-Routine Maintenance	S-MN-L-114-rev.09
Thermometer and Miscellaneous Equipment Monitoring	Support Equipment	S-MN-Q-264-rev.06
Standards and Reagents	Standard and Reagent Management and Traceability	S-MN-Q-275-rev.00

Notes:
1) Laboratory SOPs are included in Appendix A.II.

QAPP Worksheet #28
QC Samples Table (continued)

Matrix	Soil
Analytical Group	Cyanide
Concentration Level	Low
Sampling SOP	F-2[1]
Analytical SOP	SOP# S-GB-I-064-rev.2[2]
Field Sampling Firm	Wenck
Analytical Organization	Pace Analytical
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1 per Prep Batch[4]	< RL	Note 3	Analyst	Accuracy
LCS	1 per Prep Batch[4]	Recovery 80-120%*	Note 3	Analyst	Accuracy
MS/MSD	One pair per Batch of Samples, or up to 10 environmental samples, whichever is more frequent.[5]	Recovery 80-120%* RPD ≤ 20%	Note 3	Analyst	Accuracy and Precision
Initial and Continuing Calibration	Note 5	Note 5	Note 5	Analyst	Accuracy
Field Duplicates	Note 6	Note 6	Note 6	Note 6	Precision

Notes:
1) Field SOPs are included in Appendix B.
2) Laboratory SOPs are included in Appendix A.II.
3) Refer to Pace Analytical SOP# S-GB-I-064, Section 16.
4) A preparation batch is defined as any group of samples of the same matrix that are prepared together, up to 10 samples.
5) Refer to Pace Analytical SOP# S-GB-I-064, Section 11.
6) There are no Method/SOP acceptance limits or corrective action for these QC samples. The data validator will review the results for these QC samples and will then qualify the sample results, as necessary, based on the data qualification procedures specified in Appendix G.

QAPP Worksheet #30
Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Numbers	Analytical SOP[(1)]	Data Package Turnaround Time	Laboratory/Organization
Soil	PCBs	Low	See Figures	SOP# S-MN-O-432-Rev.22	2 weeks	Pace Analytical Minneapolis, MN
Soil	PAHs	Low	See Figures	SOP# S-MN-O-507-Rev.20	2 weeks	Pace Analytical Minneapolis, MN
Soil	VOCs	Med	See Figures	SOP# S-MN-O-521-Rev.24	2 weeks	Pace Analytical Minneapolis, MN
Soil	Metals	Low	See Figures	SOP# S-MN-I-492-Rev.18	2 weeks	Pace Analytical Minneapolis, MN
Soil	Cyanide	Low	See Figures	SOP# S-GB-I-064-Rev.2	2 weeks	Pace Analytical Green Bay, WI
Soil	Mercury	Low	See Figures	SOP# S-MN-I-359-Rev.18	2 weeks	Pace Analytical Minneapolis, MN

Notes:

1) Laboratory SOPs are included in Appendix A.II.

QAPP Worksheet #36
Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator
IIa / IIb	Soil	PCBs	Low	Notes 1 and 2	Diane Short
IIa / IIb	Soil	PAHs	Low	Notes 1 and 2	Diane Short
IIa / IIb	Soil	VOCs	Med	Notes 1 and 2	Diane Short
IIa / IIb	Soil	Metals	Low	Notes 1 and 2	Diane Short
IIa / IIb	Soil	Cyanide	Low	Notes 1 and 2	Diane Short
IIa / IIb	Soil	Mercury	Low	Notes 1 and 2	Diane Short

Notes:

1) Compliance with methods and procedures (Step IIa) will be determined by comparison with the QAPP requirements. Compliance with method performance criteria (Step IIb) will be determined by comparison with QAPP-specified performance criteria (Worksheets 12, 15, and 20). National Functional Guidelines for Organic Data Review (USEPA, June 2008) and Inorganic Data Review (USEPA, June 2010) will be used as guidance where QAPP requirements are not specific.

2) Data qualification procedures and definitions are included in Appendix G.

Attachment 3

Updated QAPP Appendix F - Analytical Data Validation Form (Rev. 2)

DATA VALIDATION FORM FOR INORGANICS

SDG:_____

PROJECT:_____

LABORATORY: Pace Analytical, Minneapolis, MN

SAMPLE MATRIX: Soil

SAMPLING DATE (Mo/Yr):_____ NO. OF SAMPLES:_____

ANALYSES REQUESTED: ICP Metals and Cyanide

SAMPLE NO._____

REVIEWER:_____

QA REVIEWER:_____ INITIALS/DATE:_____

Telephone Logs included Yes____ No____

Contractual Violations Yes____ No____

Comments:

I. DELIVERABLES

All deliverables were present as specified in the Statement of Work or project contract.
Yes____ No____

II. CALIBRATIONS

A. All initial instrument calibrations were performed as defined in the contract or Statement of Work (SOW). All correlation coefficients of the 3 point curve were > 0.995.
Yes____ No____ NA____

B. The initial calibration verification (ICV) and continuing calibration verification (CCV) standards were analyzed at the required frequency.
Yes____ No____

And the ICV and CCV standard percent recovery results were within the required control limits of 90 – 110% (80- 120% for Hg; 85 – 115% CN).
Yes____ No____

C. ICP/MS Tune and Calibrations

1. Mass calibration and resolution checks for both low and high mass isotopes and are within 0.1 amu of the true value.
Yes____ No____

2. And produced a peak width of approximately < 0.75 amu at 10% peak height.
Yes____ No____

3. Instrument stability: tuning solution was run a minimum of 3 times and RSD of absolute signals for all analytes was less than 5%.
Yes____ No____

D. Internal Standardization

1. A minimum of three internal standards were present in all standards and blanks at identical levels.
Yes____ No____
As applicable to limited list of metals.

2. The intensity of each internal standard was within the 60 - 130% method limits.
Yes____ No____

III. CRDL STANDARDS

A. The 2 x CRDL standards were analyzed as required in the SOW. The current EPA guidance no longer has a QC window for this standard. If required, limits have been 70 – 130%, (Co, Mn, Zn 50 – 150%).
Yes____ No____ NA____

IV. BLANKS

Note: the highest blank associated with any particular analyte is used for the qualification process and is the value entered after the "B" blank descriptor.

A. The initial calibration blanks (ICB) and continuing calibration blanks (CCB) were analyzed at the required frequency.
Yes____ No____ NA____

And the ICB and CCB results were less than the IDL.
Yes____ No____ NA____

B. And all analytes in the Leach Blank were less than the RL, or less than 2x the instrument detection limit (IDL), whichever is lower.
Yes____ No____ NA____

V. PREPARATION BLANKS

A. Preparation blanks were prepared and analyzed at the required frequency.
Yes____ No____

And all analytes in the preparation blank were less than the RL, or less than the instrument detection limit (IDL), whichever is lower.
Yes____ No____

B. Field, trip, decon rinse or other field blanks are contained and identified in the package.
Yes____ No____ NA____

And the reported results are less than the RL or less than the IDL, whichever is lower.
Yes____ No____ NA____

VI. ICP INTERFERENCE CHECK SAMPLE

A. The Interference check sample (ICS) was analyzed as required in the SOW or contract. The ICS consists of an A and an AB solution.
Yes____ No____ NA____

And the ICS percent recovery results were reported for all required ICS analytes and were within required control limits of 80% to 120%.
Yes____ No____ NA____

VII. SPIKE SAMPLE RECOVERY

A. A matrix (pre-digestion) spike sample was analyzed for each digestion group and/or matrix or as required in the SOW.

Yes____ No____

And the Matrix spike percent recoveries were within the required control limits of 70 – 130%.

Yes____ No____

B. A Post-digest or distillation spike was analyzed if required.

Yes____ No____ NA____

C. The MS/MSD samples were client samples

Yes____ No____

VIII. DUPLICATES

A. Matrix (pre-digestion) duplicate samples were analyzed at the required frequency

Yes____ No____

And the Matrix duplicate relative percent differences (RPD) were within the required control limits (Water 20%, 35% soil) or the RL limits were met if the duplicate values are < 5 x RL. If either one of the duplicate results are < 5 X RL, the RPD is not used. The QC limit used is the difference between the original and the duplicate results where a difference of $\pm$ the RL for water (2x RL for soil) is acceptable.

Yes____ No____

IX. LABORATORY CONTROL SAMPLE

A. Laboratory control samples (LCS) were analyzed at the required frequency.

Yes____ No____

And LCS recoveries were within the required control limits of 75 to 125% (or established laboratory control limits with an LCS of similar matrix to client samples).

Yes____ No____

X. SERIAL DILUTION

A. Serial Dilutions have been analyzed at the required frequency if the analyte concentrations are greater than 100 x IDL (ICPMS).

Yes____ No____ NA____

And the percent difference criteria of $\pm$ 10 % have been met.

Yes____ No____ NA____

XI. INSTRUMENT DETECTION LIMITS

A. The Instrument Detection Limits have met the Quarterly reporting requirements.
Yes____ No____ NA____

And all sample results have met the required detection limits (RL).
Yes____ No____ NA____

XII. PREPARATION AND ANALYSIS LOGS

A. All samples were prepared or analyzed within the required holding times referencing the SOW (time of sample receipt to preparation/distillation).
Yes____ No____

B. All samples were analyzed within the 40 CFR 136 (Clean Water Act) or method recommended holding times (time of sample collection to date of analysis).
Yes____ No____

C. Chains of Custody (COC)

1. Chains of Custody (COC) were reviewed and all fields were complete, signatures were present and cross outs were clean and initialed.
Yes____ No____

2. Samples were received at the required temperature and preservation.
Yes____ No____

XIII. FIELD QC

Field QC samples (duplicates, SRMs) were identified.
Yes____ No____
The field duplicates are

Field duplicates were within a guidance limit of < 50% RPD limit for soil. If values are < 5 x RL, the soil limit is $\pm$ 4xRL. Final determination will be made by the project manager.
Yes____ No____ NA____

XIV. GENERAL COMMENTS

Attachment 4

Updated QAPP Appendix G - Data Qualification Procedures and Data Qualifier Definitions (Rev. 2)

Data Qualifier Definitions

Twin Cities Army Ammunition Plant

Data Validation Qualifiers: Organic Data

J	estimated
JC#	calibration accuracy, # = a) a whole number for initial %RSD of the response factors (RF) or continuing calibration % difference of RFs, or b) a decimal number for RFs or if it is a 0.99xx number it is the correlation coefficient of the initial calibration curve.
JD#	MS duplicate precision, # = RPD between MS and MS duplicate (or LCS and LCSD)
JE	linear range exceedence, has no bias value
JH#	holding time exceeded, # = number of days exceeding holding time
JI#	internal standard recovery, # = percent recovery of the internal standard area counts compared to daily CCV
JL#	laboratory control sample recovery, # = percent recovery of the LCS
JN	tentatively identified compound
JP#	For two column GC work, # denotes an RPD > 40% between values
JMS#	matrix spike recovery, # = percent recovery of the spike
JS#	surrogate spike recovery, # = percent recovery of the spike
JT#	temperature exceedence, where # is the degrees over 6
R	rejected data
UMB#	blank contamination, # = highest concentration of method blank affecting data (UTB trip blank, UFB field blank)
UJ	compound was not detected in the analysis; however, the associated detection limit may not be accurate or precise
JQ	chromatographic or mass spectra identification issue
JA	preservation improper pH

Data Validation Qualifiers: Inorganic Data

J	estimated
JC#	calibration accuracy, # = percent recovery of the standard analyte or % RSD of ICPMS standards
JD#	duplicate precision, # = RPD between MS and MSD or parent sample and duplicate, use "J*" when sample result < 5 x RL and +/- CRDL criteria is failed
JE#	serial dilution interference, # = percent difference from undiluted value or note that the linear range has been exceeded
JH#	holding time exceeded, # = number of days exceeding holding time
JI#	ICP interference check sample, # = percent recovery of the ICS
JIS#	internal standard recovery, # = percent recovery of the internal standard area counts compared to the daily CCB
JK#	negative blank results, # = value of the negative blank
JL#	laboratory control sample recovery, # = percent recovery of the LCS
JMS#	matrix spike recovery, # = percent recovery of the spike
R	rejected data
UMB#	blank contamination, # = highest concentration of method blank affecting data (UCB calibration blank, UFB field blank)
UJ	compound was not detected in the analysis; however, the associated detection limit may not be accurate or precise
JA	preservation improper pH

Data Qualifying Procedures for Volatile Organic Data GCMS

QC Parameter	Control Limits	Data Qualification Procedure if Control Limits are Exceeded
Calibration (hardware tuning, response factors	Per Method and project QAPP	Laboratory must state in the Case Narrative that calibration was performed in accordance with method-specified criteria and that all performance criteria for calibration were met. If any performance criteria are not met, the laboratory must discuss the excursions and the data reviewer shall use professional judgment to determine the need for any data qualification. For data validation, the data qualification procedures described below should be followed.
Holding Time	(see Worksheet #19 of the QAPP)	If the holding time specified in QAPP Worksheet #19 is exceeded, qualify results as estimated (JH#). If the exceedance is more than two times the specified holding time, then non-detect results will be rejected.
Initial Calibration	Response Factors: RF > 0.05 (or > 0.01 for poor responders)	The method allows the lower RF for poor responders if the detection limits are appropriately elevated to adjust for instrument sensitivity. If RF < 0.05 (or < 0.01 for poor responders): Qualify all results < MDL as unusable (R). Qualify all results > MDL as estimated (JC#). Results may be biased low.
	RSD < 30% (or < 40% for poor responders)	A correlation coefficient "r" of > 0.99 is also acceptable for compounds with an RSD of > 20%. If RSD > 30% (or > 40% for poor responders): Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits.
Continuing Calibration	RRF 50 Standard: RF > 0.05 (or > 0.01 for poor responders)	The method allows the lower RF for poor responders if the detection limits are appropriately elevated to adjust for instrument sensitivity. If RF < 0.05 (or < 0.01 for poor responders): Qualify all results < MDL as unusable (R). Qualify all results > MDL as estimated (JC#). Results may be biased low.
	%D of ± 25% (or 40% for poor responders)	If %D > ± 25% (or > 40% for poor responders): Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits. If the response factors for either the ICAL or CCAL are low, the RF qualifier takes precedence.
Blanks (Method, Trip, Equipment Rinsate)	No Detections	If an analyte other than acetone, methylene chloride, or MEK is detected in the method blank at > RL, then samples should have been reanalyzed. If acetone, methylene chloride, or MEK is detected in the method blank at > 5 x RL, then samples should have been reanalyzed. If an analyte is detected in any type of blank but is not detected in any samples, then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are > 5 times the highest detection in a blank, (or > 10 times for common laboratory contaminants such as acetone, 2-butanone, methylene chloride, MEK), then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are < 5 times the highest detection in a blank, (or < 10 times for common laboratory contaminants such as acetone, 2-butanone, methylene chloride, or phthalates), then qualify those results as undetected (UMB or TB or FB#), as deemed appropriate.
Internal Standards	-50 to +100%	If the area counts are not within -50% to +100% of the daily standard area: Qualify all associated results as estimated (JI#).
Laboratory Control Sample	60 to 130% poor responders 40%	If LCS % Recovery is > 130%: Qualify results > MDL as estimated (JL#). Results may be biased high. Results < MDL are acceptable. If LCS % Recovery is 30-59%: Qualify all results as estimated (JL#). Results may be biased low. If LCS % Recovery is < 30%: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JL#). Results may be biased low.
Surrogates (System Monitoring Compounds)	50 to 130%	If spike % Recovery is > 130% for any surrogates, Qualify results > MDL as estimated (JS#). Results may be biased high. Results < MDL are acceptable. If spike % Recovery is 30-49% for any surrogates: Qualify all results as estimated (JS#). Results may be biased low. If spike % Recovery is < 30% for any surrogates: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JS#). Results may be biased low.
Matrix Spike/Matrix Spike Duplicate	50 to 130% Rec. 30% RPD (% Rec does not apply if original sample conc. is 4 or more times spike conc.)	If MS or MSD % Recovery is > 130%: Qualify results > MDL as estimated (JS#). Results may be biased high. Results < MDL are acceptable. If MS or MSD % Recovery is 30-49%: Qualify all results as estimated (JS#). Results may be biased low. If MS or MSD % Recovery is < 30%: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JS#). Results may be biased low. If the RPD for the MS vs MSD result is > 30%: Qualify results as estimated (JD#). If MS % recovery is outside the 50 to 130% limits, but the original sample concentration is 4 (or more) times the spike concentration, all results are acceptable. The RPD must be met even if the original sample concentration is 4 (or more) times the spike concentration.
Field Duplicates (and QA splits)	RPD of 50%, (unless sample or duplicate is < 5 times RL, then use ± 4 xRL)	If RPD is > 50%: Qualify all results as estimated (JD#). If the ± 4x RL criteria is exceeded: Qualify all results as estimated (J*#).

Data Qualifying Procedures for Air Volatile Organic Data GCMS TO-15

QC Parameter	Control Limits	Data Qualification Procedure if Control Limits are Exceeded
Calibration (hardware tuning, response factors	Per Method and project QAPP	Laboratory must state in the Case Narrative that calibration was performed in accordance with method-specified criteria and that all performance criteria for calibration were met. If any performance criteria are not met, the laboratory must discuss the excursions and the data reviewer shall use professional judgment to determine the need for any data qualification. For data validation, the data qualification procedures described below should be followed.
Holding Time	(see Worksheet #19 of the QAPP)	If the holding time specified in QAPP Worksheet #19 is exceeded, qualify results as estimated (JH#). If the exceedance is more than two times the specified holding time, then non-detect results will be rejected.
Canister Pressure	Per sample SOP	check initial and final pressures to ensure no leakage
Initial Calibration	Response Factors: RF > 0.05	The method allows the lower RF for poor responders if the detection limits are appropriately elevated to adjust for instrument sensitivity. If RF < 0.05 : Qualify all results < MDL as unusable (R). Qualify all results > MDL as estimated (JC#). Results may be biased low
	RSD < 30% (or < 40% for poor responders)	A correlation coefficient "r" of > 0.99 is also acceptable for compounds with an RSD of > 20%. If RSD > 30% (or > 40% for poor responders): Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits.
Continuing Calibration	RRF 50 Standard: RF > 0.05	The method allows the lower RF for poor responders if the detection limits are appropriately elevated to adjust for instrument sensitivity. If RF < 0.05 : Qualify all results < MDL as unusable (R). Qualify all results > MDL as estimated (JC#). Results may be biased low.
	%D of ± 25% poor responders ± 40%	If %D > ± 25% (or > 40% for poor responders): Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits. If the response factors for either the ICAL or CCAL are low, the RF qualifier takes precedence.
Method or Trip Blanks	No Detections	If an analyte other than acetone, methylene chloride, or MEK is detected in the method blank at > RL, then samples should have been reanalyzed. If acetone, methylene chloride, or MEK is detected in the method blank at > 5 x RL, then samples should have been reanalyzed. If an analyte is detected in any type of blank but is not detected in any samples, then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are > 5 times the highest detection in a blank, (or > 10 times for common laboratory contaminants such as acetone, 2-butanone, methylene chloride, or phthalates), then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are < 5 times the highest detection in a blank, (or < 10 times for common laboratory contaminants such as acetone, 2-butanone, methylene chloride, or phthalates), then qualify those results as undetected (UMB or UTB#), as deemed appropriate.
Internal Standards	-50 to +100%	If the area counts are not within -50% to +100% of the daily standard area: Qualify all associated results as estimated (JI#).
Laboratory Control Sample	60 to 130% poor responders 40%	If LCS % Recovery is > 130%: Qualify results > MDL as estimated (JL#). Results may be biased high. Results < MDL are acceptable. If LCS % Recovery is 30-59%: Qualify all results as estimated (JL#). Results may be biased low. If LCS % Recovery is < 30%: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JL#). Results may be biased low.
Surrogates (System Monitoring Compounds)	50 to 130%	If spike % Recovery is > 130% for any surrogate: Qualify results > MDL as estimated (JS#). Results may be biased high. Results < MDL are acceptable. If spike % Recovery is 30-49% for any surrogate: Qualify all results as estimated (JS#). Results may be biased low. If spike % Recovery is < 30% for any surrogate: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JS#). Results may be biased low.
Matrix Duplicate	< 30 % RPD (unless sample or duplicate is < 5 times RL, then use ± 2 xRL)	If RPD is > 30%: Qualify all results as estimated (JD#). If the ± 4x RL criteria is exceeded: Qualify all results as estimated (J*#). Note there is no matrix spike for air canisters.
Field Duplicates (and QA splits)	RPD of <50%, (unless sample or duplicate is < 5 times RL, then use ± 4 xRL)	If RPD is > 50%: Qualify all results as estimated (JD#). If the ± 4x RL criteria is exceeded: Qualify all results as estimated (J*#).

Data Qualifying Procedures for Semi-Volatile Organic Data GCMS-SIM

QC Parameter	Control Limits	Data Qualification Procedure if Control Limits are Exceeded
Calibration (hardware tuning, response factors	Per Method and project QAPP	Laboratory must state in the Case Narrative that calibration was performed in accordance with method-specified criteria and that all performance criteria for calibration were met. If any performance criteria are not met, the laboratory must discuss the excursions and the data reviewer shall use professional judgment to determine the need for any data qualification. For data validation, the data qualification procedures described below should be followed.
Holding Time	(see Worksheet #19 of the QAPP)	If the holding time specified in QAPP Worksheet #19 is exceeded, qualify results as estimated (JH#). If the exceedance is more than two times the specified holding time, then non-detect results will be rejected.
Initial Calibration	Response Factors: RF > 0.05	The method allows the lower RF for poor responders if the detection limits are appropriately elevated to adjust for instrument sensitivity. If RF < 0.05 : Qualify all results < MDL as unusable (R). Qualify all results > MDL as estimated (JC#). Results may be biased low.
	RSD < 35%	A correlation coefficient "r" of > 0.99 is also acceptable for compounds with an RSD of > 20%. If RSD > 35% : Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits.
Continuing Calibration	RRF Standard: RF > 0.05	The method allows the lower RF for poor responders if the detection limits are appropriately elevated to adjust for instrument sensitivity. If RF < 0.05 : Qualify all results < MDL as unusable (R). Qualify all results > MDL as estimated (JC#). Results may be biased low.
	%D of ± 25%	If %D > ± 25% : Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits. If the response factors for either the ICAL or CCAL are low, the RF qualifier takes precedence.
Blanks (Method, Equipment Reinstate)	No Detections	If an analyte other than phthalates are detected in the method blank at > RL, then samples should have been reanalyzed. If a phthalate is detected in the method blank at > 5 x RL, then samples should have been reanalyzed. If an analyte is detected in any type of blank but is not detected in any samples, then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are > 5 times the highest detection in a blank, (or > 10 times for common laboratory contaminants such as phthalates), then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are < 5 times the highest detection in a blank, (or < 10 times for common laboratory contaminants such as acetone, 2-butanone, methylene chloride, or phthalates), then qualify those results as undetected (UMB or UFB#), as deemed appropriate.
Internal Standards	-50 to +100%	Internal Standard: If the area counts are not within -50% to +100% of the daily standard area: Qualify all associated results as estimated (JI#).
Laboratory Control Sample	40 to 130%	If LCS % Recovery is > 130%: Qualify results > MDL as estimated (JL#). Results may be biased high. Results < MDL are acceptable. If LCS % Recovery is 25-39%: Qualify all results as estimated (JL#). Results may be biased low. If LCS % Recovery is < 25%: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JL#). Results may be biased low.
Surrogates (System Monitoring Compounds)	30 to 130%	If spike % Recovery is > 130% for more than 2 surrogates per fraction: Qualify results > MDL as estimated (JS#). Results may be biased high. Results < MDL are acceptable. If spike % Recovery is 20-29% for more than 2 surrogates per fraction: Qualify all results as estimated (JS#). Results may be biased low. If spike % Recovery is < 20% for more than 1 surrogate: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JS#). Results may be biased low.
Matrix Spike/Matrix Spike Duplicate	30 to 150% Rec. < 30% RPD (% Rec does not apply if original sample conc. is 4 or more times spike conc.)	If MS or MSD % Recovery is > 150%: Qualify results > MDL as estimated (JS#). Results may be biased high. Results < MDL are acceptable. If MS or MSD % Recovery is 20-29%: Qualify all results as estimated (JS#). Results may be biased low. If MS or MSD % Recovery is < 20%: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JS#). Results may be biased low. If the RPD for the MS vs MSD result is > 30%: Qualify results as estimated (JD#). If MS % recovery is outside the 30 to 150% limits, but the original sample concentration is 4 (or more) times the spike concentration, all results are acceptable. The RPD must be met even if the original sample concentration is 4 (or more) times the spike concentration.
Field Duplicates (and QA splits)	RPD of 50%, (unless sample or duplicate is < 5 times RL, then use ± 4 xRL)	If RPD is > 50%: Qualify all results as estimated (JD#). If the ± 4x RL criteria is exceeded: Qualify all results as estimated (J*#).

Data Qualifying Procedures for Aroclors (PCB) Organic Data by GC

QC Parameter	Control Limits	Data Qualification Procedure if Control Limits are Exceeded
Calibration (hardware tuning, response factors	Per Method and project QAPP	Laboratory must state in the Case Narrative that calibration was performed in accordance with method-specified criteria and that all performance criteria for calibration were met. If any performance criteria are not met, the laboratory must discuss the excursions and the data reviewer shall use professional judgment to determine the need for any data qualification. For data validation, the data qualification procedures described below should be followed.
Holding Time	(see Worksheet #19 of the QAPP)	If the holding time specified in QAPP Worksheet #19 is exceeded, qualify results as estimated (JH#). If the exceedance is more than two times the specified holding time, then non-detect results will be rejected.
Initial Calibration	RSD < 20% of 3 to 5 major peaks	A correlation coefficient "r" of > 0.99 is also acceptable for compounds with an RSD of > 20%. If RSD > 20% and "r' is outside of limits : Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits. biased low. ICAL must contain all PCBs on project list.
Continuing Calibration	%D ± 15% open 50% close for 3 to 5 of major peaks RRT	If %D > + 15% (50% close) : Qualify all results > MDL as estimated (JC#). Results < MDL are acceptable if the RF is within limits. Only Aroclor 1016 and 1260 required in CCAL Relative Retention Times are evaluated from the pattern of each Aroclor peak pattern.
Blanks (Method, Equipment Rinsate)	No Detections	If an analyte is detected in the method blank at > RL, then samples should have been reanalyzed. If an analyte is detected in any type of blank but is not detected in any samples, then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are > 5 times the highest detection in a blank, then all results are acceptable. If an analyte is detected in any type of blank and is also detected in sample(s) at concentration(s) that are < 5 times the highest detection in a blank, then qualify those results as undetected (UMB or UFB#), as deemed appropriate.
Laboratory Control Sample	50 - 150%	If LCS % Recovery is > 150%: Qualify results > MDL as estimated (JL#). Results may be biased high. Results < MDL are acceptable. If LCS % Recovery is 30-49%: Qualify all results as estimated (JL#). Results may be biased low. If LCS % Recovery is < 30%: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JL#). Results may be biased low.
Surrogates (System Monitoring Compounds)	30 to 130%	If spike % Recovery is > 130% for any surrogate: Qualify results > MDL as estimated (JS#). Results may be biased high. Results < MDL are acceptable. If spike % Recovery is 20-29% for any surrogate: Qualify all results as estimated (JS#). Results may be biased low. If spike % Recovery is < 20% for any surrogate: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JS#). Results may be biased low.
Matrix Spike/Matrix Spike Duplicate	30 to 150% Rec. < 30% RPD (% Rec does not apply if original sample conc. is 4 or more times spike conc.)	If MS or MSD % Recovery is > 150%: Qualify results > MDL as estimated (JS#). Results may be biased high. Results < MDL are acceptable. If MS or MSD % Recovery is 20-29%: Qualify all results as estimated (JS#). Results may be biased low. If MS or MSD % Recovery is < 20%: Qualify all results < MDL as unusable (R). Qualify results > MDL as estimated (JS#). Results may be biased low. If the RPD for the MS vs MSD result is > 30%: Qualify results as estimated (JD#). If MS % recovery is outside the 30 to 150% limits, but the original sample concentration is 4 (or more) times the spike concentration, all results are acceptable. The RPD must be met even if the original sample concentration is 4 (or more) times the spike concentration.
Field Duplicates (and QA splits)	RPD of 50%, (unless sample or duplicate is < 5 times RL, then use ± 4 xRL)	If RPD is > 50%: Qualify all results as estimated (JD#). If the ± 4x RL criteria is exceeded: Qualify all results as estimated (J*#).

Data Qualifying Procedures for Inorganic Data ICPMS, CVAA Mercury, CN

QC Parameter	Control Limits	Data Qualification Procedure if Control Limits are Exceeded
Calibration Verification	---	Laboratory must state in the Case Narrative that calibration was performed in accordance with method-specified criteria and that all performance criteria for calibration were met. If any performance criteria are not met, the laboratory shall discuss the excursions and the data reviewer shall use the National Functional Guidelines, in conjunction with professional judgment, to determine the need for any data qualification. If any of the calibration information described below is reviewed, the data qualification procedures described below should be followed.
Instrument Tuning and Internal Standards	Mass calibration within 0.1 amu; RSD < 5%; peak width ~ 0.75 amu at 10% peak height	If any performance criteria are not met, the laboratory shall discuss the excursions and the data reviewer shall use the National Functional Guidelines, in conjunction with professional judgment, to determine the necessary data qualification.
Initial & Continuing Calibration Verification (ICV & CCV)	90 to 110% 80-120 (Hg) 85-115% CN	If ICV and/or CCV % Recovery is > 125% (130% Hg, CN): Qualify results > IDL as unusable (R). Results < IDL are acceptable. If ICV and/or CCV % Recovery is 111-125% (130% Hg, CN): Qualify results > IDL as estimated (JC#). Results may be biased high. Results < IDL are acceptable. If ICV and/or CCV % Recovery is 75-89% (65-79% Hg, CN): Qualify all results as estimated (JC#). Results may be biased low. If ICV and/or CCV % Recovery is < 75% (65%, Hg, CN)): Qualify all results as unusable (R).
Holding Time	(see Worksheet #19 of the QAPP)	If the holding time specified in QAPP Worksheet #19 is exceeded, qualify results as estimated (JH#). If the exceedance is more than two times the specified holding time, then non-detect results will be rejected.
Initial & Continuing Calibration Blanks (ICB & CCB)	< IDL	If the absolute value of the blank result(s) exceeds the RL, all samples should have been re-digested and re-analyzed. If positive detection(s) between IDL and RL: Qualify results > IDL and up to 5 times the amount detected in the blank as undetected (UB#), as deemed appropriate. Results > 5 times the blank detection or < IDL are acceptable. If negative detection (with absolute value X2) between IDL and RL: Qualify all results up to 5 times the absolute value of the blank detection as estimated (JK#). Results > 5 times the absolute value of the blank detection are acceptable. If both positive and negative detections occur: Qualify results < IDL as estimated (UJ) and results > IDL as estimated (J).
Blanks (Method, Equipment Rinsate)	< IDL	If the absolute value of the blank result exceeds the RL, all samples should have been re-digested and re-analyzed. If positive detection(s) between IDL and RL: Qualify results > IDL and up to 5 times the amount detected in the blank as undetected (UMB or UFB#), as deemed appropriate. Results > 5 times the blank detection or < IDL are acceptable. If negative detection (with absolute value X2) between IDL and RL: Qualify all results up to 5 times the absolute value of the blank detection as estimated (JK#). Results > 5 times the absolute value of the blank detection are acceptable.
Interference Check Sample (ICPMS Metals)	80 to 120%	If ICS % Recovery is > 120%: Qualify results > IDL as estimated (JI#). Results may be biased high. Results < IDL are acceptable. If ICS % Recovery is 50-79%: Qualify all results as estimated (JI#). Results may be biased low. If ICS % Recovery is < 50%: Qualify all results as unusable (R). Interferences for Ba/Ba++ ; Ce/ CeO and other oxides are also checked and not qualified
Internal Standards	60 - 130%	Area counts of the Internal Standards in each sample compared to the daily calibration blank If IS %R > 130 %: Qualify detected results as estimated (JIS#) If spike % Recovery is 20-59%: Qualify all results as estimated (JS#). Results may be biased low. If spike % Recovery is < 20%: Qualify all results as unusable (R).
Serial Dilution	%D of $\pm$ 10%	If %D is > $\pm$ 10%: Qualify all results as estimated (JE#). If %D is > $\pm$ 10% but the original sample concentration is < 100 times the IDL, all results are acceptable.
Laboratory Control Sample	75 to 125% or soil control chart limits	If LCS % Recovery is > 125%: Qualify results > IDL as estimated (JL#). Results may be biased high. Results < IDL are acceptable. If LCS % Recovery is 50-74%: Qualify all results as estimated (JL#). Results may be biased low. If LCS % Recovery is < 50%: Qualify all results as unusable (R).
Laboratory Duplicate Sample	RPD of < 35%, (unless sample < 5 times RL, then use $\pm$ 2 x RL)	If RPD is > 35%: Qualify all results as estimated (JD#). If the $\pm$ 2x RL criteria is exceeded: Qualify all results as estimated (JD).

Data Qualifying Procedures for Inorganic Data ICPMS, CVAA Mercury, CN (cont'd)

Matrix Spike/Matrix Spike Duplicate	70 to 130% Rec. < 35% RPD (% Rec does not apply if original sample conc. is 4 or more times spike conc.)	If spike % Recovery is > 130%: Qualify results > IDL as estimated (JS#). Results may be biased high. Results < IDL are acceptable. If spike % Recovery is 30-69%: Qualify all results as estimated (JS#). Results may be biased low. If spike % Recovery is < 30%: Qualify all results as unusable (R). If the RPD for the MS vs MSD result is > 35%: Qualify results as estimated (JD#). If MS % recovery is outside the 70 to 130% limits, but the original sample concentration is 4 (or more) times the spike concentration, all results are acceptable. The RPD must be met even if the original sample concentration is 4 (or more) times the spike concentration. If MS % recovery is outside the 75 to 130% limits, analysis of a post digestion spike or serial dilution is required. If the post digestion spike % recovery is outside the QC limits of 80 to 120%, data validator will use professional judgment to qualify all associated data. Otherwise, qualify data as indicated above.
Field Duplicates (and QA splits)	RPD of < 50%, (unless sample or duplicate is < 5 times RL, then use $\pm$ 4xRL)	If RPD is > 50%: Qualify all results as estimated (JD#). If the $\pm$ 4xRL criteria is exceeded: Qualify all results as estimated (JD).