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# EPA REGION V SAMPLE CONFIRMATION FORM

FAX THIS COMPLETED FORM TO BRIAN P. FREEMAN AT US EPA by TUESDAY of the PRECEEDING WEEK BY 12 NOON! (312) 353-4342 SITE NAME/STATE Mides I / Mides I Indiana CERCLIS ID# IND 98067959 SITE SPILL ID # \_\_\_\_\_\_ SAMPLING DATE(S) Retrieve from CRL storage SAMPLER NAME/COMPANY Richard Boice, V.S. EPA, OSE SAMPLER PHONE # \_\_\_\_\_\_ G-4740 FAX # 3-5-54/ TYPE OF INVESTIGATION (eg. RI/FS, Enforcement, etc.) Treatability Study

ANALYSIS REQUIRED (eg. RAS VOA, RAS METALS or SAS type)*	MATRIX (SOIL/WATER/etc.)	Number of Samples
SAS for SVOCS in SPLP	Suil/Schditied Soil	26
SAS for PIB/posticides in SPLP	Suil / Solidified Soil	26
SAS for metals in SPLP	Soil / Solidified Soil	26
SAS For CN in SPLP	Soil / Soliditied Soil	13

\* ALL SAS WORK Scheduled through the Region REQUIRES 3-4 weeks lead time, and an approved SAS form.

NOTIFY THE FOLLOWING PERSONS PRIOR to SHIPMENT, FOR CONFIRMATION

RAS - Roger Nowakowski (SMO) (703) 519-1353 SAS - (If Scheduled through Region) - Cecilia Luckett (312) 886-1488

Sampling Coordinator Ruchard E. Korce DATE: 4/11/95 (Signature)

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SPLP SVOCS

ENVIRONMENTAL PROTECTION AGENCY FOR THE TEAMS TOPIC SUBSTANCES

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SPLP PESTICIDE/ PCDS

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SPLP PESTICIDE (PCBS

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U.S. ENVIRONMENTAL PROTECTION AGENCY CLP Sample Management Office P.O. Box 818 - Alexandria, Va. 22313 Phone: (703) 557-2490

SAS NO.

# SYNTHETIC PRECIPITATION LEACHING PROCEDURE FOR SEMIVOLATILE ORGANIC COMPOUNDS AND PESTICIDE/PCBS FOR THE MIDCO TREATABILITY STUDY SOIL SAMPLES SPECIAL ANALYTICAL SERVICES Client Request

## Regional Transmittal

Telephone Request

#### INTRODUCTION

A. EPA Region and Client: Region V, U.S. EPA

B. Regional Representative: Richard Boice, Remedial Project Manager

C. Telephone Number: (312) 886-4740

D. Date of Request: April 11, 1995

E. Site Name: Midco I and Midco II, Gary, Indiana

### DESCRIPTION OF WORK

1. General description of analytical service requested under this SAS:

The work under this Special Analytical Services Request (SAS) will be part of a treatability study being conducted by U.S. EPA to evaluate the effectiveness of soil treatment by solidification/ stabilization (S/S) at the Midco I and Midco II hazardous waste sites. This SAS provides for analysis of semivolatile organic compounds (SVOCs) and Pesticide/PCBs in the extract from the Synthetic Precipitation Leaching Procedure (SPLP) for both untreated soils and for for the S/S matrix after treatment.

Leachate from the soil and S/S matrix samples shall be generated using the Synthetic Precipitation Leaching Procedure (SPLP), Method 1312, November 1990 from proposed update to SW-846 (a copy of this is attached). The laboratory shall analyze the SPLP extracts for the Target Compound (TCL) List of SOCs and pesticide/PCBs. The SAS for the SOCs and pesticide/PCBs in the SPLP are being combined because these fractions can utilize the same extraction fluid. It is also possible to use the same SPLP extraction as used for the metals.

The TCL is from Exhibit C of OLMO1.0. The analysis of the extracts for SOCs and pesticide/PCBs shall be conducted in accordance with

the most recent version of OLM01.0, subject to the modifications listed in item 7 below.

2. Definition and number of work units: 26 samples plus 3 duplicates

3. Purpose of analysis: The overall objective of the study is to evaluate the potential effectiveness of soil treatment by S/S for use in remedial actions at the Midco I and Midco II Superfund sites, to define final performance standards, and to evaluate the performance of a number of binders.

4. Estimated date of sample collection: The untreated soil samples were collected in August 1993, and were treated with the binders in August 1993. The treated samples were cured at room temperature under humid conditions for 30 days and then placed into glass bottles with Teflon lined caps for shipment to CRL for analysis. CRL collected the needed amount of sample for their analyses and then returned the remainder of the sample to the glass jars.

5. Estimated dates and method of shipment: Since the time of analysis, the samples have been in storage at CRL at room temperature in the glass jars. CRL is requested to obtain the samples from storage for these additional analyses.

6. Schedule and reporting:

In accordance with Method 1312 (Section 6.6), the laboratory should analyze the SPLP extract as soon as possible following extraction, but in any event the laboratory shall meet the following deadlines:

- solvent extraction for the SOCs and pesticide/PCBs in the SPLP extracts shall be completed within 7 days of generation of the SPLP extracts;
- analyses of all solvent extracts must be completed within 40 days of generation of the SPLP extract (or earlier as necessary to meet the deadline in the following item);
- all analyses must be complete and the data package containing the analytical results submitted to U.S. EPA, Region V by June 30, 1995. The data package must include original copies of all sampling handling documentation (COCs, tags, SAS PLs, etc.) and data.

7. Analytical protocol and special technical instructions:

Leachate from the soil samples and S/S matrix samples shall be generated using the Synthetic Precipitation Leaching Procedure (SPLP), Method 1312, November 1990 from the proposed addition to SW-846 (see attachment) with the following modifications and conditions:

- a. The instructions in Sections 1.2, 1.3, and 1.4 of Method 1312 should be disregarded;
- b. The laboratory shall not correct the measured values of the organic compounds for the MS/MSD results as provided for in Section 8.2.5 of the SPLP.
- c. Extraction fluid #1 shall be used for SPLP extractions for SOCs and pesticide/PCBs (Section 5.4 of Method 1312). This extraction may be combined with the SPLP extraction for metals. The laboratory shall make sure that a large enough portion of the soil sample is extracted to generate enough extract for all the analyses including the MS/MSD analyses.
- d. For the untreated soils samples, the filtration procedure (Sections 7.1.1.3-7.1.1.7 of Method 1312) may produce filtrate. For the filtration procedure, the laboratory shall not discard water collecting on top of a soil sample, but this water shall be mixed with the sample prior to filtration. The SPLP extracts <u>may</u> be a combination of liquid filtrate and solid SPLP extract (Sections 7.2.13.2 and 7.3.14 of Method 1312).
- e. For the samples treated by S/S, the size reduction step in 7.1.3 of the SPLP must be implemented. The equipment to be used should be determined following inspection of the samples. A mortar and pestle or hammer and chisel may be appropriate.

The leachate generated by the SPLP shall be analyzed in accordance with the most recent version of OLM01.0 for SOCs and pesticide/PCBs (this includes the sample handling, storage and chain-of-custody requirements) with the following modifications and conditions:

- a. The same SPLP extraction can be used to analyze both SOCs, pesticide/PCBs, and metals.
- The operations in OLM01.0 to identify or quantify tentatively identified compounds need not be conducted;
- c. The laboratory shall prepare matrix spike/matrix spike duplicate sample (MS/MSD) using a portion of one of the SPLP extracts in each Group. The MS/MSD level in the SPLP extract shall be set at ten times the contract required quantitation level (CRQL) for each target organic compound (Section 8.2).
- d. Data requirements and QC requirements are further summarized on Attachment 1.
- 8. Reporting and documentation of results:

Reporting and documentation of results shall be in accordance with the requirements of Method 1312, and the most recent version of OLM01.0 as appropriate, with the following additions:

- The laboratory shall report any problems in sample preparation in the case narrative;
- The laboratory shall utilize and prepare sample preparation logs to record all variables in sample preparation provided for in Method 1312, including (but not necessarily limited to):
  - weights of extracted samples (25 gram minimum for 100% solids);
  - volume of any filtrate obtained;
  - parameters for determination of percent solids;
  - dates of each preparation step, with associated weights and measured volumes.
- Any QA/QC problem should be discussed with the Remedial Project Manager to help determine whether corrective action is needed.
- The schedule for submission of the samples data summary package is revised as described in Item 7 above.
- 9. Other:
- 10. Name of sampling/shipping contact:

Richard Boice, Remedial Project Manager, HSRL-6J U.S. EPA, Region V 77 West Jackson St. Chicago, Il. 60604 (312) 886-4740

## ATTACHMENT 1

## I. DATA REQUIREMENTS

PARAMETERS: Target Compound List of SOCs and pesticide/PCBs in Exhibit C of the most recent version of OLM01.0.

QUANTITATION LIMITS: As required in OLM01.0 for SOCs and pesticide/PCBs.

DUPLICATE PRECISION DESIRED: As provided for in OLM01.0 for SOCs, and pesticide/PCBs.

SURROGATES: Surrogates of OLM01.0 are required.

# II. QC REQUIREMENTS

The laboratory shall perform all QC requirements of OLM01.0 except as noted below.

AUDITS	FREQUENCY	LIMITS*
Blanks for extraction fluid. This will replace the required method blanks.	Per OLM01.0	Less than the CRQLs.
Surrogates	Each blank and each SPLP extract for organic	Per OLM01.0 for organics
MS/MSD	Per OLM01.0	As defined in OLM01.0

U.S. ENVIRONMENTAL PROTECTION AGENCY CLP Sample Management Office P.O. Box 818 - Alexandria, Va. 22313 Phone: (703) 557-2490

SAS NO.

# SYNTHETIC PRECIPITATION LEACHING PROCEDURE FOR METALS FOR MIDCO TREATABILITY STUDY SAMPLES AFTER TREATMENT BY SOLIDIFICATION/STABILIZATION (S/S) SPECIAL ANALYTICAL SERVICES Client Request

Regional Transmittal

Telephone Request

#### INTRODUCTION

A. EPA Region and Client: Region V, U.S. EPA

B. Regional Representative: Richard Boice, Remedial Project Manager

C. Telephone Number: (312) 886-4740

D. Date of Request: April 11, 1995

E. Site Name: Midco I and Midco II, Gary, Indiana

## DESCRIPTION OF WORK

1. General description of analytical service requested under this SAS:

The work under this Special Analytical Services Request (SAS) will be part of a treatability study being conducted by EPA to evaluate the effectiveness of soil treatment by solidification/stabilization (S/S) at the Midco I and Midco II hazardous waste sites. This SAS provides for analysis of metals in the extract from the synthetic precipitation leaching Procedure (SPLP) for both untreated soils and for the S/S matrix after treatment. The results will be used to evaluate the performance of S/S.

The laboratory shall generate extracts from the untreated soils, and from the S/S matrix using the Synthetic Precipitation Leaching Procedure (SPLP), Method 1312 (leaching fluid #1), November 1990 from proposed update to SW-846 (a copy of this is attached). The laboratory shall analyze the SPLP extract for metals in accordance with ILM02.0. 2. Definition and number of work units: 26 samples plus 3 duplicates

3. Purpose of analysis:

The overall objective of the study is to evaluate the potential effectiveness of soil treatment by S/S for use in remedial actions at the Midco I and Midco II Superfund sites, to define final performance standards for the S/S, and to evaluate the performance of a number of binders. The metals of primary concern in evaluating the effectiveness of the S/S are: arsenic; barium; cadmium; chromium; copper; lead; nickel; vanadium; and zinc.

4. Estimated date of sample collection: The untreated soil samples were collected in August 1993, and were treated with the binders in August 1993. The treated samples were cured at room temperature under humid conditions for 30 days and then placed into glass bottles with Teflon lined caps for shipment to CRL for analysis. CRL collected the needed amount of sample for their analyses and then returned the remainder of the sample to the glass jars.

5. Estimated dates and method of shipment: Since the time of analysis, the samples have been in storage at CRL at room temperature in the glass jars. CRL is requested to obtain the samples from storage for these additional analyses.

6. Schedule:

In accordance with Method 1312 (Section 6.6), the laboratory must analyze the SPLP extract as soon as possible following extraction. It is requested that CRL extract and analyze the samples, submit the Sample Data Package, submit the data in computer readable form, and complete the SDG file by June 30, 1995.

7. Analytical protocol and special technical instructions:

The laboratory shall generate leachate from the S/S samples using procedures in the Synthetic Precipitation Leaching Procedure (SPLP), Method 1312, November 1990 from the proposed addition to SW-846 (see attachment) with the following modifications and conditions:

- a. The laboratory shall disregard the instructions in Sections 1.2, 1.3, and 1.4 of Method 1312.
- b. Extraction fluid #1 shall be used for the SPLP extracts that will be analyzed for metals (Method 1312, Section 5.4.1). The same SPLP extraction can be used for the metals and for the SVOCs and pesticide/PCBs. The laboratory shall make sure that a large enough portion of the soil sample is extracted to

generate enough extract for all the analyses including the matrix spike analyses.

c. For the samples treated by S/S, the size reduction step in 7.1.3 of the SPLP must be implemented. The equipment to be used should be determined following inspection of the samples. A mortar and pestle or hammer and chisel may be appropriate.

The laboratory shall analyze the SPLP extracts using procedures in the most recent version of ILM02.0 (including sample handling, storage and chain-of-custody requirements) for inorganics (excluding cyanide and mercury) with the following modifications and conditions:

- a. Precise results for the SPLP before and after treatment are needed to determine the percentage reductions for arsenic, barium, cadmium, chromium, copper, lead, nickel, vanadium, and zinc. The results for the other metals are only being used as background information and screening purposes. Therefore, analysis of the metals other than those listed above shall only be by ICP only, even if the detection limit goals of ILM02.0 are not met. Since precise results are needed, it is unacceptable to analyze undigested extract as suggested in Section 7.2.14 of Method 1312 to determine whether it is above or below a regulatory limit.
- b. The laboratory need only prepare matrix spikes for the following parameters: arsenic; barium; cadmium; copper; lead; nickel; vanadium; and zinc. The laboratory shall prepare one matrix spike. The matrix spike level in the SPLP extract shall be set at ten times the contract required detection limits (CRDL) for arsenic, barium, chromium, and lead. The matrix spike level shall be set at five times the CRDL for cadmium, copper, nickel, and vanadium. The spike level shall be set at 100 times the CRDL for zinc. The purpose of these dilutions is so that the spiked sample will be in the general range of the concentration limits listed on the attached Table If the leachate after treatment with a given binder meets 1. the concentration limits or the percentage reductions listed in Table 1, the binder satisfies the Minimum Performance Standards for the S/S treatment.
- f. The laboratory shall not correct the measured values of the metals for the spiked sample results as provided for in Method 1312, Section 8.2.5. Since the untreated samples have been weathered for many years, and the treated samples are solidified, the SPLP extract is not expected to have high TDS. However, the results should be screened for the potential need to correct for matrix effects using the MS/MSD results, and interelement corrections made as needed. The need for any unusual procedures should be discussed with the Remedial Project Manager. If it is determined that reanalysis of some

samples is necessary because of the potential need to correct for matrix affects, EPA will notify the laboratory of the samples that need to be reanalyzed and the spike levels that shall be used.

- g. The laboratory shall dilute all samples to within the calibration range even if reanalysis is required.
- h. The laboratory shall use the furnace technique if necessary pursuant to the most recent version of ILM02.0 for the following metals: arsenic; cadmium; chromium; and lead.
- i. The SPLP extracts for metals shall be preserved by acidification to a pH less than 2 unless precipitation occurs (Method 1312, Sections 6.6 and 7.2.14). If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible (Method 1312, Section 7.2.14).
- j. Data requirements and QC requirements are further summarized on Attachment 1.
- 8. Reporting and documentation of results:

Reporting and documentation of results shall be in accordance with the requirements of Method 1312 and the most recent version of ILM02.0 subject to the revisions noted in item 7 above and the following additions:

- The laboratory shall report any problems in sample preparation in the case narrative;
- The laboratory shall utilize and prepare sample preparation logs to record all variables in sample preparation provided for in Method 1312, including (but not necessarily limited to):
  - weights of extracted samples (25 gram minimum for 100% solids);
  - volume of any filtrate obtained;
  - preliminary evaluation of percent solids;
  - parameters for determination of percent solids;
  - dates of each preparation step, with associated weights and measured volumes.
- Any QA/QC problems should be discussed with the Remedial Project Manager to help determine whether corrective action is needed.
- 9. Other:

10. Name of sampling/shipping contact:

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Richard Boice, Remedial Project Manager, HSRL-6J U.S. EPA, Region V 77 West Jackson St. Chicago, Il. 60604 (312) 886-4740

#### I. DATA REQUIREMENTS

PARAMETERS: Target Analyte List of metals in Exhibit C of the most recent version of ILM02.0 excluding cyanide and mercury.

DETECTION LIMITS: As required in ILM02.0 for the following metals: arsenic; barium; cadmium; chromium; copper; lead; nickel; vanadium; and zinc. For the other metals whatever detection limit is attained using the ICP will be acceptable. If necessary, detection limits can be compromised to make necessary background corrections.

DUPLICATE PRECISION DESIRED: As provided for in ILM02.0.

## II. QC REQUIREMENTS

The laboratory shall perform all QC requirements of ILM02.0 as modified by item 8 except as noted below.

AUDITS	FREQUENCY	LIMITS
Analyze extraction fluid blank, and analyze preparation blank using extraction fluid.	One for each delivery group or batch.	Less than CRDL, or less than 10% of the concentration limits listed in Table 1 (attached).
Spiked samples (SPLP extracts spiked)	One for each delivery group or batch.	As defined in ILM02.0.

Alternatively, the SPLP treated can be reduced to the following Concentration Limits. If SPLP is below a Concentration Limit listed below, no further reduction of leachate concentration is required, although the treated sample should not increase' such leachate concentration above the concentration limit.

TABLE 1.

	1110	
CONSTITUENT	PERCENTAGE	CONCENTRATION
	REDUCTION	LIMIT (ug/l)
arsenic	90	50
barium	90	2000
cadmium	95	5
chromium	95	100
copper	95	43
lead	99	15
nickel	95	100
vanadium	90	233
zinc	90	1150

#### STABILIZATION OF ORGANICS

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Using total waste analyses (using methylene chloride extraction for semivolatile organics, and methanol extraction for volatile organics), a 50% reduction in concentrations shall be attained based on total waste analyses of the sample of untreated waste (TWA rew waste ) and the sample treated by S/S (TWA treated ) calculated in accordance with the formula: U.S. ENVIRONMENTAL PROTECTION AGENCY CLP Sample Management Office P.O. Box 818 - Alexandria, Va. 22313 Phone: (703) 557-2490

SAS NO.

# SYNTHETIC PRECIPITATION LEACHING PROCEDURE FOR CYANIDE FOR THE MIDCO TREATABILITY STUDY SOIL SAMPLES (GROUP 3) SPECIAL ANALYTICAL SERVICES Client Request

Regional Transmittal

Telephone Request

INTRODUCTION A. EPA Region and Client: Region V, U.S. EPA

B. Regional Representative: Richard Boice, Remedial Project Manager

C. Telephone Number: (312) 886-4740

D. Date of Request: April 10, 1995

E. Site Name: Midco I and Midco II, Gary, Indiana

### DESCRIPTION OF WORK

1. General description of analytical service requested under this SAS:

The work under this Special Analytical Services Request (SAS) will be part of a treatability study being conducted by U.S. EPA to evaluate the effectiveness of soil treatment by solidification/ stabilization (S/S) at the Midco I and Midco II hazardous waste sites. This SAS provides for analysis of cyanide in the extract from the synthetic precipitation leaching Procedure (SPLP) for both untreated soils and for the S/S matrix after treatment. The results will be used to evaluate the performance of S/S.

Leachate from the untreated soil samples and from the S/S matrix samples shall be generated using the Synthetic Precipitation Leaching Procedure (SPLP), Method 1312, November 1990 from proposed update to SW-846 (a copy of this is attached). The laboratory shall analyze the SPLP extract for cyanide. Cyanide in the SPLP is being analyzed separately because it requires a difference extraction fluid than the other inorganic compounds. The analyses of the extracts for cyanide shall be in accordance with the most recent version of ILM02.0.

2. Definition and number of work units: 13 samples plus two duplicates.

3. Purpose of analysis: The overall objective of the study is to evaluate the potential effectiveness of soil treatment by S/S for use in remedial actions at the Midco I and Midco II Superfund sites.

4. Estimated date of sample collection: The untreated soil samples were collected in August 1993, and were treated with the binders in August 1993. The treated samples were cured at room temperature under humid conditions for 30 days and then placed into glass bottles with Teflon lined caps for shipment to CRL for analysis. CRL collected the needed amount of sample for their analyses and then returned the remainder of the sample to the glass jars.

5. Estimated dates and method of shipment: Since the time of analysis, the samples have been in storage at CRL at room temperature in the glass jars. CRL is requested to obtain the samples from storage for these additional analyses.

6. Schedule and reporting:

In accordance with Method 1312 (Section 6.6), the laboratory should analyze the SPLP extract as soon as possible following extraction, but in any event the laboratory shall meet the following deadlines:

- all analyses must be complete and the data package containing the analytical results submitted to U.S. EPA, Region V within 35 days of generation of the last SPLP extract within a sample delivery group, or by June 30, 1995, whichever is earlier. The data package must include original copies of all sampling handling documentation (COCs, tags, SAS PLs, etc.) and data.
- 7. Analytical protocol and special technical instructions:

Leachate from the soil samples and the S/S matrix shall be generated using the Synthetic Precipitation Leaching Procedure (SPLP), Method 1312, November 1990 from the proposed addition to SW-846 (see attachment) with the following modifications and conditions:

- a. The instructions in Sections 1.2, 1.3, and 1.4 of Method 1312 should be disregarded;
- b. The particle size reduction step (Section 7.3.6 of Method 1312) shall implemented for the S/S matrix samples, but is not expected to be needed for the untreated soil samples.
- c. To test samples for CN, the laboratory must conduct the extraction in a zero head space extraction (ZHE) vessel and utilize ZHE extract collection and transfer devices (Sections 4.2.1, 4.3.1, 4.6 of Method 1312).

- d. Extraction fluid #3 shall be used for the SPLP extracts that will be analyzed for cyanide (Section 5.4 of Method 1312). The reagent water used for the extraction must meet the conditions identified in both Sections 5.2.1 and 5.2.2.
- e. For the untreated soil samples, the filtration procedure (Sections 7.1.1.3-7.1.1.7 of Method 1312) may produce filtrate. For the filtration procedure, the laboratory shall not discard water collecting on top of a soil sample, but this water shall be mixed with the sample prior to filtration. The SPLP extracts <u>may</u> be a combination of liquid filtrate and solid SPLP extract (Sections 7.2.13.2 and 7.3.14 of Method 1312).

The leachate generated by the SPLP shall be analyzed in accordance with the most recent version of ILM02.0 for cyanide (this includes the sample handling, storage and chain-of-custody requirements) with the following modifications and conditions:

- a. Data requirements and QC requirements are further summarized on Attachment 1.
- 8. Reporting and documentation of results:

Reporting and documentation of results shall be in accordance with the requirements of Method 1312, the most recent version of ILM02.0 as appropriate, and with the following:

- The laboratory shall report any problems in sample preparation in the case narrative;
- The laboratory shall utilize and prepare sample preparation logs to record all variables in sample preparation provided for in Method 1312, including (but not necessarily limited to):
  - weights of extracted samples (25 gram minimum for 100% solids);
  - volume of any filtrate obtained;
  - parameters for determination of percent solids;
  - dates of each preparation step, with associated weights and measured volumes.
- Any QA/QC problem should be discussed with the Remedial Project Manager to help determine whether corrective action is needed.
- The schedule for submission of the samples data summary package is revised as described in Item 7 above.
- 9. Other:

10. Name of sampling/shipping contact:

Richard Boice, Remedial Project Manager, HSRL-6J U.S. EPA, Region V 77 West Jackson St. Chicago, Il. 60604 (312) 886-4740

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# ATTACHMENT 1

# I. DATA REQUIREMENTS

PARAMETERS: cyanide.

QUANTITATION LIMITS: As required in the most recent version of ILM02.0.

DUPLICATE PRECISION DESIRED: As provided for in ILM02.0.

# II. QC REQUIREMENTS

The laboratory shall perform all QC requirements of ILM02.0 except as noted below.

AUDITS	FREQUENCY	LIMITS
Blanks for extraction fluid. This will replace the required method blanks.	Per ILM02.0.	Per ILM02.0
MS/MSD	Per ILM02.0	Per ILM02.0.