# Residuals Unit Sampling/ Analysis Work Plan Guidance

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Maine Department of Environmental Protection 17 State House Station Augusta, ME 04333-0017

# Introduction:

Chapter 419 of Maine's Solid Waste Rules states in part: "The residual generator must develop and implement a waste characterization sampling and analytical work plan and, if required, a site monitoring plan in accordance with Chapter 405. The frequency of sampling must be adequate to represent the residual, soil or other media. The Department will require a site monitoring plan when it determines in a program or site license that a utilization program poses a potential threat to public health or safety or the environment because of the nature of the residuals utilized and/or the location, design and operation of a utilization site."

Chapter 405 includes the following excerpt, plus it details thirteen required components of a sampling/ analytical work plan.

Waste Characterization Sampling and Analytical Work Plan. A waste characterization sampling and analytical work plan must be developed for initial and ongoing characterization of solid wastes, including residuals, handled under a solid waste license. The plan must include, at a minimum, a detailed description of the contaminants of concern in the waste or residual, the sampling and analytical methods used to obtain samples and quantify contaminants, and the guidance or other references used to develop the plan. The plan must include all the information necessary to address subparagraphs (a) and (b), below, and be prepared in accordance with the specific analytical requirements of Section 6.C or 6.D.

Guidance for each of the thirteen required components is given in this document.<sup>1</sup> Information for this guidance has been taken from the following sources:

- (i) Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, November 1997; Commonwealth of Pennsylvania, Department of Environmental Protection [3620-BK-DEP2214 2/98]
- (ii) POTW Sludge Sampling and Analysis Guidance Document, August 1989; for United States Environmental Protection Agency; by Science Applications International Corporation
- (iii) Soil Testing Handbook for Professionals in Agriculture, Horticulture, Nutrient and Residuals Management, third edition, 1997; Bruce R. Hoskins, Maine Soil Testing Service/Analytical Lab, Maine Forestry & Agricultural Experiment Station, University of Maine
- (iv) Recommended Soil Testing Procedures For The Northeastern United States, 2nd Edition, Northeastern Regional Publication No. 493, December 15, 1995; Agricultural Experiment Stations of Connecticut, Delaware, Maine, Maryland, Massachusetts, New Hampshire, New Jersey, New York, Pennsylvania, Rhode Island, Vermont, and West Virginia.
- (v) Chapter 419: Agronomic Utilization of Residuals, Maine Solid Waste Rules
- (vi) Chapter 405: Water Quality Monitoring, Leachate Monitoring, and Waste Characterization, August 12, 1998; Maine Solid Waste Rules
- (vii) **Test Methods for Evaluating Solid Waste** (SW-846), Update IV, June, 2005; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response

<sup>&</sup>lt;sup>1</sup> For more information please consult the reference guides listed here or at the end of each section.

- I. Identification of parameters to be analyzed and selection rationale: Chapter 405 details testing requirements for sewage sludge for land application. Analyses are dependent on factors such as volume of sludge produced, and what types of wastes the plant processes.
  - A. Soil Nutrient Analysis: Soil nutrient analysis is required for sludge application areas.
    - Test methods: Methods for nutrient analysis have been developed for Maine/New England to best evaluate soils for this region. Analytical methods for soil nutrients should be equivalent to those listed in Tables A-1 and A-2 of the Recommended Soil Testing Procedures for the North Eastern United States, second edition, 1995; University of Delaware Agricultural Experimental Station [bulletin 493]. Laboratories using these methods should participate in the National Proficiency Testing Program. Interpretive methods used for the initial solid nutrient analysis should be equivalent to those used at the Maine Soil Testing Service of the University of Maine. For more information see the Soil Testing Handbook for Professionals in Agriculture, Horticulture, Nutrient and Residuals Management [Third Edition] Written/Revised by Bruce R. Hoskins; or phone the Maine Soil Testing Service, UMO at (207) 581-2945.
    - 2. Initial Analysis: The following parameters must be analyzed for prior to using biosolids on the site: available calcium, available magnesium, available phosphorus, available potassium, cation exchange capacity, percent CEC saturation with calcium, percent CEC saturation with magnesium, percent CEC saturation with potassium, percent CEC saturation with sodium, percent organic matter and pH.
    - 3. On-going analysis: A minimum of one composite topsoil sample per eight (8) acres of utilization area must be collected at the site prior to utilization each year that a residual will be land applied. Results of the analyses must be received and interpreted by the license holder prior to utilization. These results must be used as a factor in determining the amount of residual to be land applied. Sampling for areas less than eight acres may be necessary if the cropping practices vary across the area. You should consult with a soil scientist or other qualified individual if you have questions regarding representative soil sampling.
  - B. Residual Analysis
    - Recommendations and requirements for initial residual analysis are listed in tables B1, B2, and B3. Testing requirements are detailed in Chapter 405 of the Maine Solid Waste Rules, and depend on the types of waste processed in your treatment plant. Table B1 lists general requirements. Table B2 lists requirements for larger POTWs, and those who accept industrial wastes. Table B3 includes special testing for certain types of sludge treatment.

Additionally available nitrogen may be required by the Department. Percentages of organic nitrogen mineralized after sewage sludge is land applied can be taken from Table 419.6 in Appendix A of Maine's Solid Waste Rules, Chapter 419. Alternatively protocols developed at the Maine Soil Testing Service, UMO or other approved lab procedures may be used. Table 419.6 is reproduced below:

# Table 419.6 Percentages of Organic Nitrogen Mineralized after Sewage sludge of various types are land applied

Years after	Type of Sewage Sludge			
sludge application	Primary and waste	Aerobically	Anaerobically	Composted
	activated	Digested	Digested	
0 -1	40	30	20	10
1 - 2	20	15	10	5
2-3	10	8	5	3
3-4	5	4	3	3

For more information about alternative nitrogen mineralization testing contact the Maine Soil Testing Service, UMO at (207) 581-2945.

Table B1

Parameter	Acceptable Methods	Container	Preservation	Hold Time
Ammonia	undefined in 405.6D	plastic or glass	Cool 4°C	28 days
	SM-4500-NH3		$H_2SO_4$ pH <2 [aqueous]	
	other DEP approved			
arsenic	SW-846 Method 7060	plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 7061			
	SW-846 Method 6010			
	other DEP approved			
cadmium	SW-846 Method 7130	plastic or glass	pH<2 HNO₃ [aqueous]	6 months
	SW-846 Method 7131	-		
	SW-846 Method 6010			
	other DEP approved			
calcium	SW-846 Method 6010	plastic or glass	pH<2 HNO₃ [aqueous]	6 months
	SW-846 Method 7140			
	other DEP approved			
calcium carbonate	Calculation from calcium/r	magnesium results	2.497 [Ca result] + 4.118	[Mg result]
equivalents	[same as SM 2340B]	-		
chloride	undefined in 405.6D	plastic or glass	none	28 days
	SW-846 Method 9056			
	other DEP approved			
chromium	SW-846 Method 6010	plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 6020			
	SW-846 Method 7190			
	SW-846 Method 7191			
	other DEP approved			
copper		plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 7211			
	SW-846 Method 6010			
	other DEP approved			
iron		plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 7380			
	SW-846 Method 7381			
lead	SW-846 Method 7420	plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 7421			

Parameter	Acceptable Methods	Container	Preservation	Hold Time
	SW-846 Method 6010			
	other DEP approved			
magnesium	SW-846 Method 6010	plastic or glass	pH<2 HNO₃ [aqueous]	6 months
	SW-846 Method 7450			
	other DEP approved			
mercury	SW-846 Method 7470	plastic or glass	Cool 4°C	28 days
	SW-846 Method 7471		pH<2 HNO <sub>3</sub> [aqueous]	
	other DEP approved			
molybdenum	SW-846 Method 7480	plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 7481			
	SW-846 Method 6010			
	other DEP approved			
nickel	SW-846 Method 7520	plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 6010			
	SW-846 Method 6020			
	SW-846 Method 7521			
	other DEP approved			
Nitrate	undefined in 405.6D	plastic or glass	Cool 4°C	28 days
	SM-4500-NO3		H <sub>2</sub> SO <sub>4</sub> pH <2 [aqueous]	
	SW-846 Method 9056			
	other DEP approved			
Nitrite	undefined in 405.6D	plastic or glass	Cool 4°C	28 days
	SW-846 Method 9056		H <sub>2</sub> SO <sub>4</sub> pH <2 [aqueous]	
	other DEP approved			
percent dry solids	undefined in 405.6D	plastic or glass	Cool 4°C	7 days
	SM-2540 G			
	other DEP approved			
рН	undefined in 405.6D	plastic or glass	none	24 hours [liquids]
	SW-846 Method 9045			
	SM-4500 H <sup>+</sup>			
	other DEP approved			C manth a
salt toxicity	electrical conductivity	plastic or glass	none	6 months
	other DEP approved			C manth a
selenium	SW-846 Method 7740	plastic or glass	pH<2 HNO <sub>3</sub> [aqueous]	6 months
	SW-846 Method 7741			

Parameter	Acceptable Methods	Container	Preservation	Hold Time
	SW-846 Method 7051 SW-846 Method 6010 other DEP approved			
sodium	undefined in 405.6D SW-846 Method 6010 SW-846 Method 7770 other DEP approved	plastic or glass	pH<2 HNO₃ [aqueous]	6 months
TCLP (full suite)	SW-846 Method 1311	glass, PFTE-lined cap	Cool 4°C (for VOC analysis)	14 days (for VOC analysis)
total carbon	undefined in 405.6D SM-5310 B SW-846 Method 9060 other DEP approved	amber glass with TFE lined caps	Cool 4°C H <sub>2</sub> SO <sub>4</sub> pH <2 [aqueous]	
total Kjeldahl nitrogen	undefined in 405.6D SM-4500-N <sub>org</sub> EPA 351.3 other DEP approved	plastic or glass	Cool 4°C	28 days
total phosphorus	undefined in 405.6D SW-846 Method 6010 other DEP approved	plastic or glass	pH<2 HNO₃ [aqueous]	6 months
total potassium	undefined in 405.6D SW-846 Method 6010 SW-846 Method 7610 other DEP approved	plastic or glass	pH<2 HNO₃ [aqueous]	6 months
total volatile solids	undefined in 405.6D SM-2540 G other DEP approved	plastic or glass	Cool 4°C	7 days
zinc	SW-846 Method 7950 SW-846 Method 6010 SW-846 Method 6020 SW-846 Method 7951 other DEP approved	plastic or glass	pH<2 HNO₃ [aqueous]	6 months

Additional parameters for sewage sludge generated by POTWs with an average daily flow greater than 2.5 millions of gallons/day; POTWs with pulp and paper, tannery, textile-related or other significant industrial wastewater inputs; POTWs required to enact an

Industrial Pretreatment Program according to U.S. EPA regulations 40 CFR Part 403; and sludge or residuals from pulp and paper mills, tanneries, textile mills, and ash generators.

Parameter	Approved Methods	Container	Preservation	Hold Time
Dioxins	EPA 1613 SW-846 Method 8290 other DEP approved	amber glass, PFTE- lined cap	Cool 4°C	30 days
Dioxin TEQs	calculated from Dioxins d	ata as per Chapter 405 Ta	able 405.1 and Table 405.2	
target SVOCs	SW-846 Methods other DEP approved	glass, PFTE-lined cap	Cool 4°C Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> [aqueous with residual chlorine]	14 days [solids] 7 days [aqueous]
target VOCs	SW-846 Methods other DEP approved	glass, PFTE-lined cap	Cool 4°C HCl pH <2 [aqueous] Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> [aqueous with residual chlorine] Methanol preservation [solids] **See appropriate preparation method	14 days
total PCBs	SW-846 Method 8082 SW-846 Method 8270 other DEP approved	glass, PFTE-lined cap	Cool 4°C Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> [aqueous with residual chlorine]	14 days [solids] 7 days [aqueous]

# Table B2

# Special tests that may be required

# Table B3

Test	Methods	Notes		
Compost stability	Aerated Pile	See Chapter 405.6.D(2)g(i): temperature monitoring in a compost pile		
	Dewars Flask	See Chapter 405.6.D(2)g(ii): temperature monitoring in a container		
	Respiration	ASTM method D5975-96 Standard Test Method for Determining the Stability of		
		Compost by Measuring Oxygen Consumption		
	other DEP approved			
Pathogens:		Following treatment by one or more of the pathogen reduction standards, residuals		
Salmonella	SM 9260 D	which may contain human pathogens may require compliance testing for one or more		
Fecal Coliform	SM 9221 D or E	of these indicator parameters		
Enteric virus	ASTM D 4994-89			
Helminth ova	EPA 600/1-87-014			
	other DEP approved			
Target pesticides	SW846 8081	May be required based on a description of the process generating the residual		
	other DEP approved			
The Department may	The Department may require analysis for other parameters that, based on a description of the process generating the residual, may			
be in the residual in significant concentrations to adversely impact the utilization program.				

2. Follow-up Residual Analysis.

Follow-up sampling and analysis. The frequency of follow-up sampling and analysis will be established by license condition, and determined based on the initial analytical results for the residual, the Department's data base of analytical results, the potential for these compounds to be present in the material, and other factors as appropriate.

Sewage sludge must be analyzed for total arsenic, cadmium, chromium, copper, lead, mercury, molybdenum, nickel, selenium and zinc at the frequency in Table 405.3 unless otherwise approved by the Department based on specific characteristics.

Dry Tons of Sludge	Sampling and Analysis	Analysis Results - Reports
Produced Annually	Frequency	Due on 15th of Month
		Listed
<200	Twice yearly	July, January
200 - 1000,	Quarterly	April, July, October,
		January
1001 - 2000	Bi-monthly	March, May, July,
		September, November,
		January
>2000	Monthly	Each month

Table 405.3

# II. sample collection methods including a description of sampling equipment and how representative samples will be obtained

Sample collection methods should be tailored to your facility. Representative sampling is one of the most important aspects of monitoring. According to <u>Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge</u>, November 1997; Commonwealth of Pennsylvania, Department of Environmental Protection: "Collecting a representative sample of sewage sludge that is stored before the land application may be more difficult than collecting a representative sample of sewage sludge that is land applied continuously. For example, samples of the sewage sludge at different locations in a storage pile and at various depths may have to be collected and then composited to obtain a representative sample of the stored sewage sludge. When sewage sludge is applied to the land continuously, a single sample may be appropriate."

The sampling technique varies depending on whether the sewage sludge is flowing through pipes, moving on a conveyor, or stored in a pile or bin. Sewage sludge that flows through pipes or moves on a conveyor should be sampled at equal intervals during the time the unit operates in a day. When sampling from piles or bins, core samples should be taken from as many as points in the pile or bin as possible.

It is important to note that special precautions are required to collect samples for volatile organic analysis. Composite samples are not recommended for these samples, and measures should be taken to reduce loss of these contaminants.

Sampling methods are described in <u>Test Methods for Evaluating Solid Waste</u> (SW-846), Update III, December 1996; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response Chapter 10, and <u>POTW Sludge</u> <u>Sampling and Analysis Guidance Document</u>, August 1989; for United States Environmental Protection Agency; by Science Applications International Corporation. Sample equipment is described in <u>Test Methods for Evaluating Solid</u> <u>Waste</u> (SW-846), Update III, December 1996; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response Chapter 9.

#### III. sample point description:

Depending on the type of sewage sludge material (liquid, dewatered or dried) and the treatment process, certain sampling points will provide better samples. "To obtain a representative sample of sewage sludge, the sample must be taken from the correct locations and represent the entire amount of sewage sludge or the batch process. In some situations, the sample point location may have a dramatic effect on the monitoring results. It is important that samples be collected from a location representative of the final sewage sludge that will be land applied. Because the pollutant limits pertain to the quality of the final sewage sludge applied to the land, samples must be collected after the last treatment process. Samples should be taken from the same point and in the same manner each time a sample collection or monitoring is performed. The sampling location should be safe and accessible."<sup>2</sup>

The following table of sludge sampling points has been taken from POTW Sludge Sampling and Analysis Guidance Document, August, 1989.

Sludge Type	Sampling Point
Anaerobically	Sample from taps on the discharge side of positive displacement
Digested	pumps.
Aerobically Digested	<ul> <li>Sample from taps on discharge lines from pumps. If batch digestion is used, sample directly from the digester. Two cautions are in order concerning this practice:</li> <li>(1) If aerated during sampling, air entrains in the sample. Volatile organic compounds may purge with escaping air.</li> <li>(2) When aeration is shut off, solids separate rapidly in well-digested sludge.</li> </ul>
Thickened Sludges	Sample from taps on the discharge side of positive displacement pumps.
Heat Treatment	<ul> <li>Sample from taps on the discharge side of positive displacement pumps after decanting. Be careful when sampling heat treatment sludge because of:</li> <li>(1) High tendency for solids separation</li> <li>(2) High temperature of sample (frequently &gt;600C as sampled) can cause problems with certain sample containers due to cooling and subsequent contraction of entrained gases.</li> </ul>

# TABLE 2.2. SLUDGE SAMPLING POINTS

<sup>&</sup>lt;sup>2</sup> Sampling Manual for Pollutant Limits, Pathogen and Vector Attraction Reductions in Sewage Sludge, November 1997; Commonwealth of Pennsylvania, Department of Environmental Protection [3620-BK-DEP2214 2/98]

Sludge Type	Sampling Point
Dewatered,	Sample from material collection conveyors and bulk containers.
Dried,	Sample from many locations within the sludge mass and at various
Composted, or	depths
Thermally	
Reduced	

Dewatered:

Sludge Type	Sampling Point
Belt Filter Press, Centrifuge, Vacuum Filter Press	Sample from sludge discharge chute.
Sludge Press (plate and frame)	Sample from the storage bin; select four points within the storage bin, collect equal amount of sample from each point and combine.
Drying Beds	Divide bed into quarters, grab equal amounts of sample from the center of each quarter and combine to form a composite sample of the total bed. Each grab sample should include the entire depth of the sludge (down to the sand).
Compost piles	Sample directly from front-end loader as the sludge is being loaded into trucks to be hauled away or stored.

#### IV. sample size, sample type (e.g., grab, composite), and sample frequency: According to <u>Sampling Manual for Pollutant Limits</u>, Pathogen and Vector Attraction Reductions in Sewage Sludge, November 1997; Commonwealth of Pennsylvania,

Department of Environmental Protection

"In determining whether a sample should be collected using a single grab or composite sampling method, the following factors may be evaluated:

- How well the sewage sludge is mixed?
- Whether the sample is collected from a single batch of sewage sludge or from a stockpile made up of several batches?
- o Whether the composition of the sewage sludge varies over time?

In general, compositing several samples may provide a more representative sample than collecting one grab sample. Therefore, in most situations, composite samples should be performed. Sewage sludge is most often land applied in a solid form and may be treated in batch processes. Sewage sludge characteristics may also vary over time. For these reasons, the quality may vary from day to day or even within the sewage sludge volume itself due to the inability to completely mix sewage sludges that have high solids contents." Sample frequency should be set to adequately address this variation.

Proper sample size considerations include taking a large enough sample to be representative, but small enough to be conveniently transported. Table 4A represents minimal sample size requirements for the laboratory to perform requested testing.

Parameter	Aqueous Collection (ml)	Solid Collection (g)
Inorganic Analytes (except mercury)	600	200
Mercury	400	200
PCBs	1000	100
Volatile Organic Compounds	40	fill VOA jar to reduce headspace as much as possible (check with lab for individual method used)
Semi-volatile Organic Compounds	1000	100
Pesticides	1000	100

# Table 4A: Sample Size Guide\*\*

\*\*Note: Check with your laboratory for more information about sample size requirements for specific methods.

# V. procedures for decontamination of sampling equipment prior to sampling and between the collection of successive samples:

Adherence to proper decontamination protocol helps ensure samples are free from cross contamination. In general equipment should be washed with suitable detergent [Liquinox® or equivalent], rinsed with deionized or distilled water and dried. Some procedures include solvent rinses or oven drying steps. Specialized decontamination procedures are included in individual sampling methods and equipment owner's manuals. See Test Methods for Evaluating Solid Waste, Update III; December, 1996; Volume II, Chapter 10 or equivalent for sampling methods.

- VI. sample container, storage and preservation procedures: see (I.)
- VII. sample holding times: see (I.)

# VIII. sample handling, packaging, and transportation protocols

According to <u>POTW Sludge Sampling and Analysis Guidance Document</u>, August 1989; for United States Environmental Protection Agency; by Science Applications International Corporation:

- Sample containers must be packaged to reduce the risk of leakage. They should be held upright and cushioned from shock. Sufficient insulation/ refrigerant should be added to maintain 4°C temperature for shipment.
- Unpreserved samples without heavy contamination are not regulated under DOT. These samples may be shipped packaged as above using a commercial carrier. Transit time should be held to <24 hours.
- When environmental samples are preserved as recommended, they may be shipped as non-hazardous samples.

These guidelines assume no material is present in the samples at concentrations which would result in a "hazardous" DOT rating. Since hazardous waste cannot be land applied, these samples will likely be well

below DOT hazardous waste levels. Municipal sewage sludges labeled as hazardous are usually from failed EP Toxicity tests and occasionally from reactivity tests.<sup>3</sup>

#### IX. sample documentation (labeling, chain-of-custody, log book):

Adequate documentation of sludge sampling activities is important for general program quality assurance/quality control, and required by most monitoring regulations. Proper sampling activity documentation includes proper sample labeling, chain-of-custody procedures and a log book of sampling activities. The number of people in the chain of custody should be kept to a minimum to limit the possibility of contamination and to increase accountability.

#### Sample Labeling

Labels and ink should be waterproof. Fix labels to containers with clear waterproof tape. Tape completely around container and over label to prevent accidental label loss or ink smear during shipping and handling.

Sample labels should include the following information at a minimum:

- Sample Number (specific to sampling event i.e. location)
- Type of sample, i.e., grab, 24 hour composite, etc.
- Collector

Additional information helpful for sample identification includes:

- Sampling Organization Name
- Facility Name (being sampled)
- Bottle Number (specific to container
- Date, Time (24 hour time is preferable, i.e., 1600 vs. 4:00 p.m.)
- Sample Location
- Preservatives
- Analytical Parameter(s)
- Special Conditions or Remarks.

#### Chain-of-Custody

Each sample shipment requires a chain-of-custody record. A chain-ofcustody document provides a record of sample transfer from person to person. This document helps protect the integrity of the sample by ensuring that only authorized persons have custody of the sample. In addition, the chain-of-custody procedure ensures an enforceable record of sample transfer which is necessary if the sample results are to be used in a judicial proceeding alleging violations of sludge standards. This document shall record each sample's collection and handling history from time of collection until analysis as well as the information listed on each sample bottle. All personnel handling the sample shall sign, date and note the time of day on the chain-of-custody document.

<sup>&</sup>lt;sup>3</sup> Should hazardous material (as defined by DOT) be present, DOT regulations concerning packaging, transportation and labeling must be followed (see 49 CFR Parts 172, 173 and 178). A material is considered hazardous by DOT if it fails one of the four characteristic tests of: corrosivity, ignitability, reactivity and EP Toxicity [see Test Methods for Evaluating Solid Waste, SW 846, 1986 for exact methods].

Sampling Log Book

All sampling activities should also be documented in a bound log book. This book duplicates all information recommended for the chain-of-custody document above, and notes all relevant observations regarding sample stream conditions.

For additional information please see:

- <u>POTW Sludge Sampling and Analysis Guidance Document</u>, August 1989; for United States Environmental Protection Agency; by Science Applications International Corporation
- <u>Test Methods for Evaluating Solid Waste</u> (SW-846), Update III, December 1996; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response
- X. analytical methods:

see (I.)

XI. estimated practical quantitative limits for each parameter to be quantified: Reporting limits should be available from the laboratory used for analysis.

#### XII. sampling and analysis quality assurance/quality control procedures:

A quality assurance (QA) program should be developed and implemented at the facility to achieve the desired quality in sample collection, laboratory analysis, data validation and reporting, documentation and record keeping, According to <u>Test</u> <u>Methods for Evaluating Solid Waste</u> (SW-846), Update III, December 1996; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response a QA program will typically address the following major areas:

- Proper collection procedures, equipment, preservation methods and chain of custody procedures to ensure representative samples.
- Proper sample preparation procedures, instruments, equipment and methodologies used for the analysis of samples.
- Proper procedures and schedules for calibration and maintenance of equipment and instruments associated with the collection and analysis of samples.
- Proper record keeping to produce accurate and complete records and reports, when required.

Quality control (QC), which is part of the QA program, relates to the routine use of established procedures and policies during sample collection and analysis. The objective of QC procedures is to ultimately control both the accuracy and the precision of all analytical measurements. QC for sample collection includes the use of spiked and split samples, use of specific sampling protocol, proper decontamination of sampling equipment, and the choice of appropriate analytical methods and procedures. Laboratory quality assurance procedures should be available from the laboratory used for analysis.

Examples of sampling/ sample handling QA/QC include [as applicable]:

- Strict adherence to standard sampling procedures.
- Providing training for all sampling personnel.

- Requiring all personnel to read, sign, and follow the Sampling and Analysis Work Plan.
- Strict adherence to sample handling protocol.
- Monitoring temperature of sample refrigerators.

For more information, see Chapter One of Test Methods for Evaluating Solid Waste (SW-846), Update III; December, 1996.

 XIII. data reduction, validation and reporting methods including methods of statistical interpretation of analytical results: Data reduction, validation and reporting methods should be part of a laboratory's Quality Assurance Manual. Chapter One of Test Methods for Evaluating Solid Waste (SW-846), Update III; December, 1996 provides further guidance.

Any statistical review of analytical results should be described, and all formulas given.

If computer spreadsheets are used to manipulate data received from a laboratory, a print out of the spreadsheet showing calculation formulas may be attached to the SAP.

For further information please consult the references listed in the introduction or contact the Department of Environmental Protection, Solid Waste Division, Residuals Utilization Unit at (207) 287-2651.