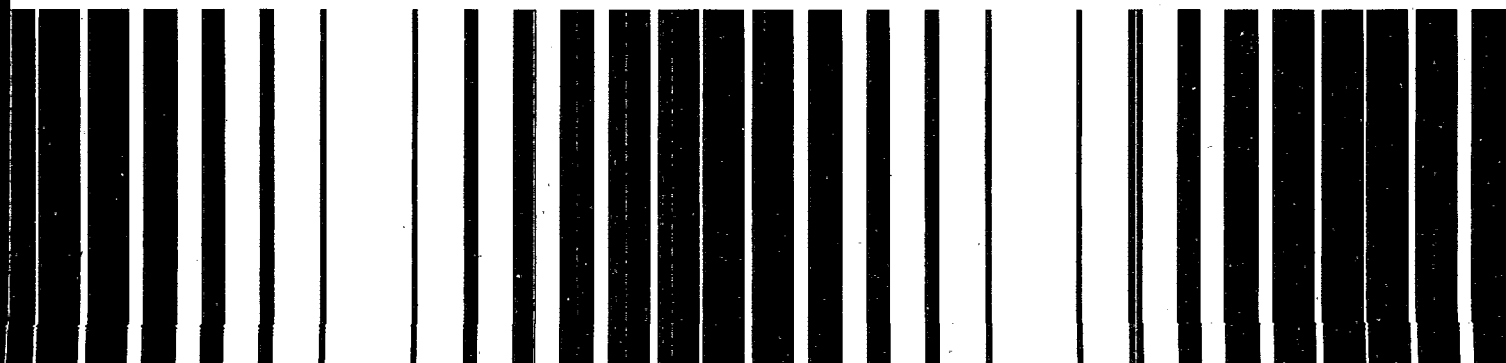


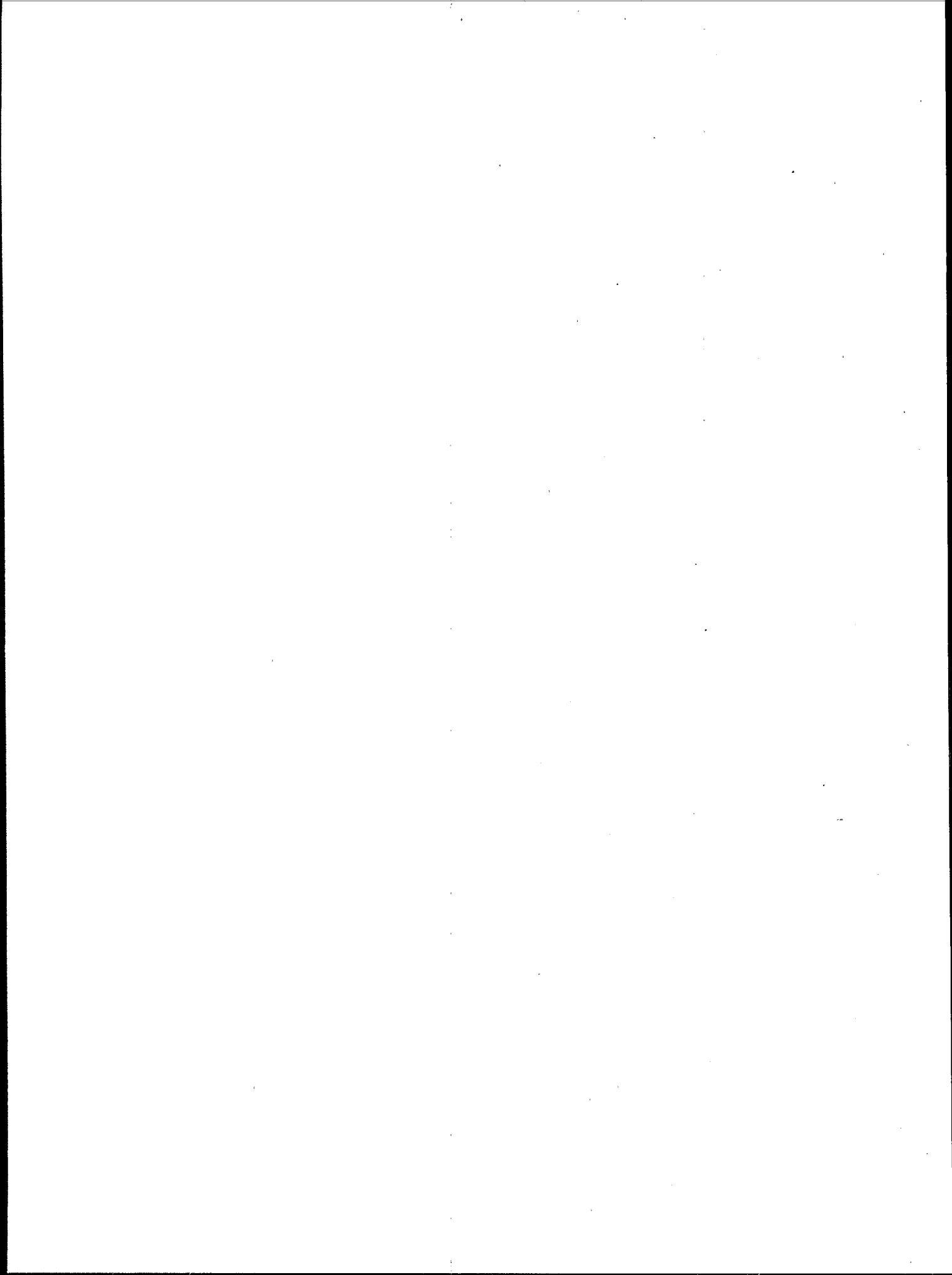


Handbook

Hazardous Waste Incineration Measurement Guidance Manual

Volume III of the Hazardous Waste Incineration Guidance Series





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Office of Solid Waste and Emergency Response
U.S. Environmental Protection Agency
Washington, DC 20460

Air and Energy Engineering Research Laboratory
Research Triangle Park, NC 27711
and
Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268



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Notice

This document has been reviewed in accordance with the U.S. Environmental Protection Agency's peer and administrative review policies and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

The guidance document is intended to provide information on how regulatory requirements in 40 CFR Subpart O may be satisfied in a wide variety of situations. This guidance document is not, in and of itself, a regulatory requirement and should not be regarded as such. Therefore, although compliance with regulatory requirements is mandatory, compliance with this guidance manual (although useful as a means of satisfying regulatory obligations) is not.

Abstract

This document provides general guidance to permit writers in reviewing the measurement aspects of incineration permit applications and trial burn plans. It is oriented to how measurements are made, not what measurements to make. The guidance deals specifically with commonly required measurement parameters and measurement methods for process monitoring, sampling and analysis aspects of trial burns and subsequent operation of the incinerator, and quality assurance/quality control (QA/QC) associated with these activities. As a guidance tool, this document introduces the major elements of incineration measurements via sample checklists, general discussion, and technical references.

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Section 1 Introduction

Subtitle C of the Resource Conservation and Recovery Act (RCRA) requires the U.S. Environmental Protection Agency (EPA) to develop, promulgate, and implement regulations which control the generation, transportation, treatment, storage, and disposal (TSD) of hazardous wastes. An integral part of these activities is reviewing and issuing permits to hazardous waste incineration facilities.

Several documents have been or are being prepared as a Hazardous Waste Incineration Guidance Series prepared by EPA to assist both the applicant and the permit writer in the RCRA Part B process leading to a final operating permit for hazardous waste incinerators. This document on measurement guidance is Volume III of this series. Below is a list of additional guidance manuals in the series:

Volume I *Guidance Manual of Hazardous Waste Incinerator Permits*, Mitre Corporation, SW-966, NTIS PB84-100577, July 1983 (document scheduled for revision). This describes the overall incinerator permitting process, highlights the specific guidance provided by other manuals, and addresses permitting issues not covered in the other manuals such as treatment of data in lieu of a trial burn. Thus it can be viewed as a road map and a good summary of all permitting issues.

Volume II *Guidance on Setting Permit Conditions and Reporting Trial Burn Results*, Acurex, EPA-625/6-89-019, 1989. Includes guidance on selecting key operating parameters, translating trial burn data into permit operating conditions, and reporting trial burn results. Also discusses planning a trial burn to achieve workable permit limits.

Volume IV *Guidance on Metals and Hydrogen Chloride for Hazardous Waste Incinerators*, Versar, 1989 (draft under EPA review). Specific guidance on limiting metals emissions from incinerators is provided. In particular, a risk assessment approach to setting limits on metal components in the waste is employed. Guidance is also provided on doing risk-based checks on HCl emissions. (Note: Earlier title was

Guidance for Permit Writers for Limiting Metal and HCl Emissions from Hazardous Waste Incinerators.)

Volume V *Guidance on PIC Controls for Hazardous Waste Incineration*, Midwest Research Institute, 1989 (draft under EPA review). Details the specific permit requirements for CO and total hydrocarbon (THC) emissions from hazardous waste incinerators in the RCRA system. Emission limits for CO and THC and the rationale for their selection are discussed. (Note: Earlier title was: *Guidance on Carbon Monoxide Controls for Hazardous Waste Incineration.*)

Volume VI *Proposed Methods for Measurement of CO, O₂, THC, HCl, and Metals at Hazardous Waste Incinerators*, 1989 (draft under EPA review). Presents a draft measurement method for the above parameters including performance specifications for continuous CO monitors.

In addition, a document has been prepared to assist regulatory personnel in observing trial burns: *Trial Burn Observation Guide* EPA-530/SW-89-027 March 1989, Midwest Research Institute, September 1988. Includes general guidance on preparation, on-site activities, and reporting aspects of observing a trial burn test.

The Hazardous Waste Incineration Measurement Guidance Manual provides general guidance to permit writers in reviewing the measurement aspects of incineration permit applications and trial burn plans. It is oriented to how measurements are made, not what measurements to make. The guidance deals specifically with commonly required measurement parameters and measurement methods for process monitoring, sampling and analysis aspects of trial burns and subsequent operation of the incinerator, and quality assurance/quality control (QA/QC) associated with these activities.

As a guidance tool, this document introduces the major elements of incineration measurements via general discussion and technical references. It is not intended to specify a complete list of measurements that should be required in every case, nor does it provide complete descriptions of all pertinent methods. The references cited provide additional descriptions of measurement

methods, and at times, measurement experts may need to be consulted for judgments in unusual situations. Testing, operating, and permitting hazardous waste incinerators typically involves best engineering and scientific judgment on a case-by-case basis. No single publication can answer *all* of the possible technical and regulatory questions related to incineration measurements.

The following sections of the report cover recommendations to permit reviewers, methods, and QA/QC. Section 2 provides some specific recommendations to permit reviewers on various aspects of the trial burn plan and permit application. Section 3 gives commonly used

methods for measuring the monitoring, sampling, and analysis parameters. Section 4 discusses the QA/QC procedures which should be addressed for the measurement methods in the quality assurance plan. Section 5 gives the references used in the report. Appendix A reproduces guidance for analyses methods given in EPA-600/8-84-002 and in SW-846. Appendix B provides checklists to be used by permit writers to help assess the completeness of measurement information in trial burn plans and incinerator permit applications. The checklists include process monitoring parameters, sampling parameters, analysis of samples, and QA/QC for the process monitoring and sampling and analysis parameters.

Section 2

Specific Recommendations for Permit Reviewers

Many aspects of RCRA incinerator trial burns are not specifically addressed in the regulations (40 CFR Parts 264 and 270). This can result in different judgments or decisions made by RCRA permit reviewers as they review permit applications. Case-by-case judgments are appropriate at times; however, a more uniform decision-making process among reviewers is needed. This section provides specific recommendations for some aspects of the trial burn and the reporting of the results to promote this uniformity.

2.1 Trial Burn Runs

Three replicate runs are recommended for each specific set of incinerator operating conditions. Sufficient waste feed must be stockpiled or readily available in order that the same waste characteristics are used for replicate runs. All three replicate runs should comply with the RCRA requirements for destruction and removal efficiency (DRE), particulate emissions, and hydrogen chloride (HCl) emissions. This provides added assurance that the incinerator can repeatedly meet the standards. If the incinerator fails only some of the standards (e.g., only particulate), measurement of only those standards that failed can be considered for a retest, provided that the key operating conditions remain the same and that any modification to the incinerator would not negatively affect the unit's ability to comply with the other performance standards.

2.2 Trial Burn Schedule

- Generally, one run per day should be scheduled. This can vary depending upon the complexity of the specific trial burn.
- A trial burn schedule indicating the overall test schedule and an anticipated daily schedule, similar to the examples shown in Table 1 and Figure 1, should be included in the trial burn plan. The daily schedule in Figure 1 assumes 2 h of actual sample time for the semivolatile organic sampling train (semi-VOST) and three volatile organic sampling train (VOST) trap pairs at a 40-min sampling time per pair, as an example.

2.3 Monitoring

- The regulations call for continuous monitoring of combustion temperature, waste feed rate, the indication of combustion gas velocity, and CO in the stack gas [40

CFR 264-347(a)(1)]. Monitoring of key process parameters should be as continuous as feasible. A recommended minimum requirement for selected key parameters is reading a measurement value at least every 15 s and recording a value at least every minute. Strip charts and/or data loggers can be used to record data. If these minimum requirements cannot be met, then justification for why less frequent measurement/recording times are acceptable should be provided by the applicant. Continuous monitoring with alarms or automatic shutdown at specified set points, without permanent data recording, may be appropriate for some parameters. Volume VI of the Guidance Series (referenced in Section 1) contains draft performance specifications for CO monitors.

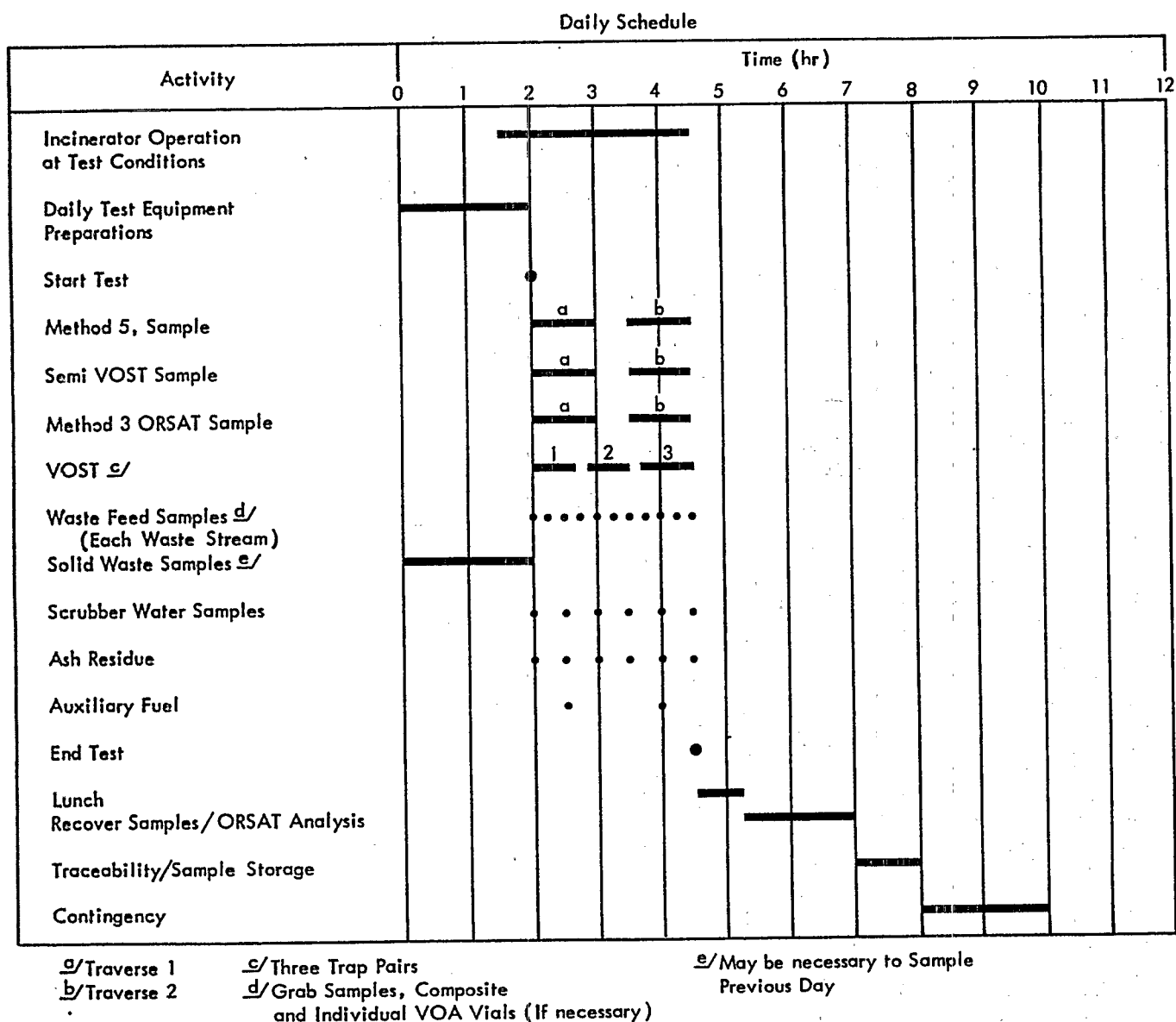
Table 1. Example of a Trial Burn Schedule

Prior to test	<ul style="list-style-type: none">• Incinerator shutdown/site modifications• "Miniburns"• Monitor(s) calibration/evaluation• Preparation of special wastes• Pretest meeting(s)
Day 1	Arrive on-site Set-up Sample solid wastes
Day 2	Complete set-up Sample solid wastes Preliminary measurements
Day 3	Run 1
Day 4	Run 2, audits
Day 5	Run 3
Day 6	Pack equipment/leave site
Day 4-8	Samples arrive at lab
Day 35-50	Sample analysis complete
Day 35-60	Preliminary results reported
Day 95	Test report submitted

2.4 Sampling and Analysis

- Waste feed samples should be collected every 15 min over the entire period of stack sampling and then be composited into one sample per test run for each waste feed. The applicant may justify less frequent sampling if data are provided to show that the

Figure 1. Example daily schedule.



homogeneity and composition of the waste feed do not vary.

- Each drum burned during the trial burn should be sampled, unless the applicant can justify why this is not necessary. Grab samples should be composited for each waste type.
- All other process samples (e.g., scrubber water, ash, etc.) should be taken every 30 min over the entire

stack sampling period, and then be composited into one of each sample type per test run. In some cases it may not be feasible to comply with this recommendation for ash sampling.

- Sampling should not begin until the incinerator operation has reached steady-state on waste feed. A minimum of 30 min of operation feeding waste is recommended, or for a rotary kiln, the greater of 30 min or the solids residence time.

- Sampling should continue through incinerator operating abnormalities unless the waste feed cutoff system shuts the incinerator down. If sampling is stopped during a trial burn, the test may be completed using the same sampling trains if the burn is completed on the same day it was started.
- Minimum stack sampling time for each run (actual sampling time not including time for port changes, changing VOST trap pairs, etc.) should be 1 h for EPA Method 5 (M5), semi-VOST, and VOST. Data from less than 1 h of sample collection would be an invalid test run. Two hours of stack sampling time is recommended as optimal. In some cases more than 2 h of sampling may be required, e.g., to achieve required detection limits. A minimum of three VOST trap pairs per run is also recommended. A fourth pair is often taken in case one pair is broken or lost due to analysis problems.
- All sampling required for a test run in the trial burn should, whenever possible, be conducted concurrently, with only the normal minor differences associated with different sampling methods (e.g., MM5 and VOST schedules in Figure 1). However, differences in sampling period start and finish due to sampling problems are allowable. Examples are a particulate train that fails a leak check at port change and sampling must be restarted with a new train, a VOST trap that breaks at the end of the sampling period so that another pair must be run, or sequential sampling required due to limitations in available sampling ports. However, all waste feed sampling must be continued for the entire period, and possibly any water effluent or ash sampling. Also, the incinerator must continue at the same process operating conditions with collection of the operating data for the entire period.
- Separate semi-VOST (SW-846 Method 0010), commonly called Modified Method 5 (MM5), and Method 5 (M5) should be used for semivolatile and nonvolatile POHCs and particulates, respectively. This is necessary since drying the particulates collected and probe rinse prior to weighing may result in loss of semivolatile POHCs.
- Semi-VOST sampling trains with XAD resin traps should not be used to collect samples for HCl analysis. XAD resins may be contaminated with chlorides prior to sampling or HCl may be retained in the XAD during sampling.
- Condensate collected in impingers after the XAD resin module in a semi-VOST sampling train should always be considered part of a semivolatile POHC sample. The condensate should be extracted and the extract combined with the extracts from other portions of the sampling train.
- The final leak check for VOST should be run at the highest vacuum used during the sampling run but not less than 1 in of mercury vacuum.
- A sampling train which develops problems during a trial burn may be validated on a case-by-case basis if it can be shown that the results were not significantly biased. For example, if an M5 train passed the leak check after sampling in the first port but failed the confirming leak check before beginning sampling in the second port due to a probe liner being broken during port change, the test could be allowed to continue after replacement of the probe liner and including rinsing of the broken liner for particulate recovery. However, if the train failed the leak check after removal from the first port, the sample would be invalid, even if it were believed that the probe liner was broken as the probe was removed from the port (i.e., it is not possible to know if the probe liner was already broken before removal from the port).
- POHCs which have a boiling point between 100° and 140°C may break through the XAD-2 resin in a semi-VOST if the sampling time is too long or may be difficult to purge from a VOST trap. The validity of the method chosen for these POHCs should be determined prior to testing (see Section 3.4.5.4). Experts in analytical chemistry may need to be consulted to determine the appropriate sampling method for these POHCs.
- Volatile POHCs should be sampled with the VOST (SW-846 Method 0030), if possible. Samples may be collected in bags if VOST samples cannot be performed. The bag sample procedure is less desirable due to potential problems with adsorption in the bag and loss of sample. Stability of the POHC to be sampled in the bag should be checked prior to sampling, if this method is used. Field blanks are essential with bag sampling.
- VOST field blanks are required, and VOST trip blanks and laboratory blanks are highly recommended.
- It is recommended that both the front (Tenax) and back (Tenax/charcoal) trap of all pairs of traps from each run be analyzed separately. The results of these separate analyses allow determination of the percent distribution of the volatile POHCs collected on the front and back traps. The samples are considered valid (no breakthrough) if the back trap contains no more than 30% of the quantity collected on the front trap. This criteria does not apply when the quantity of sample collected is low (i.e., less than 75 ng on the back trap). Some latitude in judging the validity of a sample versus this criteria can be considered based on how closely the DRE standard was met and how closely the trap distribution criteria was met. For example, if the DRE was 99.999% and the distribution was slightly higher than 30% on the back trap, one could still have confidence a 99.99% DRE was achieved. Use of three traps in series (Tenax-Tenax-Tenax/charcoal) can be considered to provide further indication of sample validity, however, use of this approach should be reviewed by an expert experienced in VOST sampling and analysis.
- The VOST protocol allows multiple pairs of traps from the same run to be combined for analysis, if increased analytical sensitivity is required. In rare cases where

this increased sensitivity is needed, a reasonable approach is to combine all front traps from a test run for one analysis and all back traps for a second analysis. This procedure retains information on distribution of POHCs between the front and back traps while allowing the greater sensitivity. The same criteria described above is used to judge the validity of the samples.

- The trial burn plan should show that a sufficient quantity of POHCs is in the waste feed to demonstrate a destruction and removal efficiency (DRE) of 99.99%, given the VOST or semi-VOST sample size and lower detection limits of the analytical instrumentation. An example calculation based on VOST sampling is shown in Table 2. Generally, a concentration of 100 ppm in the waste is considered a minimum detectable level for POHCs.
- Traceability procedures must be used for handling all samples. Full chain-of-custody procedures are typically much more labor-intensive but may be used at the applicant's option.

2.5 Reporting of Results

The results should be reported in a format which includes all information and data necessary to calculate final results, presented in as clear and succinct format as possible. This will include a description of the operating system; the operating conditions during the test; the measured quantities of POHCs, HCl, and particulate in all samples; and the calculated results. Example formats for presentation of these data are presented in the *Guidance on Setting Permit Conditions and Reporting Trial Burn Results*.

The results of the analyses for particulate emissions, HCl emissions and removal efficiency, and DRE should be reported separately for each run and should not be averaged for the trial runs. This does not preclude averaging multiple samples taken during each run.

- VOST analytical results should be reported as individual values for each trap as well as an average value for each run (as total ng/L of sample). The average amounts to dividing the total quantity (ng) on all traps by the total sample volume (L) for all traps. An illustrative example is provided in Table 3. In this example, in run 3, a sample was lost, but the total sampling period was still > 60 min, so the results are usable. Ordinarily, 120-min sampling is the goal. Note that samples were collected with four pairs of VOST traps so that the minimum recommendation of three pairs of traps per run is still met when data for one pair are lost in run 3.
- The permit reviewer should consult the *Guidance on Setting Permit Conditions and Reporting Trial Burn Results* for further guidance on how results should be reported and the *Practical Guide — Trial Burn for Hazardous Waste Incinerators*¹ and other references on how sampling and analytical data should be converted into final results. A portion of the Practical Guide is repeated below to provide practical suggestions

regarding blank correction, significant figures, rounding, and handling "less than" and "greater than" values.

Table 2. Example Calculation to Determine Whether VOST Sample Size Is Sufficient to Measure 99.99% DRE for Carbon Tetrachloride

Basis

Waste feed flow rate: 15.2 kg/min (2,000 lb/h)
POHC: Carbon tetrachloride
Waste feed concentration: 500 ppm (0.50 g/kg feed)
Stack gas flow rate: 4,500 scfm (127.4 m³/min)
Lower detection limit: 2 ng per trap

Proposed sampling

VOST: 3 trap pairs at 500 mL/min flow rate; 20 L sample/pair

1. *POHC input rate*
 $15.2 \text{ kg/min} \times 0.50 \text{ g/kg} = 7.6 \text{ g/min}$
2. *POHC stack output rate at 99.99% DRE*
 $7.6 \text{ g/min} (1 - 0.9999) = 0.00076 \text{ g/min}$
3. *POHC concentration in stack gas at 99.99% DRE*
 $\frac{0.00076 \text{ g/min}}{127.4 \text{ m}^3/\text{min}} = 0.0000060 \text{ g/m}^3 \times 10^9 \text{ ng/g} \times 10^{-3} \text{ m}^3/\text{L} = 6.0 \text{ ng/L}$
4. *Sample amount collected on one pair of traps*
 $20 \text{ L} \times 6.0 \text{ ng/L} = 120 \text{ ng}$

Since the VOST lower detection limit for carbon tetrachloride is 2 ng, the sample is sufficient to detect carbon tetrachloride to determine a DRE of 99.99% or lower. A margin of safety above the detection limit is desirable.

This calculation assumes both traps in a pair are combined for analysis. If they are analyzed separately, the distribution of mass on each trap must be considered.

2.5.1 Blank Correction

Because achievement of 99.99% DRE often results in stack concentrations that are at or below ambient or laboratory levels for POHCs, contamination of samples can be a significant problem. The purpose of blank correction procedures is to account for any portion of the sample results that represent contamination, or something other than the value intended to be measured (e.g., stack emissions).

The underlying philosophy of the procedure is based on a paper prepared by the American Chemical Society Committee on Environmental Improvement² and on experience in conducting and interpreting trial burn data. The ACS paper assumes that blank values are random samples that vary because of preparation, handling, and analysis activities. Under this assumption, blank values can be treated statistically. The "best estimate" for the blank for any particular sample is the mean of the available blanks. The ACS procedure also enables determination of whether a sample is "different from" the blank. If the sample value is not significantly different from the blank value, a sample cannot be blank-corrected. Even so, the measured sample value does provide an upper bound for the emission value and may still provide sufficient information for determining if the required DRE of 99.99% was met.

Table 3. Example Reporting of Volatile POHC Emissions

Run no.	Trap pair	Sample period (min)	Sample volume ^a (L)	POHC collected ^b (ng)		POHC concentration ^c (ng/L = $\mu\text{g}/\text{Nm}^3$)		Stack gas flow rate ^d (Nm^3/min)	Emission rate ^e ($\mu\text{g}/\text{min}$)	
				CCl_4	CCl_2CHCl	CCl_4	CCl_2CHCl		CCl_4	CCl_2CHCl
1	1	30	20.1	16	71 (60)					
	2	30	19.8	19	180 (170)					
	3	30	21.3	30 (24)	45 (34)					
	4	30	19.5	25 (19)	105 (94)					
	Total/average ^{e,f}	120	80.7	78	360	0.97	4.5	181	180	810
2	1	30	19.7	37 (31)	220 (210)					
	2	31	22.1	21 (15)	180 (170)					
	3	30	20.8	28 (22)	270 (260)					
	4	30	21.0	31 (25)	210 (200)					
	Total/average ^{e,f}	121	83.6	93	840	1.1	10.0	173	190	1700
3	1	30	20.4	14	56 (45)					
	2 ^g	—	—	—	—					
	3	29	20.2	8.6	37 (26)					
	4	30	19.9	12	48 (37)					
	Total/average ^{e,f}	89	60.5	35	108	0.58	1.8	190	110	340
Average blank value ^f (ng)				5.8	11					
Standard deviation ^f (ng)				6.9	2.7					

Note: Slow VOST results are shown; a similar format would be used for fast VOST or integrated bag sampling for volatiles.

^a Sample volume is dry standard liters of stack gas.

^b If blank corrected, that value shown in parentheses.

^c Blank corrected as applicable.

^d Stack gas flow rate is dry normal (standard) cubic meters per minute.

^e Totals for sample period, volume, and amount collected; averages for concentration, flow rate, and emission rate.

^f All blanks (both field blanks and trip blanks) were used for average and standard deviation.

^g Sample lost.

The blank correction procedure applies mainly to stack emission samples and consists of the following:

- a. Assemble data for each POHC from all of the field and trip blanks. An example of such data for VOST might be:

		Run 1	Run 2	Run 3
POHC A	Field blank	0.008 μg	<0.002 μg	0.004 μg
	Trip blank	0.005 μg	0.004 μg	0.003 μg

- b. Determine whether or not the field blanks are statistically different from the trip blanks by using the paired t-test (consult a statistics text).

If the field blanks are significantly higher than the trip blanks, use the field blank data only. If the blanks are not significantly different, use all of the blank values. Higher field blanks indicate background due to field exposure, which trip blanks do not measure.

- c. Calculate the average and standard deviation of the blanks (many calculators have statistics functions which allow you to do this easily).

- d. Determine whether or not each measured sample value is "different from" the blank value by using the following test for each sample:

s = sample value (μg)

b = (blank average) + 3 (standard deviation of blanks)

If s is greater than b , then the sample is "different" from the blanks.

- e. If the measured sample value is different from the blank value, then the blank correction procedure is applied:

Blank-corrected emission value (μg) =
measured sample value (μg) - average blank value (μg)

- f. If measured sample value is not different from blank value, then the measured sample value is used as an upper bound emission value, and the emission rate is considered less than or equal to the measured value. This results in the reporting of emission concentration and mass emission rate with a "<" sign. As a consequence, DRE would be reported with a ">" sign.

2.5.2 Significant Figures and DRE

DRE is usually reported with one or two significant figures depending on the accuracy of the measured values which go into the calculation of DRE. It is important to note that a reported DRE of 99.99% or 99.999% has only one significant figure. The reason for this is that what is actually being measured is the penetration, which is the amount of a compound which is not destroyed. That is:

DRE = 100% — Penetration

For a DRE of 99.99%, the penetration is 0.01% (one significant figure). For a DRE of 99.9916%, the penetration is 0.0084% (two significant figures).

The DRE is reported with the same number of significant figures as the least accurately measured value used in the calculations. The controlling measurement that determines the number of significant figures is usually the stack concentration. GC/MS methods can normally only report concentrations with one or two significant figures. This will result in a DRE with the same number of significant figures as reported concentrations, unless another measured value (waste feed concentration, waste feed flow rate, or stack gas flow rate) has fewer significant figures.

2.5.3 Rounding Off DRE Results

The rules on this are stated in the *Guidance Manual for Hazardous Waste Incineration Permits*:³ "... if the DRE was 99.988%, it could not be rounded off to 99.99%." In other words, your calculated value, after rounding to the proper number of significant figures, must equal or exceed 99.99% to be acceptable. (Note: This same rule applies to rounding HCl results to 99%.)

2.5.4 Reporting DRE with a "<" or ">" Sign

As mentioned in the section on blank corrections, if the sample is not "different" from the blank (greater than the average blank plus three standard deviations), then it cannot be blank-corrected. As a consequence, the DRE will be reported with a ">" sign. This reported ">" value will also occur when the POHC in the sample is undetected (below detection limit of the analysis method). But as long as the DRE is > 99.99%, this is not a problem.

In cases where both the blanks and samples have high values, a DRE below 99.99% may be preceded by a ">" sign (i.e., > 99.96%). Such a number is useless in evaluating achievement of 99.99%. Experience in using the recommended sampling methods and avoiding contamination is the only way to minimize this possibility.

Occasionally, a sample may saturate the GC/MS with the POHC in question. This will result in an emission rate with a ">" sign and a DRE with a "<" sign. If such a DRE is below 99.99%, the incinerator clearly fails. If it is above 99.99% (i.e., < 99.9964%), the number is useless. To avoid such problems, the test protocol should be chosen based on estimated concentrations. Several factors to consider include (1) collecting less sample on the VOST, (2) using a sample splitter during analysis so that only a portion of the sample is directed to the GC/MS, or (3) using alternative sampling methods (e.g., integrated bag). If these techniques are being considered, the Source Methods Standardization Branch, AREAL, EPA or OSW, EPA should be consulted.

The conclusion of this section is always design the sampling and analysis so that passage/failure of the 99.99% criterion is determinable. This can best be done by preliminary estimates of POHC concentrations in the stack (assuming 99.99% DRE) and with selection of sampling and analysis methods having appropriate upper and lower limits of detection. Experience in use of these methods to avoid contamination is also a key factor.

2.6 Continuing Analysis and Monitoring

After the trial burn test is completed and after the permit is obtained, analysis and monitoring must continue. Each type of waste feed material must be analyzed at least on an annual basis according to recent EPA policy. More frequent analysis is necessary if composition of the waste feed is highly variable or is expected to change (e.g., different source, process change, etc.).

The permit will establish the monitoring requirements for process parameters, air pollution control devices, and stack gas emissions. The exact parameters to be monitored are established on a case-by-case basis, as described in the *Guidance on Setting Permit Conditions and Reporting Trial Burn Results*. The permit monitoring requirements and continuing waste feed analyses are intended to verify continued satisfactory operation of the incinerator.

Section 3

Measurement Methods

Many methods can be used for process monitoring, sampling, and analysis of samples during a trial burn and subsequent operation of the incinerator. This review focuses on the most commonly used and recommended methods for measurement.

3.1 Specification of Method Options

The trial burn plan must present the sampling and analytical methods in sufficient detail for both review and field and laboratory implementation. Many of the referenced methods give several options which may be employed by the sampler or analyst. The trial burn plan should specify which options will be used. If selection of an option depends on the sampling conditions or sample characteristics and cannot be made at the time the plan is submitted, the decision criteria for subsequent selection of the options must be presented. Some referenced methods, especially those presented in the Arthur D. Little report, *Sampling and Analysis Methods for Hazardous Waste Combustion*,⁴ are general descriptions of the methods and may not provide sufficient detail to specify the methods. In such cases, the trial burn plan should give a detailed step-by-step procedure. Often the sampling crew and analytical laboratory will have sets of standard operating procedures (SOPs) for the common analyses encountered in hazardous waste incineration. These SOPs can be appended to the trial burn plan and cited in the text.

For many trial burns, sampling and analysis of different analytes will be performed by more than one laboratory. Few laboratories have the capabilities to do all of the analyses required. In these cases the applicant should specifically identify the laboratory who will be doing the work for each method and should specify the methods employed in the same level of detail for all laboratories. The trial burn plan may include this information or it should specify a time, sufficiently before the trial burn to allow EPA review, that it will be provided. The laboratory that conducts the sampling and analysis may not have been selected when the trial burn plan is submitted.

3.2 Process Monitoring

Two important parameters which must be continuously monitored during operation of the incinerator are the

waste feed rate and the combustion temperature. Another important parameter is the combustion chamber pressure, which is often used as a method of monitoring fugitive emissions. Methods used to measure these three parameters are discussed below. CO monitoring will be covered in Volume VI of the Guidance Series (see Section 1.0) being prepared by EPA. Other parameters, e.g., combustion gas velocity, are not covered at this time. The EPA, Office of Solid Waste, may be consulted for additional guidance.

3.2.1 Waste Feed Rate

The waste feed rate to an incinerator can be monitored in a variety of ways, depending upon the types of feeds encountered. The feeds may be solids or sludges, free-flowing liquids, or gases.

3.2.1.1 Solid-Sludge Feeds

Volumetric methods — These include calibrated augers and pumps, rotary feeders, and belt conveyors. These systems are not generally available precalibrated but must be calibrated by the user for each particular feed material. The accuracy of the method depends upon steady operation at a given speed and assumes appropriate feeders are used to ensure the cavities are always filled to capacity. Most of these methods can provide some kind of tachometer signal to indicate speed, which must be related to feed rate by performing calibration tests. These methods are generally more appropriate as secondary indicators of feed rate.

Level indicators — These include methods based upon mechanical, ultrasonic, nuclear, and radio frequency principles of operation. Nearly all tank level indicators will perform better with somewhat uniform (free-flowing) particles. This will aid in distributing the level of material evenly within the vessel, allowing for more accuracy in whatever monitoring system is used. Typically, these methods can monitor tank levels to within $\pm 1\%$.

Since level indicators cannot provide physical characteristics for the feed material (i.e., density, moisture), care must be taken when using any of these systems to account for cross-sectional area of the tank and changes in composition of the feed.

Stationary weight indicators — These methods, which include weigh hoppers/bins and platform scales, determine the dead weight of material loaded into a hopper, bin, or container. After weighing, the contents are then fed as batches into the process. All of these weigh systems give fairly accurate monitoring of weight (within $\pm 1\%$), but one must consider the batch feeding system operations before a true appraisal of the feed rate monitoring can be made.

Conveyor weighing systems — These methods include belt weighers, weigh belts/augers, and loss-in-weight feeders. All conveyor weighing systems are fairly similar in operation, mainly differing because of placement locations of the weighing device. In general, the accuracy of these systems is around $\pm 2\%$ but tends to decrease as particles become larger and less uniform in size. Sludges can be monitored with the systems, provided that wet material does not drain off the conveyor belt. Screw augers, however, can often be used in such cases to replace the conventional conveyor belt.

Momentum flowmeters — Two types of these solid flowmeters are available, based upon either impact or torque. These devices work fairly well with dry, flowable materials but are less accurate if feed particles are very large, nonuniform, or viscous. Typical accuracies are within $\pm 2\%$. Sludges are not recommended because of their viscosity and splashing effects.

Nuclear absorption — Methods based upon absorption of gamma radiation include nuclear level meters, nuclear belt or auger scales, and a combination of nuclear density meters and ultrasonic flowmeters. Nuclear absorption only measures density; therefore, another instrument must also be used to measure volume, speed, or another parameter to obtain feed rate. Nuclear instruments can be used on nearly any material including sludges. Radiation absorption is proportional to the mass present, so particle size and configuration will not greatly hinder accuracy. Sludge operations will work best with a nuclear density detector/ultrasonic flowmeter combination, enabling the process material to be fed through conventional piping. Accuracies of nuclear devices may not be as high as gravimetric systems but may be sufficient on a practical basis.

3.2.1.2 Liquid Feeds

Typical flowmeters used to monitor the liquid waste feed rate to incinerators are detailed below.

Rotameter — This type of flowmeter is available for a wide range of liquid viscosities including some light-weight slurries. It is calibrated through using a fluid of known density. Reported accuracies are within $\pm 5\%$ of full-scale.

Orifice meter — This instrument is used with gases and low viscosity fluids. Typical accuracies are $\pm 1\%$ full-scale, which is the accuracy of the differential

pressure measuring device used on a clean fluid. When used with dirty or viscous fluids, both accuracy and life of the instrument are sacrificed. An accuracy of $\pm 5\%$ may be more realistic in these cases.

Vortex shedding meter — This device is applicable to low-viscosity fluids and gases under turbulent flow conditions. The accuracy is $\pm 2\%$ under normal operations.

Positive displacement meter — This type of flowmeter is more applicable than other types for use with higher viscosity fluids. However, accuracy is highest when used with a clean, moderately viscous fluid. It cannot be used with multiphase liquids, gases, or slurries of varying density.

Mass flowmeter — This instrument, also known as a Coriolis flowmeter, applies to liquids of widely varying viscosity and density and most slurries. It has been advertised for use with gases, but that application may be rare. The reported accuracy is within $\pm 1\%$.

3.2.1.3 Gaseous Feeds

The best types of flowmeters for gases are the orifice meter and the Vortex shedding meter, discussed above under liquid feeds.

3.2.2 Combustion Temperature

Combustion temperature is usually monitored through the use of thermocouples, optical pyrometers, or both.

3.2.2.1 Thermocouples

Thermocouples are available in a variety of types, with each type constructed of specific metals or alloys. The temperature ranges and reported accuracy vary by type. The environment the thermocouple is suited for also varies. A summary of thermocouple types and limitations is given below.

Type	Materials	Upper Temp. (°F)	Thermocouple Accuracy (\pm %)	Environment
J	Iron/constantan	1400	0.75	Reducing, vacuum, or inert
E	Chromel/constantan	1650	0.50	Oxidizing or inert
K	Chromel/Alumel	2300	0.75	Oxidizing or inert
S	Pt 10% rhodium/pure Pt	2650	0.25	Oxidizing or inert (no metal tubes)
R	Pt 13% rhodium/pure Pt	2650	0.25	Oxidizing or inert (no metal tubes)
B	Pt 30% rhodium/pure Pt 6% rhodium	3100	0.50	Oxidizing or inert (no metal tubes)

Source: *Complete Temperature Measurement Handbook and Encyclopedia*, Omega Engineering Inc., 1986.⁵

The accuracies given above do not consider environmental effects. Thermocouple location in the combustion chamber, for example, greatly affects the accuracy of temperature readings. Typically, thermocouples are located at the gas exit from the combustion chamber. This is generally believed to give the best overall average

combustion chamber temperature. Other factors to consider when examining thermocouple locations are:

- Temperature readings will be affected by radiant pickup or loss if the thermocouple is located close to and within a direct line of sight of either the flame or the cold quench chamber.
- To improve accuracy and limit wall effects, the thermowell should extend 3 to 6 in. beyond the refractory and should be located where the gas velocity is high and not in a stagnant corner of the chamber.

The use of two thermocouples in separate wells is recommended to provide a check on continued proper operation. The difference in the readings between the two thermocouples should be noted during initial operation; e.g., one thermocouple will likely give a reading higher than the other one. The difference between the two readings should then be checked periodically as an indicator of problems with one of the thermocouples. If the difference changes by more than 50°F, both thermocouples should be checked for proper operation.

Following the above guidelines allows for some degree of consistency in measuring combustion temperature in an incinerator. Changes in either the thermocouple type or location should prompt reconsideration of the accuracy and representativeness of the measurement.

3.2.2.2 Optical Pyrometers

Optical pyrometers are typically used to measure the temperature of the furnace wall or an object within the furnace but can be used to measure the combustion gas temperature. In cases where the gas temperature is desired, the pyrometer is normally equipped with a closed end tube much like a thermocouple well but larger, and the pyrometer is sighted on the end of this tube. In this situation, emissivity corrections are not needed. This configuration is normally used for high temperatures when contamination or breakage of thermocouples is a problem and the cost or difficulty of replacement is high. The pyrometer will normally require calibration but should, when calibrated, be approximately as accurate as a thermocouple.

3.2.3 Combustion Chamber Pressure

Monitoring of the combustion chamber pressure is often used to ensure correct operation of the incinerator and to prevent fugitive emissions. Many combustion chambers are operated under draft (less than atmospheric pressure) conditions, which ensures that combustion off gases do not exit the chamber before passing on to the scrubber or other air pollution control equipment.

Instruments used to monitor combustion chamber pressure are known as differential pressure gauges, ΔP transducers, or draft gauges. Such instruments are composed of a bellows or diaphragm enclosed within a stainless steel casing. Deflection of the diaphragm due to

combustion chamber pressure is then measured by magnetic pickup coils also mounted within the casing. The instruments are located on the combustion chamber and typically measure pressures on the order of several inches of water.

Accuracy of a differential pressure gauge is within $\pm 1\%$ full-scale, although the combustion chamber environment severely hinders this performance. Particulate clogging, moisture, corrosion, and other contaminants are typical problems. A precise measure of combustion chamber pressure is difficult, but these instruments can be used to indicate whether the pressure within the combustion chamber is positive or negative. Calibration against an inclined manometer at least annually is recommended.

3.3 Sample Collection

Sampling methods for the waste feed, stack gases, auxiliary fuel, quench and scrubber water, and ash are given primarily in four sources: (1) *Test Methods for Evaluating Solid Waste — Physical/Chemical Methods*, SW-846⁶; (2) *Sampling and Analysis Methods for Hazardous Waste Combustion*⁷; (3) *Code of Federal Regulations*, 40 CFR Part 60, Appendix A⁷; and (4) The American Society for Testing and Materials, *Annual Book of ASTM Standards*.⁸ EPA-600/8-84-002 should be consulted first as it provides cross-references to more fully documented sources of methods (i.e., SW-846 or ASTM). These other sources should then be consulted. If there is a conflict between these documents, SW-846 has precedence. The discussion which follows addresses the most commonly used methods available for sampling these parameters.

3.3.1 Waste Feed

The objective of waste feed sampling is to obtain a representative sample of the waste feed. It is important that the sampling plan be explicit and that it explain the logic behind the selection of the sample collection scheme. For many waste feeds, representative sampling is difficult.

The waste feed for incinerators may be a free-flowing liquid, slurry, sludge, or a powdered, granular, or large-grained solid. Various methods are available for sampling these waste feeds and are described in SW-846,⁶ EPA-600/8-84-002⁴ and the ASTM Standards.⁸ These methods are discussed in further detail in the following subsections.

3.3.1.1 Free-Flowing Liquids

The most common method used for sampling free-flowing, low-viscosity liquids is tap sampling from the waste feed line or tank. Other methods that may be used for sampling from drums, tanks, or ponds include a coliwasa (composite liquid waste sampler), weighted bottle, or dipper. Each of these methods and types of equipment used are described in the following sources:

Sampling Method	SW-846 Chapter 9	EPA-600/ 8-84-002 Method	Old ASTM Standards	New ASTM Standards
Tap sampling	—	S004	D-270-75	D-4057-9.3
Collwasa	p. 49-51	S001	—	D-4057-9.6
Dipper	p. 50,53	S002	—	D-4057-9.5
Weighted bottle	p. 50,52	S003	D-270-75	D-4057-9.7
Thief	p. 50,54	S005	E-300-73	

Sampling a free-flowing liquid waste feed can be performed by obtaining tap samples from the storage tank or process feed line(s). The liquid in a storage tank should be thoroughly agitated and mixed to obtain representative samples. If the feed contains two immiscible liquid phases or solids which could stratify in the feed line, a mixer may need to be inserted in the feed line prior to the tap to obtain representative samples.

3.3.1.2 Viscous Liquids, Slurries, Sludges, and Solid Waste Samples

Sampling methods and the equipment required for viscous liquids, slurries, sludges, and solid waste feeds are described in SW-846, EPA-600/8-84-002, and the ASTM Standards. These methods are described in SW-846 and EPA-600/8-84-002 as follows:

Method Name	Type of Waste	SW-846 Chapter 9	ADL Method
Thief (grain sampler)	Dry powder or granules	p. 50,54	S005
Trier (corer)	Sludge or moist solids	p. 50,55	S006
Trowel (scoop)	Moist or dry solids	p. 50	S007
Auger	Packed solids	p. 50	—

The sampling protocols given in the ASTM Standards for the different types of waste feeds are:

Extremely viscous liquids	ASTM Standard D140-70
Crushed or powdered materials	ASTM Standard D346-75
Soil or rock-like material	ASTM Standard D420-69
Soil-like material	ASTM Standard D1452-65
Fly ash-like material	ASTM Standard D2234-76

Samples of extremely viscous liquids, slurries, and sludges are usually obtained from their containers by the methods described above. Samples of solid waste are usually obtained from the containers, waste piles, or from the process feed system, such as a conveyor belt or auger system.

3.3.2 Auxiliary Fuel

Sampling of liquid auxiliary fuel may be appropriate so that the samples may be analyzed for the POHCs and higher heating value (for total heat input), although this measurement is not required by RCRA. The methods for sampling the fuel are the same as those used for free-flowing liquid wastes. Tap sampling (S004) is the most common method used for liquid fuels.

3.3.3 Stack Gases

Stack gases must be sampled for the following parameters during the trial burn; particulates, hydrogen chloride

(HCl), H₂O, CO₂, CO, and principal organic hazardous constituent (POHC) emissions. Stack gas flow rate, volume, and temperature must be measured during the sample collection. Analyses of these samples are required by 40 CFR Part 264 (Subpart O) and 40 CFR Parts 270.19 and 270.62.⁷ Table 4 summarizes the sampling methods used for stack gases for RCRA trial burns. Many of these parameters can be sampled using the standard EPA reference methods shown in Table 5. Methods for both volatile and nonvolatile metals are included in Table 4 for completeness, since sampling may be requested. Additional information on methods for CO, oxygen, total hydrocarbon (THC), HCl, and metals, including draft performance specifications for CO and oxygen, can be found in Volume VI of the Guidance Series (see Section 1).

An important aspect of obtaining a valid stack gas sample is the experience and performance of the sampling personnel. The sampling personnel, especially the supervisor, must be adequately trained, as evidenced by either previous experience or documented training.

3.3.3.1 HCl

Hydrochloric acid (HCl) emissions are currently measured using a CEM or manual methods consisting of a collection train and/or analytical procedure. Manual collection methods normally extract a gas sample from the stack and absorb the HCl in an absorbent. The sampling train consists of a particulate filter, the absorption solution, and a provision for measuring the sampled gas volume. The EPA Method 5 train, an EPA Method 5 train modified for metals, or a specific impinger train have all been employed. The M5 trains are normally used when HCl is to be collected in addition to sampling for particulates or metals. If sampling is to be performed for only HCl, then a specific impinger train is employed. For sampling HCl using the M5 as described in the *Federal Register*, the M5 procedures are followed except that the water in the impingers is replaced with an absorbing solution. A draft method for HCl sampling, Determination of HCl Emissions from Municipal and Hazardous Waste Incinerators, is being prepared by EPA, AREAL, Source Methods Standardization Branch. This method is undergoing further revision, e.g., a better filter arrangement than shown in the current draft will be recommended.

A solution of sodium or potassium hydroxide has frequently been used to absorb the HCl. This approach has been satisfactory except that the solution also absorbs the other acid gases contained in the sample including carbon dioxide (CO₂). The use of the hydroxide solution therefore may require that a correction be applied to the sample volume for the gases that were removed before the gas volume meter in the sampling stream. This correction may be avoided by substituting other solutions for

Table 4. Sampling Methods for Stack Gases in RCRA Trial Burns

Sampling parameter	Sampling method
Particulates, Cl ⁻ , H ₂ O, nonvolatile metals (if present in feed)	M5 train with appropriate solutions in one or more of the impingers
Semivolatile and nonvolatile POHCs	Semi-VOST (SW-846 Method 0010)
Volatile POHCs	VOST (SW-846 Method 0030)
CO ₂ , O ₂	EPA Method 3 Orsat analysis of integrated bag sample
CO	Continuous monitor, proposed performance specifications are being prepared by EPA ⁹
Volatile metals (if present in feed)	M5 train with appropriate solutions in the second impinger

NOTE: POHCs = Principal organic hazardous constituents.
VOST = Volatile organics sampling train.

Table 5. EPA Reference Methods Used to Test RCRA Hazardous Waste Incinerators

Method description	
EPA Method 1:	Sample and velocity traverses for stationary sources
EPA Method 2:	Determination of stack gas velocity and volumetric flow rate (type S pitot tube)
EPA Method 2A:	Direct measurement of gas volume through pipes and small ducts
EPA Method 3:	Gas analysis for carbon dioxide (CO ₂), oxygen (O ₂), excess air, and dry molecular weight (Orsat)
EPA Method 4:	Determination of moisture content in stack gases
EPA Method 5:	Determination of particulate emissions from stationary sources

NOTE: All of these EPA methods are fully described in 40 CFR Part 60, Appendix A, revised as of July 1, 1985.⁷

the hydroxide. There is a lack of consensus on the impinger reagent most appropriate for collection of HCl. A sodium carbonate solution has been recommended, since this reagent will not absorb CO₂. There is evidence to suggest that caustic reagent is not necessary and that HCl is efficiently trapped in any aqueous medium. If this is true, then distilled water (e.g., ASTM Type II reagent water) may be the reagent of choice for collection of HCl. The draft method being prepared by EPA recommends dilute acid solutions.

3.3.3.2 Volatile Organics

The methods used to sample the POHCs and other organics depend upon their volatility. Volatile POHCs (generally those with a boiling point between 30° and 100°C, see Section 3.4.5.4.1) are sampled using a VOST. The VOST and its operation are fully described in the *Protocol for the Collection and Analysis of Volatile POHCs Using VOST*,⁹ the two-volume study *Validation of the Volatile Organic Sampling Train (VOST) Protocol*,¹⁰ the EPA-600/8-84-002⁴ as Method S012, and Part III, Chapter 10 of SW-846 as Method 0030.

Several variations on the basic VOST design are available from vendors or may be constructed by the sampling organization. The sampling plan should show a schematic of the train actually used and also describe the exact apparatus and operation. Critical aspects include

the train setup, probe position in the actual stack geometry to be tested, leak check procedures, and sample handling. The latter is particularly important, since the volatile organics in ambient air as well as other sources of contamination can affect the accuracy of results. Therefore, care must be taken to prevent contamination of the sorbent cartridges before and after the collection of the stack gas sample.

In planning the VOST sample collection, the sample volume required to demonstrate the 99.99% DRE should be calculated as described in Section 2.4 and Table 2. The volume of the VOST collection can then be scaled accordingly. If numeric values for POHCs at concentrations much higher than the value needed for the 99.99% DRE must be reported, a second VOST collection with a smaller sample volume may be required to prevent saturation of the GC/MS system.

As noted in the documents describing VOST, a variety of methods are available for generating standards for calibration of the GC/MS. The VOST protocol¹⁰ specifies the injection of a methanolic standard solution onto the cartridge followed by flash evaporation of the methanol. This is workable but presents several practical problems. Very volatile compounds can evaporate from the methanolic solution. The residual methanol prevents scanning the mass spectrometer below about mass 34 (M + 2 of meth-

anol). There is also a possibility that some POHCs may react with the methanol. Other alternatives listed in the validation documents¹⁰ are gas cylinder standards, permeation devices, and static gas bottle standards.

Gas cylinder standards are prepared from outside sources and certified by the supplier. These cylinders can be purchased to contain various POHCs at various gas concentrations.

Permeation tubes and other devices can generate a reliable, accurate gas mixture. They are commercially available or can be prepared for most POHCs. However, setting up, maintaining, and monitoring a permeation system is expensive and time-consuming. In addition, the concentrations of the POHCs in the gas stream are governed by their permeation rate, so the flexibility to generate different concentrations is limited.

Static gas bottle standards are generated by evaporating a known amount of the POHCs in a gas bottle to give a known gaseous concentration. This is a simple standard preparation method; however, accuracy can be a problem if evaporation is incomplete or the POHC adsorbs to the wall of the vessel.

The EPA also operates a program to develop organic gas audit materials and provide these audit gas cylinders for use in VOST performance audits during trial burns. Four different audit cylinders are available that contain the volatile organic compounds shown in Table 6. Any federal, state, or local agency or its contractor planning hazardous waste trial burn tests may request a performance audit by contacting Mr. Robert Lampe, USEPA, Environmental Monitoring Systems Laboratory, Quality Assurance Division, Research Triangle Park, North Carolina 27711.

Collection of stack gas samples in Tedlar bags is listed as an option for sampling volatile organics in Method S011

in EPA-600/8-84-002; however, several problems with the analysis of bag samples severely limit the utility of this option. Samples are collected in 30-L Tedlar gas bags using an integrated gas sampling train. The bags are then transported to the analytical laboratory, and if the VOST traps are saturated, a small volume of gas from the bag is transferred to a clean VOST trap for analysis. This approach presents some practical problems. Tedlar bag samples have a relatively short holding time of 1 to 2 days for some specified compounds according to EPA Method 23.¹² Therefore, the use of Tedlar bags is practically limited to tests where the VOST analysis is being conducted on-site or within a very short holding time. The bag sample procedure is less desirable than VOST due to potential problems with adsorption in the bag and loss of sample. Stability of the POHC to be sampled in the bag should be checked prior to sampling, if this method is used. Field blanks are essential with bag sampling.

3.3.3.3 Semivolatile Organics

Semivolatile POHCs and other organics with boiling points above 100°C are sampled with a semi-VOST (sometimes referred to as a Modified Method 5 [MM5] train). Those organic compounds with boiling points between 100° and 140°C may also be suitable for sampling with VOST. However, experts in analytical chemistry should be consulted for sampling of compounds in this boiling point range.

The semi-VOST is an EPA Method 5 train which has been modified by placing a sorbent module (usually containing XAD-2 resin) before the first impinger. This train is identified in Part III, Chapter 10 of SW-846 as Method 0010,⁶ and in EPA-600/8-84-002 as Method S008.⁴ The sampling train is fully described in *Modified Method 5 and Source Assessment Sampling System Operations Manual*,¹³ and in the two-volume report, *Laboratory and Field Evaluation of the Semi-VOST (semivolatile organic sampling train) Method*.¹⁴ Sampling trains with XAD resin traps should not be used to collect HCl.

Table 6. VOST Audit Compounds*

Group I compounds	Group II compounds	Group III compounds	Group IV compounds
Carbon tetrachloride Chloroform Perchloroethylene Vinyl chloride Benzene	Trichloroethylene 1,2-Dichloroethane 1,2-Dibromoethane Acetonitrile Trichlorofluoromethane (F-11) Dichlorodifluoromethane (F-12) Bromomethane Methyl ethyl ketone 1,1,1-Trichloroethane	Vinylidene chloride 1,1,2-Trichloro-1,2,2-trifluoroethane (F-113) 1,2-Dichloro-1,1,2,2-tetrafluoroethane (F-114) Acetone 1-4 Dioxane Chlorobenzene	Acrylonitrile 1,3-Butadiene Ethylene oxide Methylene chloride Propylene oxide Ortho-xylene
Group I ranges	Group II ranges	Group III ranges	Group IV ranges
7 to 90 ppb 90 to 430 ppb 430 to 10,000 ppb	7 to 90 ppb 90 to 430 ppb	7 to 90 ppb 90 to 430 ppb	7 to 90 ppb 430 to 10,000 ppb

Source: "Performance Audit Results for Volatile POHC Measurements," JAPCA, Vol. 38, No. 6, June 1988.¹¹

* All gas standards are in a balance gas of nitrogen.

Each stack-testing organization uses a slightly different variation on the basic semi-VOST. The sampling plan should show a schematic of the train actually to be used and briefly describe the semivolatile collection apparatus, as well as cite the protocol to be followed. Critical aspects include the train setup and position of the XAD cartridge. Contamination from grease in the glassware fittings in the train has caused problems with the subsequent analysis. Therefore, most guidance (see, for example, Reference 4) states that no grease (either hydrocarbon or silicone) is to be used in the train. This lack of grease may cause problems with demonstrating a satisfactory leak check but ensures against gross sample contamination which can render the samples unanalyzable.

In recovering the sample from the sampling train and subsequent analysis, it must be emphasized that *all* interior portions of the train prior to and including impingers that contain condensate after the XAD-2 sorbent module must be considered as part of the semivolatile organics sample. This includes nozzle and probe rinses, impinger contents, the filter, the XAD-2, and all glassware rinses. In addition, it should be noted that the filter from this sample collection should not be used for the particulate measurement. Upon heating to constant weight to measure the particulate, the filter may lose some or all of the semivolatile organic analytes. The contents of the impingers after the XAD-2 sorbent module should be analyzed for all POHCs, but is particularly important for very water-soluble compounds.

3.3.3.4 Metals

When specific metals can be identified as potential emissions, sampling will probably be best addressed by selecting a specific sampling and analytical approach. Specific methods which have been developed include EPA Method 12 for lead, Method 101A for mercury, Methods 103 and 104 for beryllium, and Method 108 for arsenic. In most cases, however, sampling will be required for multiple metals. For this, the draft metals protocol *Methodology for the Determination of Trace Metal Emissions in Exhaust Gases From Stationary Source Combustion Processes* (prepared by the EPA, AREAL, Source Methods Standardization Branch) describes the only system that has been proposed to collect both the volatile and non-volatile fraction of the stack gases. This draft protocol will be incorporated into a methods document under preparation by EPA, OSW as background for proposed amendments to the RCRA incinerator regulations.⁹ Sampling for hexavalent chromium presents several problems. These problems are the stability of the sample and recovery efficiencies when separating low level samples. Both oxidizing and reducing materials may affect the stability of the samples and produce errors in the determination. EPA is currently working on a suitable procedure to collect chromium(VI) stack samples. A separate sampling train used only for collection of chromium(VI) is recommended.

3.3.4 Quench and Scrubber Waters

Samples of quench and scrubber waters (both input and output) are usually taken by the tap sampling method (S004) or by dipper (S002). Weighted bottles (S003) can also be used. The corresponding ASTM and SW-846 methods are listed in Section 3.3.1.

3.3.5 Incinerator Ash (Residue)

The method most commonly used for sampling ash is the trowel method (S007). The protocols given in the ASTM Standards or SW-846 methods manual for sampling solid materials given previously in Section 3.3.1 provide additional information.

3.3.6 Documentation

The trial burn plan should address documentation of all sample collection activities. Sample collection times and conditions must be recorded when the samples are collected. For the VOST and semi-VOST, defined periodic readings of temperature and flow parameters must be recorded. Generally, example data recording forms should be provided in the trial burn plan. Often computers or programmable calculators are used for calculation of the sampling parameters. These should be addressed in the plan with sufficient information to document how calculations were made.

3.4 Chemical Analysis

The recommended methods for analyzing samples taken during the trial burn or routine operation of an RCRA hazardous waste incinerator are found primarily in SW-846,⁶ EPA-600/8-84-002,⁴ 40 CFR Part 60, Appendix A,⁷ and in the ASTM Standards.⁸ EPA-600/8-84-002 should be consulted first as it provides cross-references to more fully documented sources of methods (i.e., SW-846 or ASTM). These other sources should then be consulted. If there is a conflict between these documents, SW-846 has precedence.

The following sections discuss sample shipping, receipt, and documentation; planning the analysis; general analytical considerations; analysis of waste feed; analysis of stack samples; and analysis of other effluent streams.

3.4.1 Sample Shipping, Receipt, Documentation, and Storage

The trial burn plan must contain provisions for orderly and well-documented transfer of the samples from the incinerator to the laboratory including documentation of how samples were handled.

The trial burn plan should address:

- a. Sample storage requirements on-site and during storage at the laboratory prior to analysis.
- b. Holding times prior to analysis.

c. Sample labeling.

d. Documentation procedures for handling and transfer of samples.

Sample storage conditions and times should be specified. Holding times and storage conditions prior to extraction can be particularly important depending on the type of sample. Between analysis steps, appropriate storage conditions must also apply. The holding time and archiving conditions for samples and extracts after the analysis also should be specified. If possible, they should be held long enough to permit reanalysis if requested.

The sample labeling procedures and logging (notebook, forms, barcode) of the samples should be addressed.

Documentation of sample shipping, receipt, and transfer must be addressed in the plan. Generally, traceability forms accompany the samples and are signed and dated at each event. If formal chain-of-custody is to be used, additional documentation certifying the unbroken chain and security of the samples is required.

3.4.2 Planning the Analysis

The analysis must be carefully planned in order to prevent costly mistakes or schedule slippage. The trial burn plan should address how samples will be transferred to the appropriate laboratory personnel and how the personnel will be instructed on the analytical objectives. Written instructions to the analysts should address the following: sample numbers, storage location, the analytical methods to be used, the analysis objective and limit of detection required if not implicit in the method, analysis sequence, QC requirements (duplicates, spikes, blanks, etc.), other special instructions, and due date. The plan should indicate the presence of or have attached SOPs.

The first step in planning the analysis is to identify the target analytes. The process for selection of the organic hazardous constituents to be determined in the waste feed is described in Section III.B of the EPA-600/8-84-002⁴ and in Part III, Chapter 13 of SW-846.⁵ The general characteristics of the waste feed are determined first by proximate analysis, then survey analysis, and finally by directed analysis. Based on these results, a limited number of organics are selected for recommendation as principal organic hazardous constituents (POHCs). This selection procedure is not discussed in detail in this report.

One important consideration in selecting the POHCs is to avoid those which are difficult to analyze. Table 7 shows a select list of "problem" POHCs, the cause of the problem, and some possible solutions to the problem.

3.4.3 General Analytical Considerations

3.4.3.1 Glassware Cleaning and Tracking

All glassware used for the analyses must be properly cleaned. Each laboratory must develop adequate standard operating procedures for glassware cleaning. Separate cleaning procedures are generally required for organic and inorganic analysis, since different interferences are of concern.

It is highly advisable to have a glassware tracking system in place to prevent cross-contamination of glassware. The analyses described in this document address both high level (e.g., feed samples at 30% POHC concentration) and trace analysis (e.g., the same POHC in the stack gas sample at the nanogram-per-gram level). Thus, the potential for cross-contamination is great.

A glassware screening system is also advisable to verify the adequacy of the glassware cleaning.

3.4.3.2 Standards, Reagents, and Solvents

Standards, reagents, and solvents must be of the appropriate purity. New lots should be checked for purity. New standards should also be checked for chemical identity and their concentration verified.

3.4.3.3 Physical Sample Preparation

Some samples may require homogenization, blending, aliquoting, or compositing. Basic guidance is given in Methods P001, P002, and P003 of the EPA-600/8-84-002⁴ and in Part III, Chapter 9 of SW-846.⁵ Specific procedures should be detailed in the trial burn plan.

3.4.4 Analysis of Waste Feed

The waste feed must be sampled and analyzed in accordance with 40 CFR Parts 270.19, 270.62, and 264.13.⁷ Specifically, the waste feed must be analyzed for higher heating value, viscosity (if liquid), and the hazardous organic constituents listed in 40 CFR Part 261, Appendix VIII.⁷ Analysis is required for any of the ~ 300 constituents in Appendix VIII, which may be reasonably expected to be present for waste characterization or for selected constituents from the list for trial burns. Many other parameters may be determined to fully characterize the waste. Various methods and techniques are available to prepare the field samples for analysis. Table 8 lists the sample preparation methods described in the EPA-600/8-84-002. Table 9 lists the sample preparation and introduction techniques described in SW-846.⁸ Table 10 shows the recommended analytical methods that may be used to characterize the waste feed.

Physical analyses give information on the physical characteristics and general chemical composition of the waste including the following:

Table 7. Selected Problem POHCs

Compound	Cause of problem	Possible solution
Acetonitrile	Water soluble	
Acetyl chloride	Decomposition	
Aflatoxins	High toxicity	
Aniline	Water soluble	
Benzeneearsonic acid	Low volatility	Derivatize with HI
Benidine	Decomposition?	
Bis(chloromethyl)ether	Decomposition	
2-Butanone peroxide	Reactive	
2-sec-Butyl-4,6-dinitrophenyl	Acidic, extracts poorly	
Carbon oxyfluoride	Decomposition?	
Chloral	Water soluble	
Coal tars	Complex mixture	
Creosote	Complex mixture	
Cyanogen	Gas	
Cyanogen bromide	Gas	
Cyanogen chloride	Gas	
Cycasin	Low volatility	
N,N-Diethylhydrazine	Unstable	
1,1-Dimethylhydrazine	Unstable	
1,2-Dimethylhydrazine	Unstable	
1,4-Dioxane	Water soluble	Sample with Semi-VOST train
1,2-Diphenylhydrazine	Unstable	
Diphenylamine	Basic, extracts poorly	
Formaldehyde	Water soluble	Derivatize to sample
Formic acid	Water soluble	
Hydrazine	Unstable	
Hydroxydimethylarsine oxide	Low volatility	Derivatize with HI
Iron dextran	High molecular weight	GPC
Maleic anhydride	Unstable	
Maleic hydrazide	Unstable	
Mustard gas	Highly toxic	
Nitroglycerin	Explosive	
Phenylmercury acetate	Low volatility	Derivatize with HI
Phosgene	Highly toxic	
Pyridine	Water soluble	
Selenourea	Low volatility	Special HPLC column
Toluene diisocyanate	Water soluble	

- Heating value
- Viscosity (if liquid)
- Ash content
- Total organic chlorine
- Moisture content
- Solid content
- Elemental composition (optional)

The heating value and viscosity (for liquids) are required by RCRA. The ash content is generally required as an indicator of inorganic loading and other factors which may affect the amount of particulate generated. Total organic chloride is needed to determine the HCl removal efficiency. The other analyses are not required but may be of value to the incinerator operator in characterizing the waste feed and in operating the incinerator.

A method for analysis of total organic chloride in waste is under development by EPA, AREAL. In certain situations measurement of total chloride in the waste may be substituted for total organic chloride. The Source Methods Standardization Branch, AREAL or OSW, should be consulted on this topic.

3.4.4.1 Analysis of Waste Feed for POHCs

A major objective of the trial burn is to measure the DRE of selected POHCs. To do this, the sampling and analysis program must measure both the input and output rates of the POHCs. Only those organics selected for measurement (i.e., the POHCs) need to be measured in the waste feed during a trial burn.

The RCRA hazardous compounds are given in 40 CFR Part 261, Appendix VIII.⁷ The methods for analyzing various matrices for the POHCs are given in EPA-600/8-84-002⁴ as Methods A101 to A190 and in SW-846⁶ as Methods 8010 and 8310. Table 11 lists the methods given in EPA-600/8-84-002, and Table 12 lists the methods given in SW-846.

Specific methods for the Appendix VIII compounds (including inorganic compounds) given in EPA-600/8-84-002 and SW-846 are shown in the appendix.

3.4.4.2 Analysis for Inorganics

Inorganic analysis is not addressed in detail in this manual. The applicable analytical methods are cited for completeness in Table 13. For further information, the reader is referred to EPA-600/8-84-002⁴ and SW-846.⁶

3.4.5 Analysis of Stack Samples

Stack samples are analyzed for POHCs to determine DRE, hydrogen chloride, and particulate as required in 40 CFR 264.345.⁷ In addition, other analyses may be required for specific demonstrations. The recommended methods for analysis of stack samples are listed in Table 14. This section describes the methods generally used and discusses some potential problems and solutions.

3.4.5.1 Hydrogen Chloride Analysis

The hydrogen chloride in the stack gas must be measured to demonstrate an emission rate of less than 4 lb/hr or 99% removal before discharge to the atmosphere (40 CFR 264.345). (It may be possible to use data from analysis of chlorine in the waste to determine compliance with an uncontrolled HCl emission rate or to determine control device inlet HCl rates for calculation of efficiency.) A number of analytical methods have been employed to analyze the impinger catch for HCl. These methods include the automated ferricyanide colorimetric Methods 9250 and 9251 and the titrimetric Method 9252 from SW-846, ion chromatography ASTM Method D-4327-84 and Method 300.0 from Chemical Analysis of Water and Wastes. Ion chromatography is the preferred method. Because of the greater detail provided, the ASTM

Table 8. Sample Preparation Methods Given in EPA-600/8-84-002

Sample preparation method	Method number
Representative aliquots (composites) of field samples	
Liquids (aqueous and organic)	P001
Sludges	P002
Solids	P003
Surrogate addition to sample aliquots for organic analysis	
Volatile organics	P011
Basic extractable organics	P012
Acidic extractable organics	P013
Neutral extractable organics	P014
Extraction of organic compounds	
Aqueous liquids	P021
Semivolatiles	P021a
Volatiles	P021b
Sludges (including gels and slurries)	P022
Semivolatiles	P022a
Volatiles	P022b
Organic liquids	P023
Solids	P024
Semivolatiles by homogenization	P024a
Semivolatiles by Soxhlet extraction	P024b
Volatiles	P024c
Drying and concentrating solvent extracts	P031
Digestion procedures for metals	P032
Sample cleanup procedures	
Florisil column chromatography	P041
BioBeads SX-3	P042
Silica gel chromatography	P043
Alumina column chromatography	P044
Liquid/liquid extraction	P045

Source: Arthur D. Little, Inc., "Sampling and Analysis Methods for Hazardous Waste Combustion," EPA-600/8-84-002, PB84-155845, February 1984.⁴

D-4327-84 procedure is recommended over Method 300.0. The other methods mentioned above may be suitable in some circumstances. The draft EPA method referred to in Section 3.3.3.1 specifies ion chromatography and provides details.

3.4.5.2 Particulate Analysis

Particulate must be measured in the stack gas to demonstrate a maximum particulate emission concentration of no greater than 180 mg/Nm³, corrected to 7% O₂ (50% excess air) (40 CFR 264.345). The particulate is measured gravimetrically according to the procedures established in EPA Reference Method 5 (40 CFR 60).

Two procedures are available. In the first procedure, the filter is desiccated and then weighed to a constant weight. In the alternate procedure, the filter is oven-dried for 2 to 3 h at 105°C (220°F) and cooled in a desiccator

Table 9. Sample Preparation and Introduction Techniques Given in SW-846^a

Sample preparation and introduction techniques	Method number
<i>Sample workup techniques</i>	
<i>Inorganics</i>	
Acid digestion of waters for total recoverable or dissolved metals for analysis by FLAA ^b or ICP ^c	3005
Acid digestion of aqueous samples and extracts for total metals analysis by FLAA or ICP	3010
Acid digestion for aqueous samples and extracts for total metals analysis by GFAA ^d	3020
Dissolution procedure for oils, greases, or waxes	3040
Acid digestion of sediments, sludges, or waxes	3050
<i>Organics</i>	
Organic extraction and sample preparation	3500
Separatory funnel liquid-liquid extraction	3510
Continuous liquid-liquid extraction	3520
Soxhlet extraction	3540
Sonication extraction	3550
Waste dilution	3580
<i>Sample introduction techniques</i>	
Purge and trap method	5030
Protocol for analysis of sorbent cartridges from VOST ^e	5040
<i>Sample cleanup</i>	
Cleanup	3600
Alumina column cleanup	3610
Alumina column cleanup and separation of petroleum wastes	3611
Florisil column cleanup	3620
Silica gel cleanup	3630
Gel-permeation cleanup	3640
Acid-base partition cleanup	3650
Sulfur cleanup	3660

^a Source: U.S. Environmental Protection Agency/Office of Solid Waste, Washington, DC, "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846, Third Edition, November 1986.⁵

^b FLAA = flame atomic absorption spectroscopy.

^c ICP = inductively coupled plasma spectroscopy.

^d GFAA = graphite furnace atomic absorption spectroscopy.

^e VOST = volatile organic sampling train.

before the filter is weighed to a constant weight. Note that the filter used for particulate analysis should not also be used as part of the semivolatile organic analysis, since the drying-to-constant-weight procedure may lose some of the semivolatile organics.

The probe rinse is checked for any leakage during transport. The liquid is measured volumetrically or gravimetrically to the nearest ± 1 mL (± 1 g). The contents are transferred to a tared 250-mL beaker. The probe rinse is evaporated to dryness at ambient temperature and pressure. The beaker is weighed to a constant weight, and the results are reported to the nearest 0.1 mg.

Table 10. Analytical Methods for Characteristics of RCRA Hazardous Waste Feed Samples

Characteristics of hazardous wastes	Analysis method	Source ^a
Ignitability: Pensky-Martens closed-cup method	1010 D-93-80 C001	SW-846 ASTM ADL
Ignitability: Setaflash closed-cup method	1020 D-3278-78 C001	SW-846 ASTM ADL
Corrosivity toward steel	1110 C002	SW-846 ADL
pH electrometric measurement	9040 C002	SW-846 ADL
Reactivity	8.3 C003	SW-846 ADL
Extraction procedure (EP) toxicity	1310 C004	SW-846 ADL
*Appendix VIII hazardous constituents	See Appendix	
Ultimate analysis (elemental composition)	A003	ADL
*Viscosity	A005 D-445-79	ADL ASTM
*Higher heating value	A006 D-2015-77 D-3286-77	ADL ASTM ASTM
Chlorides	D-808-81	ASTM
Ash: Sample drying and ignition	A001a,b D-1888-78	ADL ASTM
Ash: Thermogravimetric analysis	A002 D-1888-78	ADL ASTM
Total organic carbon (TOC)	9060 A004	SW-846 ADL
Total organic halides (TOX) ^b	9020 A004	SW-846 ADL
Total and amenable cyanide	9010	SW-846
Sulfides	9030	SW-846

Note: * Required under RCRA.

^a Sources:

ADL = Arthur D. Little, Inc., "Sampling and Analysis Methods for Hazardous Waste Combustion," EPA-600/8-84-002, PB84-155845, February 1984.⁴

SW-846 = U.S. Environmental Protection Agency/Office of Solid Waste, Washington, DC, "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846, Third Edition, November 1986.⁵

ASTM = American Society for Testing and Materials, "Annual Book of ASTM Standards," Philadelphia, Pennsylvania.⁶

^b The methods listed were developed for wastewater and are not applicable to organic waste. If this analysis is necessary, the Source Methods Standardization Branch, AREAL, EPA or OSW, EPA should be consulted.

Weights are reported separately as filter weight and probe rinse.

3.4.5.3 Oxygen and Carbon Dioxide

Oxygen and carbon dioxide are collected in sample bags (e.g., Mylar, Tedlar) and analyzed by EPA Reference Method 3 (see Table 5). The method uses the Orsat apparatus to measure the volumetric change in liquid volume with selective absorption of oxygen or CO₂. Samples must be analyzed within 3 h, so the analysis is almost always conducted on-site.

Integrated bag samples, if required, must be collected over the entire test period.

3.4.5.4 Analysis for POHCs

The POHCs are divided into two general groups for analysis — volatile and semivolatile organics. Methods for these groups are discussed below. Analysis for metals may also be required and is presented briefly below.

Volatile POHCs — The volatile POHCs are collected either on the VOST or in Tedlar gas bags (S011) for the high concentration volatiles which saturate the

Table 11. Analytical Methods for Principal Organic Hazardous Constituents (POHCs) Given in EPA-600/8-84-002

Description of analysis methods	Analysis method
Volatiles	A101
Purging procedure for the analysis of aqueous liquids	A101a
Purging procedure for the analysis of sludges	A101b
Purging procedure for the analysis of solids	A101c
Extractables	A121
HPLC/UV generalized procedure	A122
HPLC/UV generalized procedure	A123
Aldehydes — derivatization procedures	A131
Aldehydes — HPLC analysis	A132
Carboxylic acids	A133
Alcohols	A134
Phosphine	A136
Fluorine	A137
Gases — cyanogens and phosgene	A138
Gases — mustards	A139
Gases	A141
Acid chlorides	A144
Aflatoxins	A145
Brucine	A148
Citrus red No. 2	A149
Cycasin	A150
Ethylene oxide	A156
2-Fluoroacetamide	A157
Lasiocarpine	A160
Phenacetin	A174
Strychnine	A180
Oximes	A183
Tris(1-aziridinyl)phosphine sulfide	A190

Source: Arthur D. Little, Inc., "Sampling and Analysis Methods for Hazardous Waste Combustion," EPA-600/8-84-002, PB84-155845, February 1984.⁴

VOST, as described in Section 3.3.3.2. The sorbent tubes from the VOST are analyzed by thermal desorption/gas chromatography/mass spectrometry as described in the VOST Method 5040 in SW-846.⁶ Several variations on the basic thermal desorption unit design are available from vendors or may be constructed by the analytical laboratory. The desorption unit must be leakand contamination-free, as demonstrated by QC sample performance. The laboratory must also have the capability and experience to generate reliable standards for quantitation.

VOST analyses require not only adequate facilities, but also experience and good technique. A major potential problem is the presence of background compounds in the samples as evidenced by the blank samples. Background contamination can generally be traced to problems with the trap cleanup, sample collection, or sample handling (see Section 3.3.3.3). In addition, care must be taken that the analysis step does not introduce contamination. Cross contamination from previous high-level samples can be a problem. Contamination from ambient air can also be a problem.

The VOST method (5040 in SW-846⁶) indicates that VOST is valid for compounds with a boiling point of less than 100°C (and generally higher than 30°C) and should be validated prior to use outside this range. There is

occasionally a need to consider use of VOST for compounds with boiling points in the 100° to 130° range. A compound with a boiling point in this range may be difficult to purge from the VOST trap. The validity of the method for these POHCs should be determined prior to testing; this can be done by using previous test data or by validating the method using QA samples. For example, the VOST protocol provides a QA procedure whereby the sample traps are spiked with the compound of interest and analyzed to determine percent sample recovery.

One operational problem with VOST is sample overload during the analysis. The objective of the VOST analysis is to demonstrate a DRE of 99.99% or greater. The amount of a POHC collected on the VOST is dependent on both the DRE and the amount fed into the incinerator. The amount of POHC fed should be scaled to yield the POHC at a concentration near the middle of the GC/MS range, assuming 99.99% DRE. Thus very low quantities from the VOST trap indicate far greater than the required DRE, and very high quantities indicate far less than the required DRE. High VOST analysis results may be above the cutoff for the DRE requirement. Depending on the data-reporting requirements, precise quantitation of high values may not be necessary if the 99.99% DRE requirement is not met. In this case the VOST sample volume and the calibration range of the analysis can be scaled to demonstrate the 99.99% DRE with less concern about quantitation of values much greater or lower than the regulatory target. The best approach for most trial burns is to scale the POHC feed rate to match the needed collection on the VOST. If this is not possible, use a second sampling method (i.e., bag sample) as a backup in case the VOST samples saturate the GC/MS.

An option listed in EPA-600/8-84-002⁴ by which a sample of stack gas is collected in a Tedlar bag (Method S011) presents several problems with respect to the short holding times of these samples, as discussed in Section 3.3.2.3. If Tedlar bag samples are used to quantitate high-level samples, a known volume of gas is pulled through a clean VOST trap. The gas volume to be collected on the trap is determined from an estimation of the concentration of the POHC from the saturated peak obtained in the analysis of the first VOST trap. The trial burn plan must include appropriate QA measures to demonstrate that field samples are valid for the holding times and gas matrix actually encountered from the test.

Semivolatile POHCs — The semivolatile POHCs are sampled using a semi-VOST with an XAD-2 sorbent module, as described in Section 3.3.3.3. Nonvolatile POHCs (boiling points above 300°C) are also collected by the semiVOST sampling train.

The semivolatile and nonvolatile POHCs are generally analyzed by extraction followed by GC/MS analysis. The appropriate standard methods are cited in Tables 8, 9, 11, and 12, and Appendix A. Tables 8 and 9 list the sample preparation and introduction techniques. Tables 11 and

Table 12. Analytical Methods for Principal Organic Hazardous Constituents (POHCs) Given in SW-846

Description of analytical methods	Analysis method
Gas chromatographic methods (GC)	
Halogenated volatile organics	8010
Nonhalogenated volatile organics	8015
Aromatic volatile organics	8020
Acrolein, acrylonitrile, acetonitrile	8030
Phenols	8040
Phthalate esters	8060
Organochlorine pesticides and PCBs*	8080
Nitroaromatics and cyclic ketones	8090
Polynuclear aromatic hydrocarbons	8100
Chlorinated hydrocarbonsd	8120
Organophosphorus pesticides	8140
Chlorinated herbicides	8150
Gas chromatographic/mass spectroscopy methods (GC/MS)	
GC/MS method for volatile organics	8240
GC/MS method for semivolatile organics:	
Packed column technique	8250
Capillary column technique	8270
GC/MS method for PCDD/PCDF ^b	8280
High performance liquid chromatography (HPLC)	
HPLC — polynuclear aromatic hydrocarbons	8310

Source: U.S. Environmental Protection Agency/Office of Solid Waste, Washington, DC, "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846, Third Edition, November 1986.⁶

* PCBs = polychlorinated biphenyls

^b PCDD = polychlorinated dibenzo-p-dioxins
PCDF = polychlorinated dibenzofurans

Table 13. Analytical Methods for Inorganics

Inorganics	ADL method number	Analysis method*	SW-846 method number	Analysis method*
Aluminum	A021	ICAP	6010	ICAP
Antimony	A221	AAS, DAM AAS, GFM	7020 6010 7040 7041	AAS, DAM ICAP AAS, DAM AAS, GFM
Arsenic	A222	AAS, GFM AAS, GH	6010 7060 7061	ICAP AAS, DAM AAS, GH
Barium	A021 A223	ICAP AAS, DAM AAS, GFM	6010 7080	ICAP AAS, DAM
Beryllium	A021 A224	ICAP AAS, DAM AAS, GFM	6010 7090 7091	ICAP AAS, DAM AAS, GFM
Boron	A021	ICAP	6010	ICAP
Cadmium	A021 A225	ICAP AAS, DAM AAS, GFM	6010 7130 7131	ICAP AAS, DAM AAS, GFM
Calcium	A021	ICAP	6010	ICAP
Cobalt	A021	ICAP	7140 6010	AAS, DAM ICAP
Chromium	A021 A226	ICAP AAS, DAM AAS, GFM	7200 7201 6010 7190 7191	AAS, DAM AAS, GFM ICAP AAS, DAM AAS, GFM
Hexavalent chromium*	— — — —	— — — —	7195 7196 7197 7198	COPRTN C C/E DPP
Copper	A021	ICAP	6010	ICAP
Iron	A021	ICAP	7210 6010 7380	AAS, DAM ICAP AAS, DAM
Lead	A021 A227	ICAP AAS, DAM AAS, GFM	6010 7420 7421	ICAP AAS, DAM AAS, GFM
Magnesium	A021	ICAP	6010	ICAP
Manganese	A021	ICAP	7450 6010	AAS, DAM ICAP
Mercury	A228	CV, AAS	7460 7470 7471	AAS, DAM CV, AAS, L CV, AAS, S
Molybdenum	A021	ICAP	6010 7480 7481	ICAP AAS, DAM AAS, GFM
Nickel	A021 A229	ICAP AAS, DAM AAS, GFM	6010 7520	ICAP AAS, DAM
Osmium	A021 A230	ICAP AAS, DAM AAS, GFM	7550	AAS, DAM
Phosphorus	A021	ICAP	—	—
Potassium	A021	ICAP	6010	ICAP
Selenium	A231	AAS, GFM AAS, GH	7610 6010 7740 7741	AAS, DAM ICAP AAS, GFM AAS, GH
Silicon	A021	ICAP	6010	ICAP
Silver	A021 A232	ICAP AAS, DAM AAS, GFM	6010 7760 7761	ICAP AAS, DAM AAS, GFM
Sodium	A021	ICAP	6010	ICAP
Strontium	A021 A233	ICAP AAS, DAM AAS, GFM	7770	AAS, DAM

Table 13. (continued)

Inorganics	ADL method number	Analysis method*	SW-846 method number	Analysis method*
Thallium	A021 A234	ICAP AAS, DAM AAS, GFM	6010 7840 7841	ICAP AAS, DAM AAS, GFM
Thorium	A021	ICAP	—	—
Titanium	A021	ICAP	—	—
Vanadium	A021 A235	ICAP AAS, DAM AAS, GFM	6010 7910 7911	ICAP AAS, DAM AAS, GFM
Zinc	A021	ICAP	6010 7950	ICAP AAS, DAM
Zirconium	A021	ICAP	—	—
Anions	A251	IC	—	—
Total cyanides	A252	T, C	9010 9012	C —
Phosphides	A253	GC/FPD	—	—
Sulfides	—	—	9030	T

* ICAP = inductively coupled argon plasma emission spectroscopy
AAS, DAM = atomic absorption spectroscopy, direct aspiration method
AAS, GFM = atomic absorption spectroscopy, graphite furnace method
AAS, GH = atomic absorption spectroscopy, gaseous hydride method
COPRTN = coprecipitation method
CV, AAS = cold vapor/atomic absorption spectroscopy
CV, AAS, L = cold vapor/atomic absorption spectroscopy, for liquids
CV, AAS, S = cold vapor/atomic absorption spectroscopy, for solids
C = colorimetric method
C/E = chelation/extraction method
T = titration method
IC = ion chromatography
GC/FPD = gas chromatography/flame photometric detector
DPP = differential pulse polarography

Sources: Arthur D. Little, Inc., "Sampling and Analysis Methods for Hazardous Waste Combustion," EPA-600/8-84-002, PB84-155845, February 1984.⁴

U.S. Environmental Protection Agency/Office of Solid Waste, Washington, DC, "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846, Third Edition, November 1986.⁶

* The methods listed for hexavalent chromium were developed for water samples.

12 list applicable instrumental analysis methods. Appendix A lists all of the analysis methods for the Appendix VIII hazardous constituents by compound or element in alphabetical order.

Each semi-VOST sample returns to the laboratory in several fractions which must be treated and ultimately combined to yield a full sample. Typically the fractions are (1) filter, (2) sorbent trap, (3) front-half organic rinse, (4) back-half organic rinse, and (5) condensate from and rinses of impingers. The fractions are spiked with surrogates, extracted, combined, cleaned up, and then analyzed.

Surrogate compounds are generally isotopically labeled (e.g., deuterated) analogs of the POHCs or similar com-

pounds not found in the waste feed which can be spiked into the samples to monitor recovery of the native POHCs. Since several fractions must be extracted using different techniques, surrogates may be split into groups to monitor recovery of each extraction.

Table 14. Analytical Methods for Stack Gas Samples

Sample	Analysis parameter ^a	Analysis method
M5 train		
Filter, probe rinse	Particulate	EPA Method 5 ^b
Water impingers	Cl ⁻ (HCl)	Ion chromatography or EPA 352.2 ^c
Caustic impinger	Cl ⁻ (HCl)	Ion chromatography or EPA 352.2 ^c
Multiple metals train	Metals	See Table 13
Semi-VOST		
Filter, probe rinse	SV-POHCs	See Appendix A
XAD-2	SV-POHCs	GC/MS
Condensate	SV-POHCs	
VOST	V-POHCs	GC/MS per SW-846, Method 5040
Mylar gas bag	CO ₂ , O ₂	EPA Method 3 ^b
Tedlar gas bag	V-POHCs	Transfer to Tenax trap and GC/MS per SW-846, Method 5040

^a SV-POHCs = semivolatile and nonvolatile principal organic hazardous constituents
V-POHCs = volatile principal organic hazardous constituents

^b Source: 40 CFR Part 60, Appendix A.7

^c Reference: "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1979.¹⁵

The filter and sorbent traps are generally Soxhlet-extracted with dichloromethane (Method 3540), benzene, hexane, or other organic solvent. The organic rinse fractions are combined with the filter and sorbent trap extracts. The aqueous fractions are extracted using liquid-liquid partition in a separatory funnel (e.g., EPA Method 3510 using dichloromethane) or by continuous liquid-liquid extraction (e.g., EPA Method 3520 with dichloromethane). A second extraction of aqueous fractions with methyl-t-butyl ether may be necessary when the POHC is a water-soluble compound (e.g., pyridine).

The sample extracts are then evaporatively concentrated to the desired volume. Kuderna-Danish evaporation and other techniques are described in the appropriate SW-846 extraction methods.

A variety of adsorbent column and other cleanup methods are available for cleanup of organic extracts prior to analysis. Several are listed in SW-846 (Section 4.2.2) and EPA-600/8-84-002 (P041-P045). The selection of the sample cleanup technique is dependent on the type and concentration of the interferences. Prior experience with

the matrix or screening of the samples can help characterize the interferences. For many trial burns with relatively clean stack gases, no cleanup may be required. If the POHC levels are low enough to require special mass spectral techniques, such as selected ion monitoring, cleanup is more likely to be required.

Implementation of the extraction, concentration, and cleanup methods requires not only the detailed written methods and appropriate reagents and materials, but also qualified personnel. Effective execution of these methods, as measured by surrogate compound recoveries and the amount of interferences, comes only after extensive training in general trace organic laboratory methods and experience in the specific methods.

Although not specifically required, most laboratories choose to analyze the extracts for POHCs by GC/MS. The ability of this technique to quantitate the POHCs reliably makes it cost-effective relative to the additional laboratory cleanup required for GC with other detectors. In addition, there is a much greater chance of obtaining higher than true values due to interferences with other detectors, which can result in a reported DRE that is lower than actual.

The GC/MS data must be interpreted both qualitatively and quantitatively. Methods 8240, 8250, 8270, and other applicable methods give general guidance on data interpretation. Qualitative data interpretation entails matching the retention time and spectral characteristics of the sample with the standards. The QA plan must specify criteria on the retention time and spectral characteristics (e.g., presence of ions, ion intensity ratios, etc.). The POHCs are quantitated by the internal standard method. Specifically, the area of the unknown is ratioed to that of the internal standard, multiplied by the amount of the internal standard, and also multiplied by the response factor for that compound relative to the internal standard. The details of the quantitation must be specified in the trial burn plan including number and levels of standards, analysis sequence, blanks, and repeat injections. In addition, the trial burn plan should address contingencies for interferences, high level samples, samples outside the standard curve, and other anticipated problems.

For many high-efficiency incinerators, the POHCs in the stack gas will be below the lower limit of quantitation (LOQ) or even below the limit of detection (LOD). The trial burn plan should give the criteria for LOQ and LOD and should also state how samples in these ranges will be reported. Reporting of low-level numbers can affect the calculation of the DRE and can also cause confusion in the trial burn report review process.

Data interpretation not only requires qualified and experienced personnel, but also time. Sufficient time must be allowed in the analysis and reporting schedule for the data reduction.

Metals.— Analysis of stack gas samples for metals may be conducted during some trial burns and the applicable methods are listed in Table 13. The draft protocol for the multiple metals train discussed in Section 3.3.3.4 contains additional information on analysis. It employs a hydrofluoric acid/nitric acid digestion in microwave bombs for the probe rinse and filter samples resulting from stack sampling. It also offers an option of either a hydrofluoric acid/nitric acid digestion in microwave bombs or a nitric acid/hydrogen peroxide digestion on a hot plate for the impinger contents except the potassium permanganate impinger, which is analyzed for mercury only. An aliquot of the other (combined) impinger contents is also reserved for mercury analysis.

3.4.6 Analysis of Other Effluent Streams

In addition to the stack samples, samples from the other effluent streams must be analyzed. Although not calculated in the DRE, a quantitative analysis of the scrubber water, ash residues, and other residues is required by 40 CFR, Part 270.62 for the purpose of estimating the fate of POHCs. This section addresses analysis of quench and scrubber water samples and ash samples. Analysis methods for other effluent streams should be adaptable from similar waste matrices, as described in Section 3.4.4.

General characterization (e.g., proximate analysis) and inorganic analysis of the water and ash samples can be conducted using the same methods used for waste feed samples, as discussed in Section 3.4.4 and summarized in Table 10.

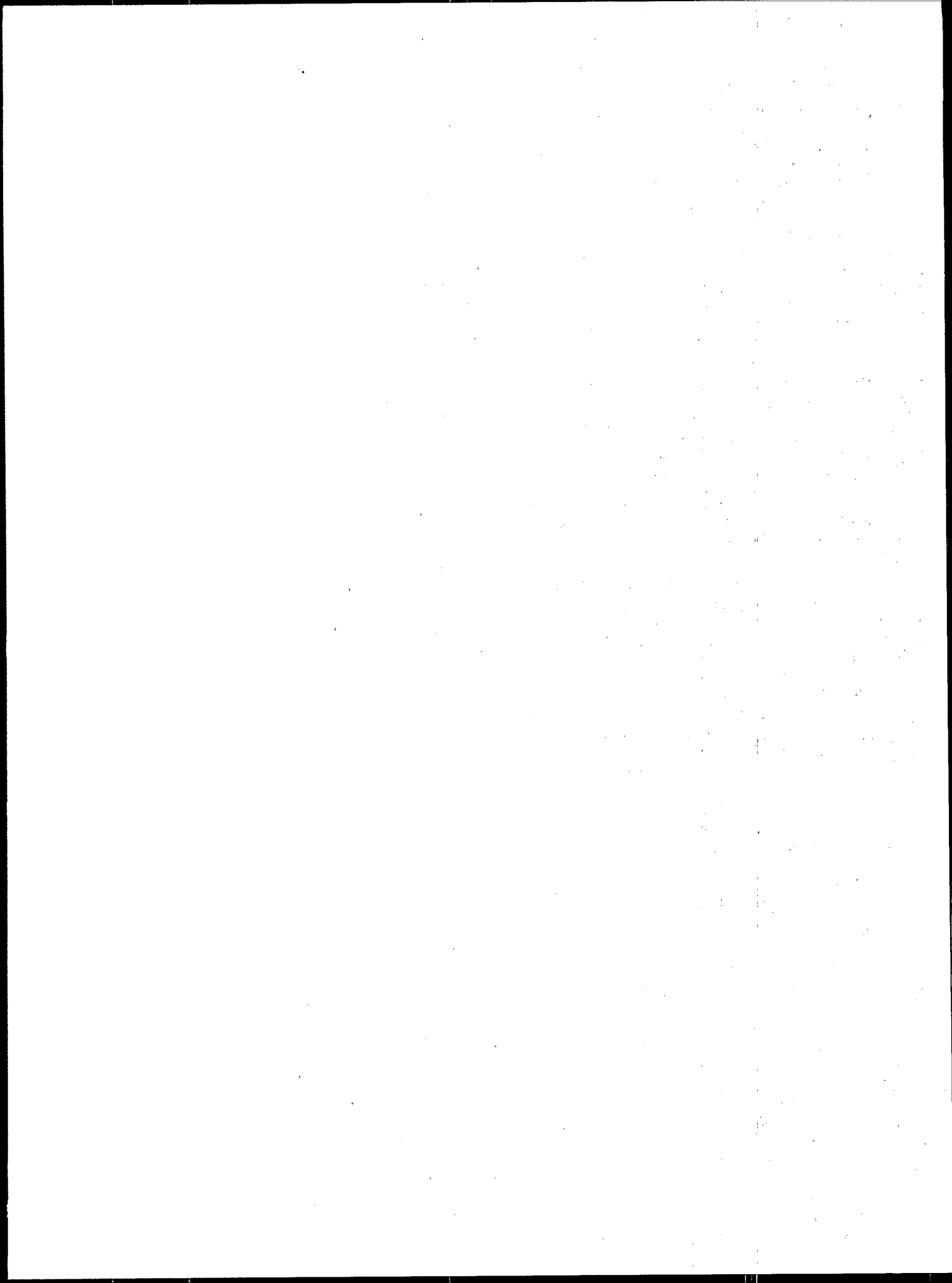
3.4.6.1 Quench and Scrubber Water

For semivolatile analysis, water samples are extracted using liquidliquid partition in a separatory funnel (Method

3510 in SW-846 using dichloromethane) or continuous liquid-liquid extraction (Method 3520 in SW-846 using dichloromethane). The sample extracts are then evaporatively concentrated to the desired volume. Kuderna-Danish evaporation and other techniques are described in the appropriate SW-846 extraction methods. If needed, a variety of adsorbent column and other cleanups are available for cleanup of organic extracts prior to analysis. Several are listed in SW-846 (Section 4.2.2) and EPA-600/8-84-002 (P041-P045). For many trial burns with relatively clean stack gas and thus clean scrubber water, no cleanup will be required. Most laboratories choose to analyze the quench and scrubber water extracts by GC/MS. Methods 8250, 8270, and other applicable methods give general guidance on analysis of semivolatiles and data interpretation. For volatile POHCs, the samples are prepared for and analyzed by purge and trap using the A. D. Little A101 series methods or the SW-846 Methods 5030 (preparation) and 8240 (analysis). Section 3.4.5.4.2 addresses the GC/MS analysis in more detail.

3.4.6.2 Ash Samples

To determine semivolatile POHCs in ash samples, the samples are Soxhletextracted using dichloromethane (Method 3540 in SW-846), benzene, hexane, or other organic solvent. The sample extracts are then evaporatively concentrated to the desired volume, cleaned up, and analyzed by GC/MS as described for the water samples in the preceding section. To determine volatile POHCs in ash samples, the samples are prepared in a tetraglyme dispersion (Method 5030 in SW-846, A101C in EPA-600/8-84-002) and then analyzed by purge and trap.



Section 4

Quality Assurance/Quality Control

4.1 Data Quality Objectives

The subject of data quality objectives (DQOs) has recently been addressed by the Quality Assurance Management Staff (QAMS) of EPA in a publication entitled *Development of Data Quality Objectives: Description of Stages I and II*.¹⁶ This document describes an approach to designing environmental data collection programs based on the development of DQOs which is intended to define the quality of the data needed to achieve an acceptable level of confidence and provide adequate information to make regulatory decisions and recommendations. The DQO process provides a logical, objective, and quantitative framework for finding an appropriate balance between the time and resources that will be used to collect data and the quality of the data needed to make the decision.

Data quality objectives are statements of the level of uncertainty that a decision maker is willing to accept in results derived from environmental data, when the results are going to be used in a regulatory or programmatic decision. These quantitative DQOs must be accompanied by clear statements of:

- The decision to be made.
- Why environmental data are needed and how they will be used.
- Time and resource constraints on data collection.
- Descriptions of the environmental data to be collected.
- Specifications regarding the domain of the decision.
- The calculations, statistical or otherwise, that will be performed on the data in order to arrive at a result.

Once DQOs have been developed and a design for the data collection activity expected to achieve these objectives has been selected, DQOs are used to define quality assurance (QA) and quality control (QC) programs that are specifically tailored to the data collection program being initiated. A QA Project Plan is prepared documenting all of the activities needed to ensure that the data collection program will produce environmental data of the type and quality required to satisfy the DQOs. Without first developing DQOs, a QA program can only be used to document the quality of data obtained, rather

than to ensure that the quality of data obtained will be sufficient to support an Agency decision. This approach to DQOs is recommended when planning QA for an RCRA trial burn.

4.2 General Discussion of QA Project Plan

The quality assurance/quality control (QA/QC) procedures for the process monitoring, sampling, and analytical activities for a trial burn and the continuing operation of a hazardous waste incinerator must be included in a quality assurance (QA) project plan which accompanies the permit application. The QA plan should be based on the guidelines document issued by the Office of Monitoring Systems and Quality Assurance of EPA entitled *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*.¹⁷ The document has identified 16 essential elements of a QA plan, which are shown in Table 15.

Table 15. Essential Elements of a QA Project Plan

1. Title Page
2. Table of Contents
3. Project Description
4. Project Organization and Responsibility
5. QA Objectives
6. Sampling Procedures
7. Sample Custody
8. Calibration Procedures and Frequency
9. Analytical Procedures
10. Data Reduction, Validation, and Reporting
11. Internal Quality Control Checks
12. Performance and Systems Audits
13. Preventative Maintenance
14. Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness
15. Corrective Action
16. Quality Assurance Reports to Management

Source: U.S. Environmental Protection Agency/Office of Monitoring Systems and Quality Assurance, Office of Research and Development, Washington, D.C., "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80 (December 29, 1980).¹⁷

A QA plan prepared for a trial burn must contain *specific* QA/QC procedures for the process monitoring, sampling, and analytical activities identified in the trial burn plan and permit application. The plan should contain sufficient detail to allow the permit writer to assess the adequacy of the QA/QC procedures that will be used.

The discussion that follows first presents a general discussion of the 16 elements required in a QA plan and then discusses specific guidance for assessing precision and accuracy for individual measurement methods.

4.2.1 Title Page

The title page of the QA plan must be signed by approving personnel. The plan must be approved by the project leader's immediate supervisor and the quality assurance manager (QAM). If the QA plan is prepared by a subcontractor to the permit applicant, the title page should also contain the approving signatures of the subcontractor's program manager and QAM.

4.2.2 Table of Contents

The table of contents should list the introduction, the 16 essential elements of the QA plan, and any appendices attached to the plan (such as standard operating procedures, etc.).

4.2.3 Project Description

The project description is presented in detail in the permit application. In the QA plan, a brief summary of the project description should be provided and reference to the applicable section(s) of the permit application should be made. The summary should, however, provide sufficient detail so that the reviewer of the QA plan can understand the important elements of the project plan.

4.2.4 Project Organization and Responsibility

The project organization responsible for all QA/QC activities should be provided in an organization chart that gives the names, titles, and line authority of the individuals. The resumes and QA/QC responsibilities for each individual in the organization should be provided. This organization should include the following:

- Project leader
- Quality assurance manager (QAM)
- Field sampling task leader
- Field sampling quality control coordinator
- Analytical task leader
- Analytical quality control coordinator
- Quality control and data manager
- Data analysis task leader

4.2.5 QA Objectives

The primary QA objective for any project is to ensure that the measurement data collected are precise, accurate,

complete, and representative. This section of the QA plan should give specific precision, accuracy, and completeness objectives for each process measurement and analysis performance. Guidance for precision, accuracy, and completeness objectives for specific measurement parameters is given in Section 4.3.

The terms *precision*, *accuracy*, *completeness*, and *representativeness* may be defined as follows:

- *Precision* is the degree of agreement between repeated measurements of one property using the same method or technique and is usually expressed as a range percent (R%) for a small number of data points ($2 \leq n \leq 8$) or as percent relative standard deviation (%RSD) for a large ($n > 8$) number of data points.
- *Accuracy* is the degree of agreement of a measurement (or average of measurements of the same thing), X , with an accepted reference or true value, T , usually expressed as percent accuracy (%A) defined as $X/T (X100)$.
- *Completeness* is a measure of the amount of valid data obtained from a measurement system compared to the amount of data collected that was expected, usually expressed as a percent.
- *Representativeness* expresses the degree to which measurement data and samples precisely, accurately, and completely characterize the process conditions.

4.2.6 Sampling Procedures

The sampling procedures to be used in the trial burn and continuing operation of the incinerator should be specified in the trial burn plan and permit application. Sampling procedures should include the sampling methods, sampling locations, sampling frequencies, sampling equipment, and data recording methods. All forms for recording sampling data should be given.

The QA plan should present any details necessary to describe the sampling procedures that are not given in the trial burn plan and permit application. Particular attention should be given to any anticipated deviations from referenced sampling methods.

4.2.7 Sample Custody

Two procedures can be used to document sample custody for a trial burn: traceability and chain-of-custody. Traceability procedures are acceptable to EPA and should be used unless litigation is anticipated, in which case the applicant may select the more rigorous chain-of-custody procedures.

The QA plan should describe all of the elements of sample traceability including sample labeling, preservation, packing, shipping, and laboratory receiving and storage procedures. All documentation and record-keeping forms such as field sampling traceability forms, field logbook, sample analysis request sheet, laboratory traceability forms, and laboratory logbook should be described. Stor-

age of samples after analysis must also be addressed.

If the chain-of-custody procedure will be used, then a complete description of the procedure must be given in the QA plan. Chain-of-custody procedures acceptable to EPA are fully described in SW-846, Section 1.3.⁶

4.2.8 Calibration Procedures and Frequency

The calibration procedures for all monitoring and sampling equipment to be calibrated should be specifically defined. For each piece of equipment, the calibration technique, reference standard(s), acceptance limits, and frequency of calibration should be specified.

Calibration of analytical instruments is usually specified in the reference methods. If the analytical methods do not specify calibration procedures or deviations from the methods are anticipated, the calibration procedures must be fully described. The calibration standards, calibration curves, average response factors, and/or relative response factors to be used for each instrument and analysis should be specified. The methods for recording and storing calibration data should be given.

4.2.9 Analytical Procedures

The analytical procedures to be used to analyze samples collected should be specified in the trial burn plan and permit application. Analytical procedures should include the analytical methods, instrumentation and equipment, and data-recording methods. All forms to be used for recording analytical data should be given.

The QA plan should provide any details necessary to describe the analytical procedures that are not given in the trial burn plan and permit application. Particular attention should be given to any anticipated deviations from referenced procedures and methods.

4.2.10 Data Reduction, Validation, and Reporting

All procedures for the reduction of monitoring, sampling, and analytical data collected should be specified. Equations, calculations, and conversion factors should be given for determining specific results to be reported, such as stack emissions, waste feed concentrations, etc. Methods used to obtain and reduce analytical data, such as method internal standards, recovery internal standards, relative response factors, and qualitative and quantitative peak identification, should be given.

Procedures to validate the integrity and quality of the data acquired through readings, interpretations, and calculations should be specified. Some examples of data validation procedures are:

- a. Verification by the analytical task leader that all raw data generated have been properly stored.
- b. Examination of at least 5% of the raw data (e.g., chromatograms) by the analytical task leader and analytical QCC to verify adequacy of documentation, confirm

peak shape and resolution, ensure computer was sensing peaks appropriately, etc.

- c. Confirmation that raw areas for internal standards and calibration standards and raw and relative areas for surrogate compounds are within acceptable limits around the expected value.
- d. Verification that all associated blank, standard, and QC data are reported along with analytical results.
- e. Examination of all field data forms by the field sampling task leader and field sampling QCC.
- f. Verification of all calculations for one test run by a second reviewer.

The test results and operating parameters to be reported should be specified in a table including the units (e.g., lb/h) for each parameter. All documentation (e.g., field data sheets, calculations, GC/MS printouts, etc.) that will be delivered with the reported results should be identified.

4.2.11 Internal Quality Control Checks

All internal quality control methods should be described for monitoring, sampling, and analysis activities. Internal quality controls serve to document the validity of the data obtained and to control the quality of the data as it is being generated.

Items which should be included in the QA plan as internal quality control checks include the following:

- Calibration standards and devices
- Zero/span gases
- Audit gas cylinders
- Blank samples
 - Field trip blanks
 - Field equipment blanks
 - Method blanks
 - Reagent and solvent blanks
- Method internal standards
- Recovery internal standards
- Spiked blanks
- Spiked samples
- Replicate analyses
- Matrix/spike duplicate analyses
- Surrogates
- Calibration checks

4.2.12 Performance and Systems Audits

The QA plan should describe the types of performance and systems audits that will occur and identify the responsible individuals. Performance of equipment and personnel should be audited periodically by the project leader, field sampling leader, analytical task leader, the

quality assurance officer, and the quality control coordinators. Systems audits should also be performed periodically on monitoring systems, sampling, and analytical systems.

4.2.13 Preventive Maintenance

The frequency and types of scheduled preventive maintenance procedures used for process monitoring equipment, sampling equipment, and analytical equipment and instrumentation should be given in the QA plan. In addition, any nonscheduled maintenance procedures which may occur when troubleshooting should be provided. It is desirable that the preventive maintenance procedures be described in standard operating procedure (SOP) manuals for the various types of equipment used.

Logbooks for maintenance performed by field sampling and laboratory personnel should be maintained. Equipment logbooks should also be kept for maintenance performed by internal service departments and outside service departments.

4.2.14 Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness

The specific procedures that will be used to assess the precision and accuracy of measurement data on a routine basis must be given. The procedures should include equations to calculate precision and accuracy. Examples are given below.

4.2.14.1 Precision

For data sets with a small number of points ($2 \leq n \leq 8$), the estimate of precision can be expressed as range percent (R%):

$$R\% = \frac{C_1 - C_2}{\bar{C}} \times 100$$

where: C_1 = highest value determined

C_2 = lowest value determined

\bar{C} = mean value of the set

and

$$\bar{C} = \sum_{i=1}^n \frac{C_i}{n}$$

where: C_i = i th determination

n = number of determinations

For large data sets ($n > 8$), the estimate of precision can be expressed as percent relative standard deviation (%RSD):

$$\text{Standard deviation (SD)} = \frac{\sum_{i=1}^n \sqrt{(C_i - \bar{C})^2}}{n-1}$$

$$\%RSD = \frac{100\% \times SD}{\bar{C}}$$

The methods for determining how precision will be determined for each measurement parameter should be given.

4.2.14.2 Accuracy

Accuracy can be determined for process monitors and analyses from performance and audit samples (i.e., standards supplied as blind audits by the QCC to determine percent accuracy [A%]); for analyses as percent recovery (R%) of native analytes from blanks spiked with native analytes prior to sample preparation; and for analyses as percent recovery of internal standards (RS%) spiked prior to sample preparation. Example equations for accuracy calculations are given below.

For performance samples:

$$A\% = \frac{\text{Amount found}}{\text{True value}} \times 100$$

For samples spiked with native analyte:

$$R\% = \sum_{i=1}^n \frac{\text{Amount calculated} - \text{native amount prior to spiking}}{\text{Amount spiked}} \times \frac{100}{n}$$

$$RS\% = \sum_{i=1}^n \frac{\text{Amount calculated} - \text{amount of internal standard spiked}}{\text{Amount of internal standard spiked}} \times \frac{100}{n}$$

4.2.15 Corrective Action

Corrective action procedures that will be taken if problems are detected during system audits, performance audits, and data collection; if data are lost; or if significant QA problems develop which must be described in the QA plan. The individuals responsible for initiating corrective action procedures should be identified and the methods for reporting corrective actions should be described.

4.2.16 Quality Assurance Reports to Management

The quality control coordinators, in cooperation with the project leader, analytical task leader, field sampling task leader, and data analysis task leader should inspect critical areas of the project requiring QA/QC activities. The inspections should include a review, where applicable, of the following:

- Staff qualifications
- Equipment calibration and maintenance records
- Instrument performance
- Protocol adherence
- Sample custody
- Document control
- Data entry including error handling, correction, and additions
- Data traceability and completeness
- Data calculation and validation
- Internal QC data
- External QA data
- Data accuracy, precision, and completeness

The results of inspections/audits should be reported by the QAM to the project leader and corporate management; summaries should be included in the final report.

The QCCs should independently maintain a QA file for the project. At the end of the project, the QA project file should be turned over to the QA manager.

4.3 Guidance for Precision and Accuracy Objectives

The quality assurance objective of any project is to provide reliable data for documenting the performance of the incinerator.

Specific precision and accuracy objectives for general analytical procedures are given in Table 16. Objectives for semivolatile POHCs are goals recommended by EPA in the publication *Sampling and Analysis Methods for Hazardous Waste Combustion*.⁴ Objectives for chlorine and hydrogen chloride are estimated from past analysis of similar types of samples.

Quality assurance objectives for completeness and representativeness should be determined from the data quality objectives (DQOs) and should reflect specific requirements of the project.

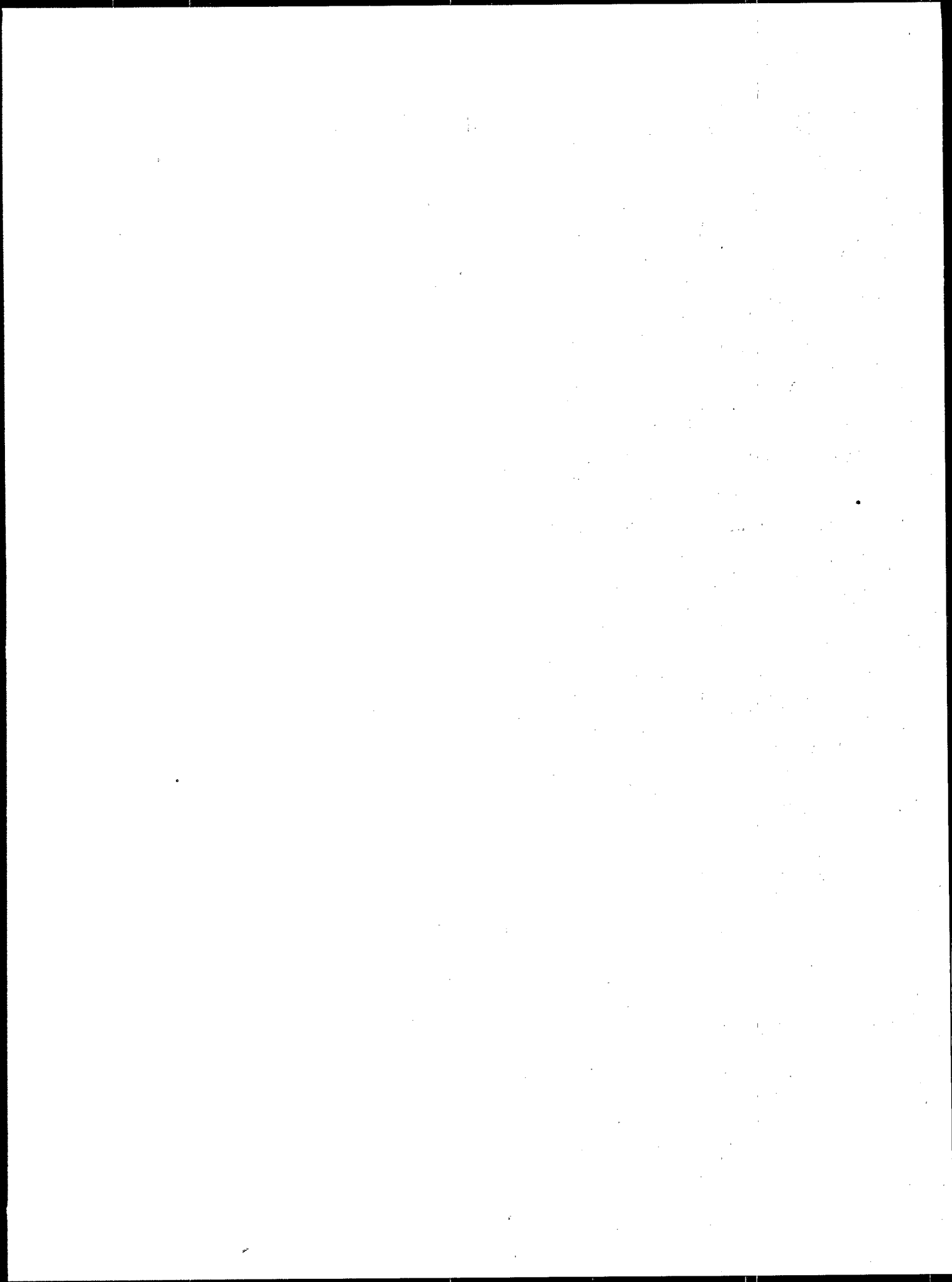
Table 16. Summary of Precision and Accuracy Objectives

Parameter	Matrix	Precision ^a (range % or % RSD)	Accuracy ^b mean recovery (%)
Semivolatile POHC	Stack emissions	< 30	≥ 50
	XAD-2		
	Filter		
	Water		
	Front-half rinse		
	Back-half rinse		
	Aqueous waste	< 30	≥ 70
	Sludge	< 30	≥ 50
	Solid waste	< 30	≥ 50
	Scrubber water	< 30	≥ 70
	Ash/residual	< 30	≥ 50
	Organic liquid	< 30	≥ 70
Volatile POHC	VOST traps	< 50	50-150% ^c
	(stack emissions)		
	Aqueous waste	< 30	NA ^c
	Sludge	< 30	NA
	Solid waste	< 30	NA
	Scrubber water	< 30	NA
Chlorine	Organic liquid waste	< 30	NA
	Aqueous waste	< 10	NA
	Sludge	< 10	NA
	Solid waste	< 10	NA
	Organic liquid waste	< 10	NA
Hydrogen chloride	KOH solution	15	100 ± 15

^a Procedures for assessing precision are presented in Section 4.2.14.

^b Procedures for assessing accuracy are presented in Section 4.2.14.

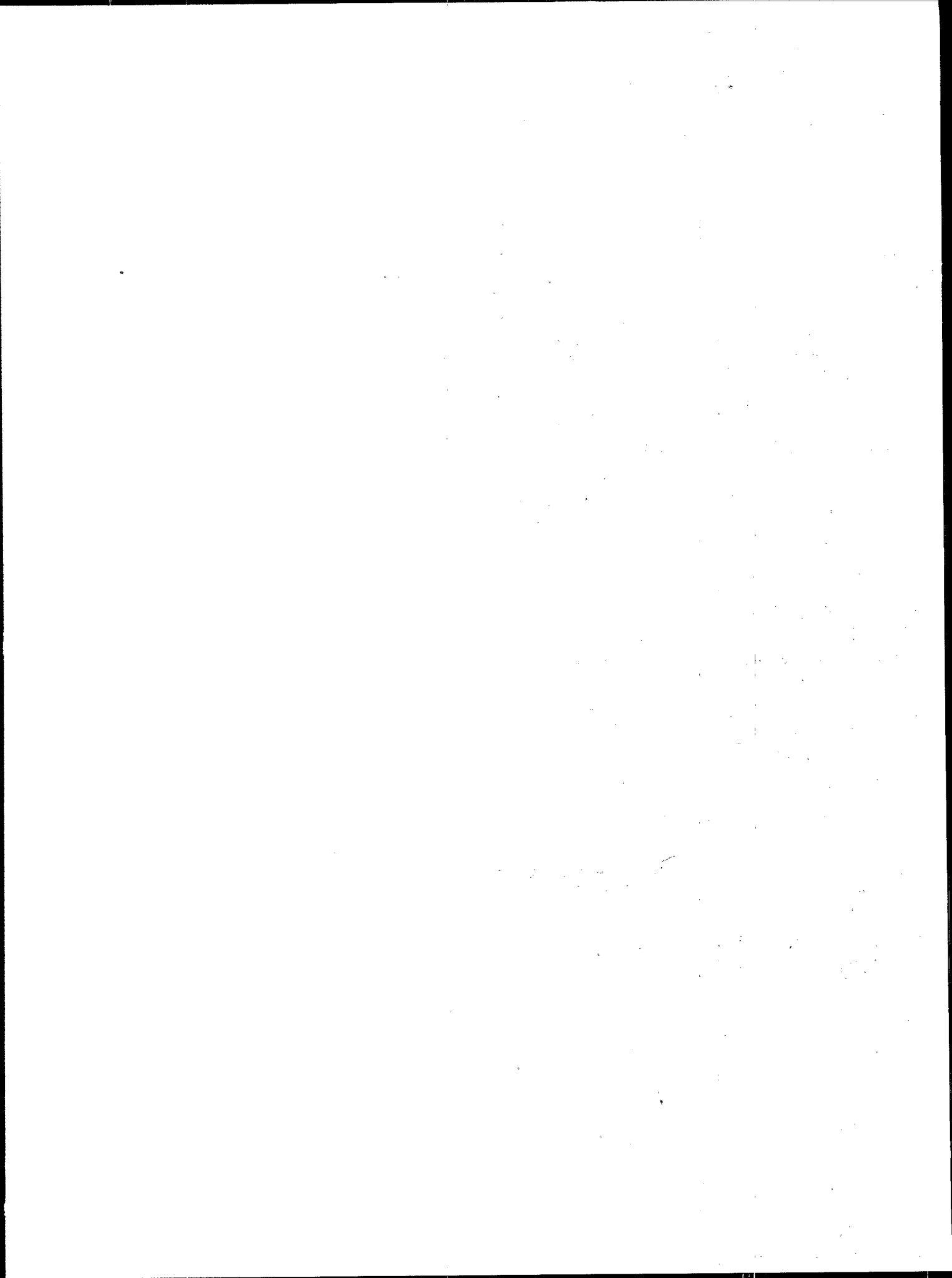
^c Not available.



Section 5

References

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Appendix A

Analysis Methods for Appendix VIII Hazardous Constituents Given in EPA-600/8-84-002 and SW-846

Analysis Methods for Appendix VIII Hazardous Constituents

Compound	EPA-600/8-84-002 Method No. ^a	SW-846 Method No. ^b	Compound Type ^c	Analysis Method ^d
Acetonitrile	A101	8030, 8240	V	GC, GC/MS
Acetophenone	A121	8250	SV	GC/MS
3-(α -Acetylbenzyl)-4-hydroxy-coumarin and salts (Warfarin)	A122	8250	SV	HPLC, GC/MS
2-Acetylaminofluorene	A121	8250	SV	GC/MS
Acetyl chloride	A144	*	V	GC/MS
1-Acetyl-2-thiourea	A123	8250	SV	HPLC, GC/MS
Arcolein	A101	8030, 8240	V	GC, GC/MS
Acrylamide	A101	8015, 8240	V	GC, GC/MS
Acrylonitrile	A101	8030, 8240	V	GC, GC/MS
Aflatoxins	A145	8250	*	HPLC, GC/MS
Aldrin	A121	8080, 8250	SV	GC, GC/MS
Allyl alcohol	A134	8240	V	GC/MS
Aluminum phosphide	A253	—	S	GC/FPD
4-Aminobiphenyl	A121	8250	SV	GC/MS
6-Amino-1,1a,2,8,8a,8b-hexahydro-8-(hydroxymethyl)-8a-methoxy-5-methylcarbamate azirino[2',3':3,4]pyrrolo[1,2-a]indole-4,7-dione(ester) (Mitomycin C)	A122	*	SV	HPLC
5-(Aminomethyl)-3-isoxazolol	A121	8250	SV	GC/MS
Amitrole	A121	8250	SV	GC/MS
Aniline	A121	8250	SV	GC/MS
Antimony and compounds, N.O.S.	A221	7040, 7041	M	AAS
Aramite	A121	8250	SV	GC/MS
Arsenic and compounds, N.O.S.	A222	7060, 7061	M	AAS
Arsenic acid	A222	7060, 7061	M	AAS
Arsenic pentoxide	A222	7060, 7061	M	AAS
Arsenic trioxide	A222	7060, 7061	M	AAS
Auramine	A121	8250	SV	GC/MS
Azaserine	A123	*	SV	HPLC
Barium and compounds, N.O.S.	A223	7080, 7081	M	ICAP, AAS
Barium cyanide	A223	7080	M	ICAP, AAS
	A252	9010	CN	T, C
Benz(c)acridine	A121	8250	SV	GC/MS
Benz(c)anthracene	A121	8100, 8250, 8310	SV	GC, GC/MS
Benzene	A101	8020, 8240	V	GC, GC/MS
Benzene, 2-amino-1-methyl	—	*		
Benzene, 4-amino-1-methyl	—	*		
Benzenearsonic acid	A222	7060, 7061	M	AAS
Benzene, dichloromethyl-	A121	8120, 8250	SV	GC, GC/MS
Benzenethiol	A121	8250	SV	GC/MS
Benidine	A121	8250	SV	GC/MS
Benzo(b)fluoranthene	A121	8100, 8250, 8310	SV	GC, GC/MS
Benzo(j)fluoranthene	A121	8100, 8250, 8310	SV	GC, GC/MS
Benzo(a)pyrene	A121	8100, 8250, 8310	SV	GC, GC/MS
p-Benzoquinone	A121	8250	SV	GC/MS
Benzotrithloride	A121	8120, 8250	SV	GC, GC/MS
Benzyl chloride	A121	8010, 8120, 8250	SV	GC, GC/MS
Beryllium and compounds, N.O.S.	A224	—	M	ICAP, AAS

Analysis Methods for Appendix VIII Hazardous Constituents (continued)

Compound	EPA-600/8-84-002 Method No. ^a	SW-846 Method No. ^b	Compound Type ^c	Analysis Method ^d
Bis(2-chloroethoxy)methane	A121	8010, 8240, 8250	SV	GC, GC/MS
Bis(2-chloroethyl)ether	A121	8010, 8240, 8250	SV	GC, GC/MS
N,N-Bis(2-chloroethyl)-2-naphthyl-amine	A121	*	SV	GC/MS
Bis(2-chloroisopropyl) ether	A121	8010, 8240, 8250	SV	GC, GC/MS
Bis(chloromethyl) ether	A121	8010, 8250	SV	GC, GC/MS
Bis(2-ethylhexyl) phthalate	A121	8060, 8250	SV	GC, GC/MS
Bromoacetone	A101	*	V	GC/MS
Bromomethane	A101	8010, 8240	V	GC, GC/MS
4-Bromophenyl phenyl ether	A121	8250	SV	GC/MS
Brucine	A148	8250	SV	GC/FID, HPLC
2-Butanone peroxide	A121	8250	SV	GC/MS
Butyl benzyl phthalide	A121	8060, 8250	SV	GC, GC/MS
2-sec-Butyl-4,6-dinitrophenol (DNBP)	A121	8040, 8250	SV	GC, GC/MS
Cadmium and compounds, N.O.S.	A225	7130, 7131	M	ICAP, AAS
Calcium chromate	A226	7190, 7191	M	ICAP, AAS
Calcium cyanide	A252	9010	CN	ICAP, T, C
Carbon disulfide	A101	8015, 8240	V	GC/MS
	A141	*	G	GC/TCD
Carbon oxyfluoride	A101	*	V	GC/MS
Chloral (as hydrate)	A131	8010, 8240	V	GC, GC/MS, HPLC
Chlorambucil	A122	*	SV	HPLC
Chlordane (α and γ isomers)	A121	8080, 8250	SV	GC, GC/MS
Chlorinated benzenes, N.O.S.	A101	8010, 8240	V	GC, GC/MS
	A121	8020, 8250	SV	GC, GC/MS
Chlorinated ethane, N.O.S.	A101	8010, 8240	V	GC, GC/MS
Chlorinated fluorocarbons, N.O.S.	A101	*	V	GC/MS
Chlorinated naphthalene, N.O.S.	A121	8120, 8250	SV	GC, GC/MS
Chlorinated phenol, N.O.S.	A121	8040, 8250	SV	GC, GC/MS
Chloroacetaldehyde	A131	8010, 8240	V	GC, GC/MS, HPLC
Chloroalkyl ethers, N.O.S.	A101	*	V	GC/MS
p-Chloroaniline	A121	*	SV	GC/MS
Chlorobenzene	A101	8020, 8240	V	GC, GC/MS
Chlorobenzilate	A121	*	SV	GC/MS
p-Chloro-m-cresol	A121	8040, 8250	SV	GC, GC/MS
	A122	*	SV	HPLC
1-Chloro-2,3-epoxypropane	A101	*	V	GC/MS
2-Chloroethyl vinyl ether	A101	8010, 8240	V	GC, GC/MS
Chloroform	A101	8010, 8240	V	GC, GC/MS
Chloromethane	A101	8010, 8240	V	GC, GC/MS
Chloromethyl methyl ether	A101	8010	V	GC, GC/MS
2-Chloronaphthalene	A121	8120, 8250	SV	GC, GC/MS
2-Chlorophenol	A121	8040, 8250	SV	GC, GC/MS
	A122	*	SV	HPLC
Chloroprene	—	*	SV	HPLC
1-(o-Chlorophenyl)thiourea	A123	*	SV	HPLC
3-Chloropropene	—	*	SV	HPLC
3-Chloropropionitrile	A121	8250	SV	GC/MS
Chromium and compounds, N.O.S.	A226	7190, 7191, 7195, 7196, 7197	M	ICAP, AAS
Chrysene	A121	8100, 8250, 8310	SV	GC, GC/MS
Citrus Red No. 2	A149	*	SV	HPLC
Coal tars	A121	*	SV	GC/MS
Copper cyanide	A252	9010	CN	T, C
Creosote	A121	8100, 8250	SV	GC, GC/MS
Cresols	A121	8040, 8250	SV	GC, GC/MS
Crotonaldehyde	A123	*	SV	HPLC
	A131	*	SV	GC/MS
Cyanides (soluble salts and complexes), N.O.S.	A252	9010	CN	T, C
Cyanogen	A138	9010	G	GC/TCD, T, C
Cyanogen bromide	A138	9010	G	GC/TCD, T, C
Cyanogen chloride	A138	9010	G	GC/TCD, T, C
Cycasin	A150	*	*	*
2-Cyclohexyl-4,6-dinitrophenol	A121	8040, 8250	SV	GC, GC/MS
Cyclophosphamide	—	—	—	—
Daunomycin	A122	*	SV	HPLC
DDD	A121	8080, 8250	SV	GC, GC/MS
DDE	A121	8080, 8250	SV	GC, GC/MS
DDT	A121	8080, 8250	SV	GC, GC/MS
Diallate	A121	*	SV	GC/MS

Analysis Methods for Appendix VIII Hazardous Constituents (continued)

Compound	EPA-600/8-84-002 Method No. ^a	SW-846 Method No. ^b	Compound Type ^c	Analysis Method ^d
Dibenz(a,h)acridine	A121	8100	SV	GC, GC/MS
Dibenz(a,i)acridine	A121	8100	SV	GC, GC/MS
Dibenz(a,h)anthracene	A121	8100, 8310, 8250	SV	GC, GC/MS
7H-Dibenzo(c,g)carbazole	A121	8100	SV	GC, GC/MS
Dibenzo(a,e)pyrene	A121	8100	SV	GC, GC/MS
Dibenzo(a,h)pyrene	A121	8100	SV	GC, GC/MS
Dibenzo(a,i)pyrene	A121	8100	SV	GC, GC/MS
1,2-Dibromo-3-chloropropane	A101	8010, 8240	V	GC, GC/MS
1,2-Dibromoethane	A101	8010, 8240	V	GC, GC/MS
Dibromomethane	A101	8010, 8240	V	GC, GC/MS
Di-n-butyl phthalate	A121	8060, 8250	SV	GC, GC/MS
Dichlorobenzene (meta, ortho and para isomers)	A101	8010, 8120	V	GC, GC/MS
	A121	8250	SV	GC/MS
Dichlorobenzene, N.O.S.	A101	8010, 8120	V	GC, GC/MS
	A121	8250	SV	GC/MS
3,3'-Dichlorobenzidine	A121	8250	SV	GC/MS
1,4-Dichloro-2-butene	A101	8010, 8240	V	GC, GC/MS
Dichlorodifluoromethane	A101	8010	V	GC, GC/MS
1,1-Dichloroethane	A101	8010, 8240	V	GC, GC/MS
1,2-Dichloroethane	A101	8010, 8240	V	GC, GC/MS
trans-1,2-Dichloroethene	A101	8010, 8240	V	GC, GC/MS
Dichloroethylene, N.O.S.	A101	8010	V	GC, GC/MS
1,1-Dichloroethylene	A101	8010	V	GC, GC/MS
Dichloromethane	A101	8010, 8240	V	GC, GC/MS
2,4-Dichlorophenol	A121	8040, 8250	SV	GC, GC/MS
	A122		SV	HPLC
2,6-Dichlorophenol	A121	8040, 8250	SV	GC, GC/MS
	A122		SV	HPLC
2,4-Dichlorophenoxyacetic acid	A122	8150, 8250	SV	GC, GC/MS, HPLC
	A133		SV	GC/MS
Dichlorophenylarsine	A222	7060, 7061	M	AAS
Dichloropropane, N.O.S.	A101	8010, 8240	V	GC, GC/MS
1,2-Dichloropropane	A101	8010, 8240	V	GC, GC/MS
Dichloropropanol, N.O.S.	A121	8120, 8250	SV	GC, GC/MS
Dichloropropene, N.O.S.	A101	8240	V	GC/MS
1,3-Dichloropropene	A101	8240	V	GC/MS
Dieldrin	A121	8080	SV	GC, GC/MS
1,2:3,4-Diepoxybutane	A121	*	SV	GC/MS
Diethylarsine	A222	7060, 7061	M	AAS
N,N-Diethylhydrazine	A121	*	SV	GC/MS
0,0-Diethyl S-methyl ester of phosphorodithioic acid	A121	8250	SV	GC/MS
0,0-Diethylphosphoric acid, 0-p-nitrophenyl ester	A121	8250	SV	GC/MS
Diethyl phthalate	A121	8060, 8250	SV	GC, GC/MS
0,0-Diethyl 0-2-pyrazinyl phosphorothioate	A121	8250	SV	GC/MS
Diethylstilbestrol	A123	*	SV	HPLC
Dihydrosafrole	A121	*	SV	GC/MS
3,4-Dihydroxy-α-(methylamino)methyl benzyl alcohol [Epinephrine]	A123	*	SV	HPLC
Diisopropylfluorophosphate (DFP)	A121	*	SV	GC/MS
Dimethoate	A121	8140	SV	GC, GC/MS
3,3'-Dimethoxybenzidine	A121	*	SV	GC/MS
p-Dimethylaminoazobenzene	A121	*	SV	GC/MS
7,12-Dimethylbenz(a)anthracene	A121	*	SV	GC/MS
3,3'-Dimethylbenzidine	A121	*	SV	GC/MS
Dimethylcarbamoyl chloride	A144	*		
1,1-Dimethylhydrazine	A121	*	SV	GC/MS
1,2-Dimethylhydrazine	A121	*	SV	GC/MS
3,3-Dimethyl-1-(methylthio)-2-butanone,0-((methylamino) carbonyl)oxime [Thiofanox]	A183	*	*	GC/FPD
α,α-Dimethylphenethylamine	A121	*	SV	GC/MS
2,4-Dimethylphenol	A121	8040, 8250	SV	GC, GC/MS
Dimethyl phthalate	A121	8060, 8250	SV	GC, GC/MS
Dimethyl sulfate	A121	8250	SV	GC/MS
Dinitrobenzene, N.O.S.	A121	8090, 8250	SV	GC, GC/MS
4,6-Dinitro-o-cresol (and salts)	A121	8040, 8250	SV	GC, GC/MS
	A122		SV	HPLC
2,4-Dinitrophenol	A121	8040, 8250	SV	GC, GC/MS
	A122		SV	HPLC
2,4-Dinitrotoluene	A121	8090, 8250	SV	GC, GC/MS
2,6-Dinitrotoluene	A121	8090, 8250	SV	GC, GC/MS

Analysis Methods for Appendix VIII Hazardous Constituents (continued)

Compound	EPA-600/8-84-002 Method No. ^a	SW-846 Method No. ^b	Compound Type ^c	Analysis Method ^d
Di-n-octyl phthalate	A121	8060, 8250	SV	GC, GC/MS
1,4-Dioxane	A101	*	V	GC/MS
Diphenylamine	A121	*	SV	GC/MS
1,2-Diphenylhydrazine	A121	*	SV	GC/MS
Di-n-propylnitrosamine	A121	*	SV	GC/MS
Disulfoton	A121	8140	SV	GC, GC/MS
2,4-Dithioburet	A121	*	SV	GC/MS
Endosulfan	A121	8080, 8250	SV	GC, GC/MS
Endrin (and metabolites)	A121	8080, 8250	SV	GC, GC/MS
Ethyl carbamate	A121	*	SV	GC/MS
Ethyl cyanide	A252	9010	CN	T, C
Ethylenebisdithiocarbamic acid (salts and esters)	—	*		
Ethyleneimine	A121	*	SV	GC/MS
Ethylene oxide	A156	*	V	GC/FID
Ethylenethiourea	A123	*	SV	HPLC
Ethyl methacrylate	A121	*	SV	GC/MS
Ethyl methanesulfonate	A121	*	SV	GC/MS
Fluoranthene	A121	8100, 8250, 8310	SV	GC, GC/MS
Fluorine	A137	—	G	*
2-Fluoroacetamide	A157	*	*	GC/FID
Fluoroacetic acid, sodium salt	A121	*	SV	GC/MS
Formaldehyde	A131	8015, 8240	V	GC, GC/MS, HPLC
Formic acid	A101	*	V	GC/MS
	A121	8250	SV	GC/MS
	A133	*	SV	GC/MS
Glycidylaldehyde	A131	*	SV	GC/MS, HPLC
Halomethane, N.O.S.	A101	*	V	GC/MS
Heptachlor	A121	8080, 8250	SV	GC, GC/MS
Heptachlor epoxide (α, β, and γ isomers)	A121	8080, 8250	SV	GC, GC/MS
Hexachlorobenzene	A121	8120, 8250	SV	GC, GC/MS
Hexachlorobutadiene	A121	8120, 8250	SV	GC, GC/MS
Hexachlorocyclohexane (all isomers)	A121	8120	SV	GC, GC/MS
Hexachlorocyclopentadiene	A121	8120, 8250	SV	GC, GC/MS
Hexachlorodibenzo-p-dioxins	—	8280	SV	GC/MS
Hexachlorodibenzofurans	—	8280	SV	GC/MS
Hexachloroethane	A101	8010, 8240	V	GC, GC/MS
	A121	8120, 8250	SV	GC, GC/MS
1,2,3,4,10,10-Hexachloro-1,4,4a,5,8,8a-hexahydro- 1,4:5,8-endo,endo-dimethanonaphthalene	A121	*	SV	GC/MS
Hexachlorophene	A121	*	SV	GC/MS
Hexachloropropene	A101	*	V	GC/MS
Hexaethyl tetraphosphate	A121	*	SV	GC/MS
Hydrazine	A101	*	V	GC/MS
	A141	*	G	GC/TCD
Hydrocyanic acid	A141	*	G	GC/TCD
	A251	*	AN	IC
Hydrofluoric acid	A251	*	AN	IC
Hydrogen sulfide	A141	*	G	GC/TCD
Hydroxydimethylarsine oxide	A222	7060, 7061	M	AAS
Indeno(1,2,3-c,d)pyrene	A121	8100, 8250, 8310	SV	GC, GC/MS
Iodomethane	A101	*	V	GC/MS
Iron dextran (complex)	—	*		
Isocyanic acid, methyl ester	A101	*	V	GC/MS
Isobutyl alcohol	A134	*	V	GC/MS
Isosafrole	A121	*	SV	GC/MS
Kepon	A121	8080	SV	GC, GC/MS
Lasiocarpine	A160	*	*	
Lead and compounds, N.O.S.	A227	7420, 7421	M	ICAP AAS
Lead acetate	A227	7420, 7421	M	ICAP AAS
Lead phosphate	A227	7420, 7421	M	ICAP AAS
Lead subacetate	A227	7420, 7421	M	ICAP AAS
Maleic anhydride	A121	8250	SV	GC/MS
Maleic hydrazide	A121	*	SV	GC/MS
Malononitrile	A121	*	SV	GC/MS
Melphalan	A122	*	SV	HPLC
Mercury fulminate	A228	7470, 7471	M	CV/AAS
Mercury and compounds, N.O.S.	A228	7470, 7471	M	CV/AAS
Methacrylonitrile	A121	*	SV	GC/MS
Methanethiol	A101	*	V	GC/MS
Methapyrilline	A121	*	SV	GC/MS

Analysis Methods for Appendix VIII Hazardous Constituents (continued)

Compound	EPA-600/8-84-002 Method No. ^a	SW-846 Method No. ^b	Compound Type ^c	Analysis Method ^d
Metholmyl	A122	8250	SV	GC, GC/MS, HPLC
Methoxychlor	A121	8080	SV	GC, GC/MS
2-Methylaziridine	A121	*	SV	GC/MS
3-Methylcholanthrene	A121	8100	SV	GC, GC/MS
Methylchlorocarbonate	—	*		
4,4'-Methylenebis(2-chloroaniline)	A121	*	SV	GC/MS
Methyl ethyl ketone (MEK)	A101	8015, 8240	V	GC, GC/MS
	A121	*	SV	GC/MS
Methyl hydrazine	A101	*	V	GC/MS
	A121	*	SV	GC/MS
2-Methylacetonitrile	A121	*	SV	GC/MS
Methyl methacrylate	A121	*	SV	GC/MS
Methyl methanesulfonate	A121	*	SV	GC/MS
2-Methyl-2-(methylthio)propionaldehyde-0- (methylcarbonyl)oxime	A183	*	*	GC/FPD
N-Methyl-N'-nitro-N-guanidine	A121	*	SV	GC/MS
Methyl parathion	A121	8140	SV	GC, GC/MS
Methylthiouracil	A121	*	SV	GC/MS
Mustard gas	A139	*	V	GC/FPD
Naphthalene	A121	8100, 8250, 8310	SV	GC, GC/MS
1,4-Naphthoquinone	A121	8090, 8250	SV	GC, GC/MS
1-Naphthylamine	A121	*	SV	GC/MS
2-Naphthylamine	A121	*	SV	GC/MS
1-Naphthyl-2-thiourea	A123	*	SV	HPLC
Nickel and compounds, N.O.S.	A229	7520, 7521	M	ICAP, AAS
Nickel carbonyl	A229	7520, 7521	M	ICAP, AAS
Nickel cyanide	A229	7520, 7521	M	ICAP, AAS
	A252	9010	CN	T, C
Nicotine (and salts)	A121	*	SV	GC/MS
Nitric oxide	A141	*	G	GC/TCD
p-Nitroaniline	A121	*	SV	GC/MS
Nitrobenzene	A121	8090, 8250	SV	GC, GC/MS
Nitrogen dioxide	A141	*	G	GC/TCD
Nitrogen mustard (and hydrochloride salt)	A139	*	V	GC/FPD
Nitrogen mustard N-Oxide (and hydrochloride salt)	A139	*	V	GC/FPD
Nitroglycerine	A121	*	SV	GC/MS
4-Nitrophenol	A121	8040, 8240	SV	GC, GC/MS
	A122	8250	SV	GC/MS, HPLC
4-Nitroquinoline-1-oxide	—	*		
Nitrosamine, N.O.S.	A121	8250	SV	GC/MS
N-Nitrosodi-n-butylamine	A121	8250	SV	GC/MS
N-Nitrosodiethanolamine	A121	8250	SV	GC/MS
N-Nitrosodiethylamine	A121	8250	SV	GC/MS
N-Nitrosodimethylamine	A121	8250	SV	GC/MS
N-Nitroso-N-ethylurea	A121	8250	SV	GC/MS
N-Nitrosomethylethylamine	A121	8250	SV	GC/MS
N-Nitroso-N-methylurea	A121	8250	SV	GC/MS
N-Nitroso-N-methylurethane	A121	8250	SV	GC/MS
N-Nitrosomethylvinylamine	A121	8250	SV	GC/MS
N-Nitrosomorpholine	A121	8250	SV	GC/MS
N-Nitrosornicotine	A121	8250	SV	GC/MS
N-Nitrosopiperidine	A121	8250	SV	GC/MS
N-Nitrosopyrrolidine	A121	8250	SV	GC/MS
N-Nitrososarcosine	A121	8250	SV	GC/MS
5-Nitro-o-toluidine	A122	*	SV	HPLC
Octamethylpyrophosphoramidate	A121	*	SV	GC/MS
Osmium tetroxide	A230	*	M	ICAP, AAS
7-Oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid Paraldehyde	A133	*	SV	GC/MS
	A131	8015, 8240	V	GC, GC/MS, HPLC
Parathion	A121	8140	SV	GC, GC/MS
Pentachlorobenzene	A121	*	SV	GC/MS
Pentachlorodibenzo-p-dioxins	—	8280	SV	GC/MS
Pentachlorodibenzofurans	—	8280	SV	GC/MS
Pentachloroethane	A121	*	SV	GC/MS
Pentachloronitrobenzene (PCNB)	A121	*	SV	GC/MS
Pentachlorophenol	A121	8040, 8250	SV	GC, GC/MS
Phenacetin	A174	*	SV	HPLC

Analysis Methods for Appendix VIII Hazardous Constituents (continued)

Compound	EPA-600/8-84-002 Method No. ^a	SW-846 Method No. ^b	Compound Type ^c	Analysis Method ^d
Phenol	A121	8040, 8250	SV	GC, GC/MS
	A122		SV	HPLC
Phenylenediamine	A121	*	SV	GC/MS
Phenylmercury acetate	A228	7470, 7471	M	CV/AAS
N-Phenylthiourea	A123	*	SV	HPLC
Phosgene	A138	*	G	GC/TCD
Phosphine	A136	*	V	GC/FPD
Phosphorodithioic acid, O,O-diethyl S-(ethylthio)methyl ester [Phorate]	A121	8140	SV	GC, GC/MS
Phosphorothioic acid, O,O-dimethyl O-(p-((dimethylamino)sulfonyl) phenyl) ester [Famphur]	A121	8140	SV	GC, GC/MS
Phthalic acid esters, N.O.S.	A121	8060	SV	GC, GC/MS
Phthalic anhydride	A121	8090, 8250	SV	GC, GC/MS
2-Picoline	A121	8090, 8250	SV	GC, GC/MS
Polychlorinated biphenyl, N.O.S.	A121	8080, 8250	SV	GC, GC/MS
Potassium cyanide	A252	9010	CN	T, C
Potassium silver cyanide	A232	7760, 7761	M	ICAP, AAS
	A252	9010	CN	T, C
Pronamide	A121	*	SV	GC/MS
1,3-Propane sulfone	A121	*	SV	GC/MS
n-Propylamine	A121	*	SV	GC/MS
Propylthiouracil	A121	*	SV	GC/MS
2-Propyn-1-ol	A134	*	V	GC/FID, GC/MS
Pyridine	A121	8090, 8250	SV	GC, GC/MS
Reserpine	A122	*	SV	HPLC
Resorcinol	A134	*	SV	GC/FID, GC/MS
Saccharin (and salts)	A121	*	SV	GC/MS
	A123		SV	HPLC
Safrole	A121	*	SV	GC/MS
Selenious acid	A231	7740, 7741	M	AAS
Selenium and compounds, N.O.S.	A231	7740, 7741	M	AAS
Selenium sulfide	A231	7740, 7741	M	AAS
Selenourea	A231	7740, 7741	M	AAS
Silver and compounds, N.O.S.	A232	7760, 7761	M	ICAP, AAS
Silver cyanide	A232	7760, 7761	M	ICAP, AAS
	A252	9010	CN	T, C
Sodium cyanide	A252	9010	CN	T, C
Streptozotocin	A122	*	SV	HPLC
Strontium sulfide	A233	*	M	ICAP, AAS
Strychnine (and salts)	A180	*	SV	HPLC
1,2,4,5-Tetrachlorobenzene	A121	8120, 8250	SV	GC, GC/MS
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	A121	8280	SV	GC/MS
Tetrachlorodibenzo-p-dioxins	—	8280	SV	GC/MS
Tetrachlorodibenzofurans	—	8280	SV	GC/MS
Tetrachloroethane, N.O.S.	A101	8010, 8240	V	GC, GC/MS
1,1,1,2-Tetrachloroethane	A101	8010, 8240	V	GC, GC/MS
1,1,2,2-Tetrachloroethane	A101	8010, 8240	V	GC, GC/MS
Tetrachloroethylene	A101	8010, 8240	V	GC, GC/MS
Tetrachloromethane	A101	8010, 8240	V	GC, GC/MS
2,3,4,6-Tetrachlorophenol	A121	8040 8250	SV	GC, GC/MS
	A122		SV	HPLC
Tetraethyldithiopyrophosphate	A121	*	SV	GC/MS
Tetraethyl lead	A227	7420, 7421	M	ICAP, AAS
Tetraethylpyrophosphate	A121	*	SV	GC/MS
Tetranitromethane	A101	*	V	GC/MS
Thallium and compounds, N.O.S.	A234	—	M	ICAP, AAS
Thallic oxide	A234	—	M	ICAP, AAS
Thallium(I) acetate	A234	—	M	ICAP, AAS
Thallium(I) carbonate	A234	—	M	ICAP, AAS
Thallium(I) chloride	A234	—	M	ICAP, AAS
Thallium(I) nitrate	A234	—	M	ICAP, AAS
Thallium selenite	A234	—	M	ICAP, AAS
Thallium(I) sulfate	A234	—	M	ICAP, AAS
Thioacetamide	A123	*	SV	HPLC
Thiosemicarbazide	A123	*	SV	HPLC
Thiourea	A123	*	SV	HPLC
Thiuram	A122	*	SV	HPLC
Toluene	A101	8020, 8240	V	GC, GC/MS
Toluenediamine, N.O.S.	A121	8250	SV	GC/MS
2,4-Toluenediamine	A121	8250	SV	GC/MS
2,6-Toluenediamine	A121	8250	SV	GC/MS

Analysis Methods for Appendix VIII Hazardous Constituents (continued)

Compound	EPA-600/8-84-002 Method No. ^a	SW-846 Method No. ^b	Compound Type ^c	Analysis Method ^d
3,4-Toluenediamine	A121	8250	SV	GC/MS
o-Toluidine hydrochloride	—	*		
Tolylene diisocyanate	A121	8250	SV	GC/MS
Toxaphene	A121	8080, 8250	SV	GC, GC/MS
Tribromomethane	A101	8010, 8240	V	GC, GC/MS
1,2,4-Trichlorobenzene	A121	8120, 8250	SV	GC, GC/MS
1,1,1-Trichloroethane	A101	8010, 8240	V	GC, GC/MS
1,1,2-Trichloroethane	A101	8010, 8240	V	GC, GC/MS
Trichloroethene	A101	8010, 8240	V	GC, GC/MS
Trichloromethanethiol	A121	*	SV	GC/MS
Trichloromonofluoromethane	A101	8010, 8240	V	GC, GC/MS
2,4,5-Trichlorophenol	A121	8040, 8250	SV	GC, GC/MS
	A122		SV	HPLC
2,4,6-Trichlorophenol	A121	8040, 8250	SV	GC, GC/MS
	A122		SV	HPLC
2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)	A122	8150, 8250	SV	GC, GC/MS
	A133		SV	GC/MS
2,4,5-Trichlorophenoxypropionic acid (2,4,5-TP) (Silvex)	A122	8150, 8250	SV	GC, GC/MS
	A133		SV	GC/MS
Trichloropropane, N.O.S.	A101	8010, 8240	V	GC, GC/MS
1,2,3-Trichloropropane	A101	8010, 8240	V	GC, GC/MS
0,0,0-Triethyl phosphorothioate	A121	*	SV	GC/MS
sym-Trinitrobenzene	A121	*	SV	GC/MS
Tris-(1-aziridinyl)phosphine sulfide	A190	*	V	GC/FPD
Tris(2,3-dibromopropyl) phosphate	A121	*	SV	GC/MS
Trypan blue	A123	*	SV	HPLC
Uracil mustard	—	*		
Vanadic acid, ammonium salt	A235	*	M	ICAP, AAS
Vanadium pentoxide	A235	*	M	ICAP, AAS
Vinyl chloride	A101	8010, 8240	V	GC, GC/MS
Zinc cyanide	A252	9010	CN	T, C
Zinc phosphide	A253	—	S	GC/FPD

^a Source: Arthur D. Little, Inc., "Sampling and Analysis Methods for Hazardous Waste Combustion," EPA-600/8-84-002, PB84-155845, February 1984.

— = No method given.

^b Sources: (1) "Test Methods for Evaluating Solid Waste — Physical/Chemical Methods," EPA/OSW, Third Edition, SW-846, November 1986.
(2) 40 CFR Part 261, Appendix III.

(3) All VOST tube analysis must be by Method 5040. Other methods are applicable to other matrices.

* = Method not specified for the substance in SW-846, but SW-846 methods may apply.

— = No method given.

^c V = Volatile substance

SV = Semivolatile substance

M = Metal

CN = Cyanide

S = Solid

AN = Anion

G = Gas

* = Not found

^d GC = Gas chromatography

GC/MS = Gas chromatography/mass spectrometry

GC/FID = Gas chromatography with flame ionization detector

GC/TCD = Gas chromatography with thermal conductivity detector

GC/FPD = Gas chromatography with flame photometric detector

HPLC = High performance liquid chromatography

AAS = Atomic absorption spectroscopy

CV/AAS = Cold vapor/atomic absorption spectroscopy

ICAP = Inductively coupled argon plasma emission spectroscopy

IC = Ion chromatography

C = Colorimetry

T = Titration

— = No method found

* = No method available

Appendix B

Measurement Checklists

B.1 Introduction

Checklists have been prepared to assist the permit writer in determining the completeness of a trial burn plan and permit application relative to process monitoring, sampling, and analysis of samples. These checklists show the parameters required by regulation or typically recommended for measurement during a trial burn and the essential information which should be given in the trial burn plan for the measurement, sampling, or analysis of each parameter. Table B-1 is the checklist for the monitoring parameters, Table B-2 is for the sampling parameters, and Table B-3 is for the analysis of samples.

One important aspect of any trial burn plan or permit application is the description of the QA/QC procedures. A checklist of the QA/QC items which should be addressed in the project Quality Assurance (QA) plan for each of the monitoring, sampling, and analysis parameters is given in Table B-4.

The checklists should be filled in by the permit writer as the trial burn plan and permit application are reviewed. In each column indicate Y for yes, N for no, I for incomplete, or NA for not applicable. The blanks which have an N or I result in an incomplete trial burn plan and permit application and should be subsequently addressed to complete the documents. The checklists address only information related to measurements; they do not include other information required for complete applications and trial burn plans.

The following discussion provides a description of each checklist, followed by the checklists.

B.2 Process Monitoring

The process monitoring checklist (Table B-1) contains both parameters that must be monitored *continuously* during the trial burn and subsequent operation of the incinerator, as provided in the *Code of Federal Regulations*, 40 CFR Parts 264.345 and 264.347, and others typically recommended for measurement during trial burns. Parameters on the checklist are (1) waste feed rate, (2) combustion temperature, (3) a measure of combustion gas velocity, (4) combustion chamber pressure, (5) carbon monoxide concentration (CO) in the stack gases, (6) oxygen concentration in the stack gases, (7) the waste feed pressure, (8) auxiliary fuel flow rate and pressure, (9) waste atomization airflow rate and pressure,

(10) input quench water flow rate, (11) input and output scrubber water flow rates and output pH, (12) other air pollution control device(s) parameters, (13) waste feed cutoff system parameters, and (14) other parameters as appropriate.

The essential information which should be included in the trial burn plan and permit application for each selected process monitoring parameter is shown in the columns in Table B-1. This information includes (1) whether or not the parameter will be monitored, (2) the monitoring method, (3) monitoring equipment, (4) monitoring location, (5) monitoring frequency, and (6) method of data recording. These subjects should be addressed in the trial burn plan and permit application. Other information such as methods of data reduction and storage, calibration of equipment, and inspection and maintenance of the equipment should be given in the project QA plan.

B.3 Sampling

The sampling checklist (Table B-2) contains a list of samples which may be taken during the trial burn and analyzed in accordance with 40 CFR Parts 264.341, 264.342, 264.343, 264.345, 270.19, and 270.62.⁷ The samples required by regulation are (1) waste feed, (2) stack gases, (3) scrubber water, (4) ash, and (5) other samples, as appropriate. Collection of auxiliary fuel and scrubber inlet samples are optional. None of these samples are required during the subsequent operation of the incinerator unless specified in the permit.

The essential information which should be included in the trial burn plan for each sampling parameter is shown in the columns of Table B-2. This information includes (1) whether or not the sample(s) will be taken, (2) the sampling method, (3) sampling equipment, (4) sampling location, (5) sampling frequency, and (6) method of data recording. These subjects should be addressed in the trial burn plan. Other information such as calibration and maintenance of the equipment, data reduction, and data storage should be given in the project QA plan.

B.4 Analysis

The checklist for analysis of the samples (Table B-3) contains parameters which may be evaluated for the trial burn, in accordance with 40 CFR Parts 270.19 and

270.62.⁷ Some of the analyses including the principal organic hazardous constituents (POHCs) in the auxiliary fuel and metals may be appropriate only in some cases.

Essential information which should be included in the trial burn plan for each analytical parameter is shown in the columns in Table B-3. This information includes (1) whether or not the sample(s) will be analyzed, (2) analytical procedure, (3) analytical equipment, (4) sample traceability, and (5) data recording. These subjects should be addressed in the trial burn plan. Other information such as calibration and maintenance of equipment, data reduction and validation, data reporting, and data storage should be given in the project QA plan.

B.5 Quality Assurance/Quality Control

Each permit application and trial burn plan must have a quality assurance (QA) plan. The QA plan must address all data-gathering activities (e.g., process monitors as well as sampling and analytical activities). This plan should conform to the specifications established in

SW-846, *Test Methods for Evaluating Solid Waste — Physical/Chemical Methods*,⁸ and *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*,¹⁷ and must address all measurement parameters.

The purpose of the QA plan is to establish a specific program to (1) help ensure that the monitoring data, sampling, and analytical activities meet specific quality objectives; and (b) routinely assess the quality of the data. The QA/QC checklist (Table B-4) includes all of the monitoring, sampling, and analytical parameters given in the previous three checklists and shows the QA/QC items required for each parameter in the *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*.¹⁷ Each of the QA/QC items is discussed in Section 4.0.

It should be noted that there is redundancy among the information required in Tables B-1 to B-4. This information may be provided in either the trial burn plan and permit application or the QA plan, or both. QA/QC information provided in the trial burn plan should be referenced in the QA plan.

Table B-1. Checklist for Process Monitoring Parameters for RCRA Incinerators

Monitoring parameter	To be monitored*	Monitoring method	Equipment	Monitoring location	Monitoring frequency	Data recording
Waste feed rate ^a						
Stream No. 1						
Stream No. 2						
Stream No. 3						
Combustion temperature(s) ^a						
Combustion gas velocity indicator ^a						
Combustion chamber pressure						
Primary						
Secondary						
CO in stack gases ^a						
Oxygen in stack gases						
Waste feed pressure(s)						
Auxiliary fuel						
Feed rate						
Pressure						
Waste atomization						
Airflow						
Pressure						
Quench water						
Input flow rate						
Scrubber water						
Input flow rate						
Output flow rate						
Output pH						
Air pollution control device(s) parameters						
Waste feed cutoff system						
Other						

* Indicate Y = Yes; N = No; I = Incomplete; NA = Not applicable.

^a Continuous monitoring specifically required by RCRA regulations.

Table B-2. Checklist for Sampling Parameters for RCRA Incinerators

Sampling parameter	To be sampled*	Sampling method	Equipment	Sampling location	Sampling frequency	Data recording
Waste feed						
Stream No. 1						
Stream No. 2						
Stream No. 3						
Stack gases						
Particulates						
HCl						
H ₂ O						
POHCs						
O ₂						
CO ₂						
Stack gas flow rate						
Stack gas temperature						
Auxiliary fuel						
Quench water						
Inlet						
Scrubber water						
Inlet						
Outlet						
Ash						
Other						

* Indicate Y = Yes; N = No; I = Incomplete; NA = Not applicable.

Table B-3. Checklist for Analysis of Samples for RCRA Incinerators

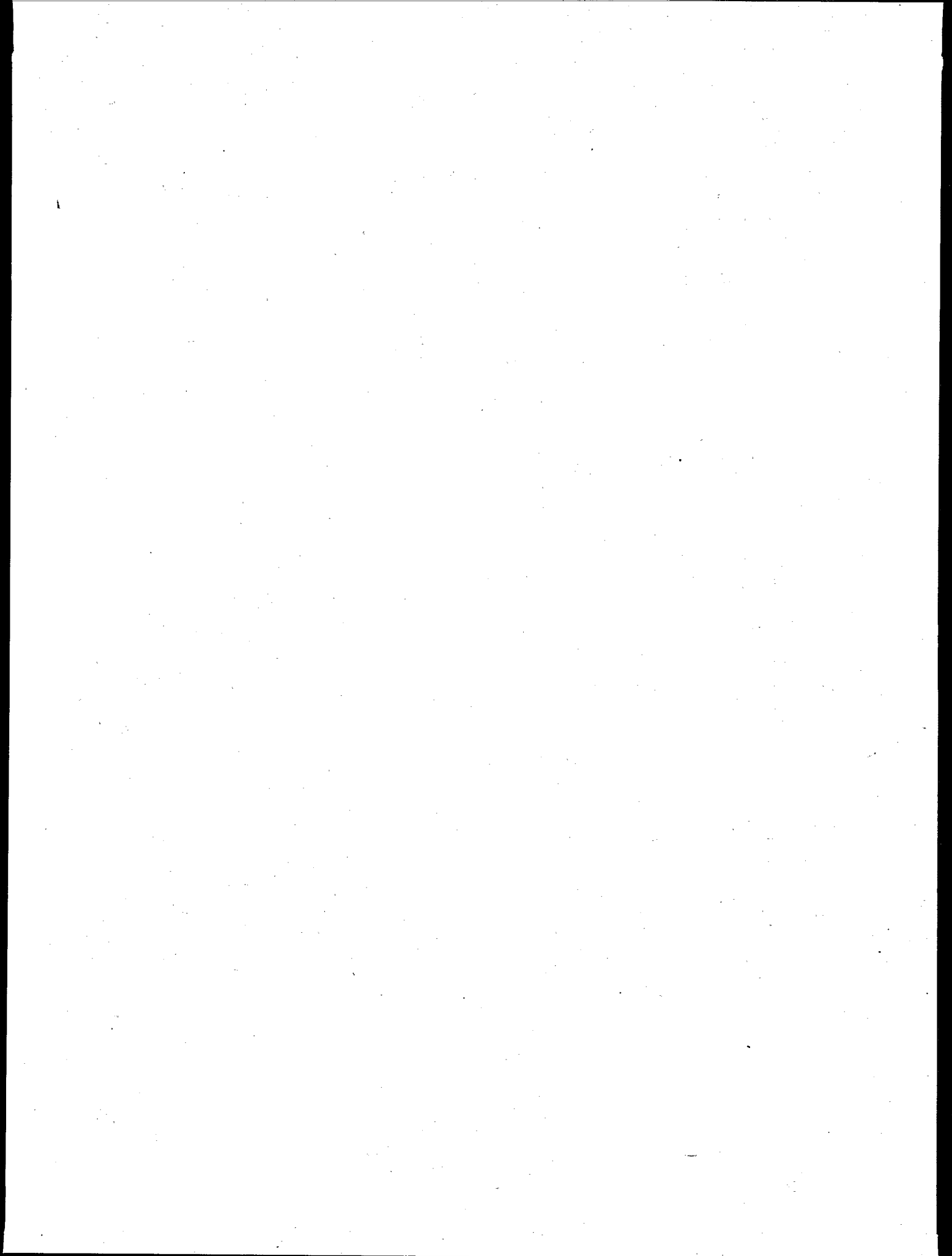
Analytical parameter	To be analyzed*	Procedure	Equipment	Sample traceability	Data recording
Waste feed analysis					
High heating value					
Chlorine					
POHCs					
Ash					
Viscosity					
H ₂ O					
Metals					
Volatile matter					
Stack samples					
Particulates					
HCl					
POHCs					
O ₂					
CO ₂					
Metals					
Auxiliary fuel					
POHCs					
HHV					
Chlorine					
Ash					
Quench water input					
POHCs					
pH					
Scrubber water input					
POHCs					
pH					
Scrubber water output					
POHCs					
pH					
Ash residue					
POHCs					
Heavy metals					
TCLP					
Other					

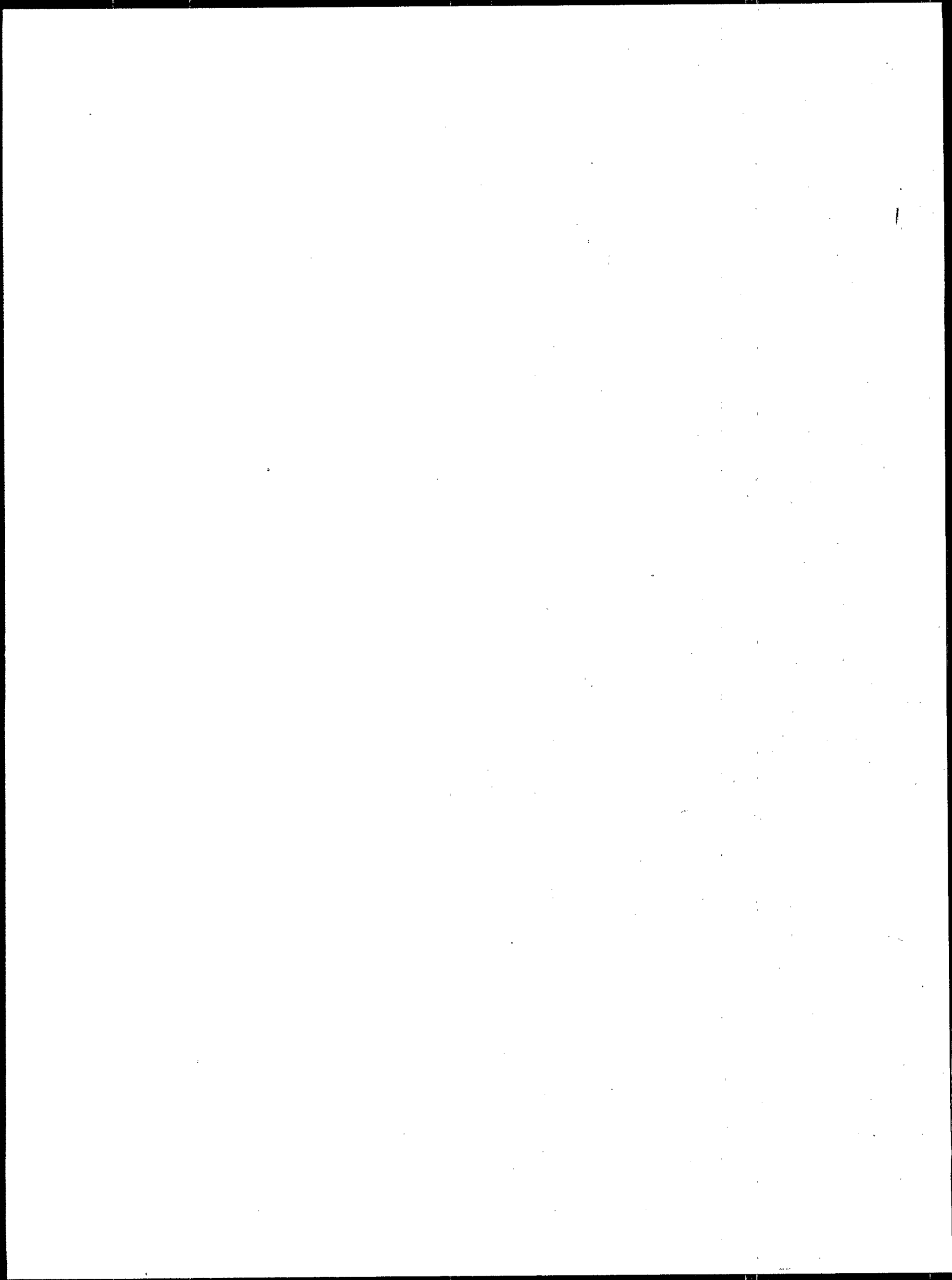
* Indicate Y = Yes; N = No; I = Incomplete; NA = Not applicable.

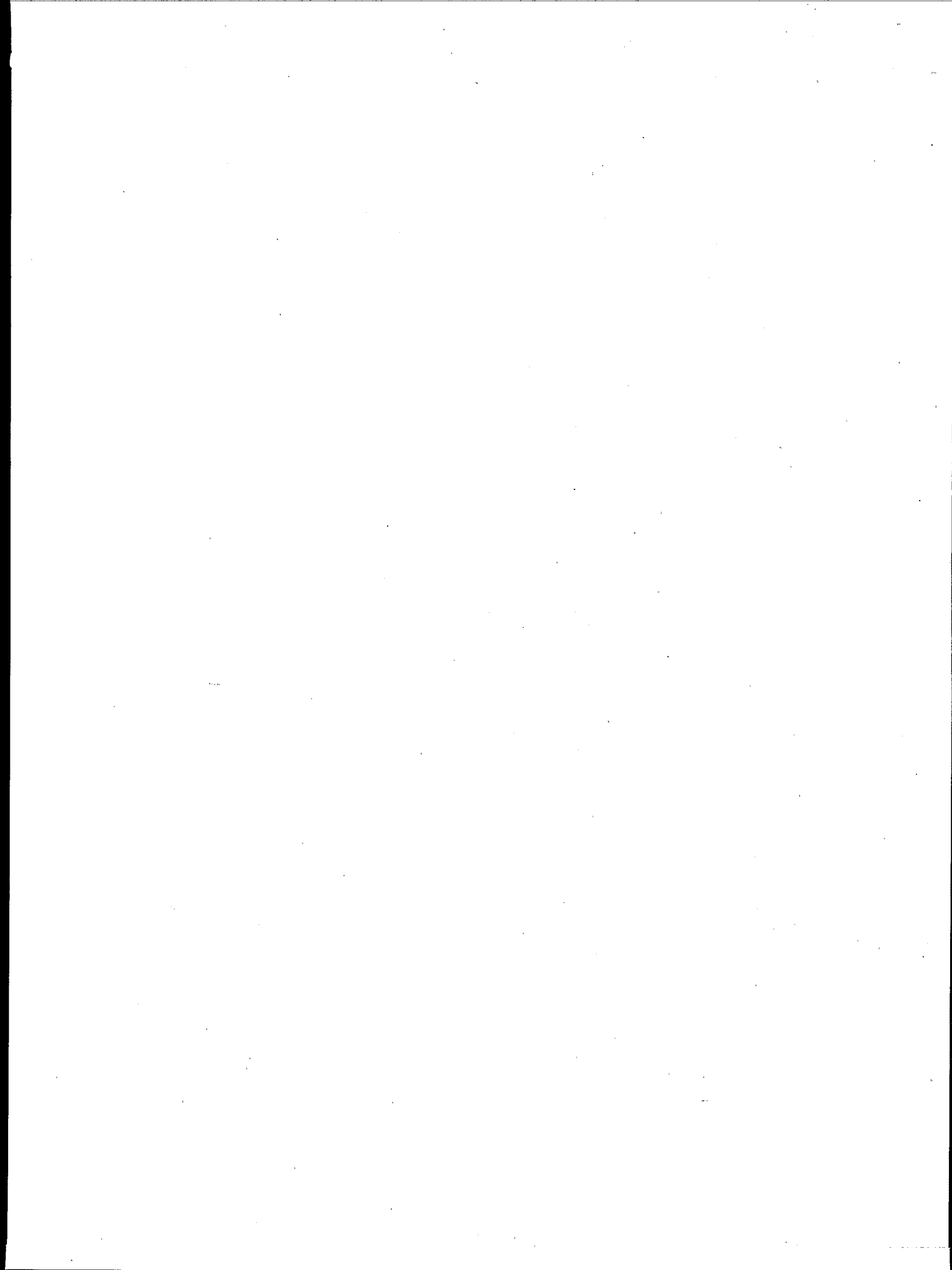
Table B-4. Quality Assurance/Quality Control Checklist for Monitoring, Sampling, and Analysis Parameters for Incinerators

Monitoring, sampling, and analysis parameters	Quality assurance/quality control items*							Precision/ accuracy	Representativeness
	Method/ procedure	Sample traceability	Equipment calibration	Equipment maintenance	Data recording	Data reporting	Internal QC checks		
Monitoring									
Waste feed rate	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Combustion temperature	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Combustion gas velocity	_____	N/A	_____	_____	_____	_____	_____	_____	_____
CO in stack gas	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Auxiliary fuel	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Atomization pressure	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Combustion chamber pressure	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Quench water	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Scrubber water	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Air pollution control devices	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Waste feed cutoff system	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Other	_____	N/A	_____	_____	_____	_____	_____	_____	_____
Sampling									
Waste feed sampling	_____	_____	_____	_____	_____	_____	_____	_____	_____
Stack gas sampling	_____	_____	_____	_____	_____	_____	_____	_____	_____
Auxiliary fuel	_____	_____	_____	_____	_____	_____	_____	_____	_____
Scrubber water	_____	_____	_____	_____	_____	_____	_____	_____	_____
Ash sampling	_____	_____	_____	_____	_____	_____	_____	_____	_____
Other	_____	_____	_____	_____	_____	_____	_____	_____	_____
Analysis									
Waste feed analysis	_____	_____	_____	_____	_____	_____	_____	_____	_____
High heating value	_____	_____	_____	_____	_____	_____	_____	_____	_____
Chlorides	_____	_____	_____	_____	_____	_____	_____	_____	_____
POHCs	_____	_____	_____	_____	_____	_____	_____	_____	_____
Ash	_____	_____	_____	_____	_____	_____	_____	_____	_____
Viscosity	_____	_____	_____	_____	_____	_____	_____	_____	_____
Stack samples	_____	_____	_____	_____	_____	_____	_____	_____	_____
Particulates	_____	_____	_____	_____	_____	_____	_____	_____	_____
HCl	_____	_____	_____	_____	_____	_____	_____	_____	_____
O ₂	_____	_____	_____	_____	_____	_____	_____	_____	_____
CO ₂	_____	_____	_____	_____	_____	_____	_____	_____	_____
POHCs	_____	_____	_____	_____	_____	_____	_____	_____	_____
Auxiliary fuel POHCs	_____	_____	_____	_____	_____	_____	_____	_____	_____
Scrubber water	_____	_____	_____	_____	_____	_____	_____	_____	_____
POHCs	_____	_____	_____	_____	_____	_____	_____	_____	_____
pH	_____	_____	_____	_____	_____	_____	_____	_____	_____
Ash residue	_____	_____	_____	_____	_____	_____	_____	_____	_____
POHCs	_____	_____	_____	_____	_____	_____	_____	_____	_____
Heavy metals	_____	_____	_____	_____	_____	_____	_____	_____	_____
Other	_____	_____	_____	_____	_____	_____	_____	_____	_____
General QA/QC									
Project description	_____	_____	_____	_____	_____	_____	_____	_____	_____
Project organization and responsibility	_____	_____	_____	_____	_____	_____	_____	_____	_____
Organization chart	_____	_____	_____	_____	_____	_____	_____	_____	_____
Resumes of key individuals	_____	_____	_____	_____	_____	_____	_____	_____	_____
Description of individual responsibilities	_____	_____	_____	_____	_____	_____	_____	_____	_____
Performance and system audits	_____	_____	_____	_____	_____	_____	_____	_____	_____
Internal performance audits	_____	_____	_____	_____	_____	_____	_____	_____	_____
Internal systems audits	_____	_____	_____	_____	_____	_____	_____	_____	_____
External performance audits	_____	_____	_____	_____	_____	_____	_____	_____	_____
External systems audits	_____	_____	_____	_____	_____	_____	_____	_____	_____
Corrective action	_____	_____	_____	_____	_____	_____	_____	_____	_____
QA reports to management	_____	_____	_____	_____	_____	_____	_____	_____	_____
Types of reports	_____	_____	_____	_____	_____	_____	_____	_____	_____
Individual(s) responsible	_____	_____	_____	_____	_____	_____	_____	_____	_____
Frequency	_____	_____	_____	_____	_____	_____	_____	_____	_____

* Indicate Y = Yes; N = No; I = Incomplete; NA = Not applicable.







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