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PRACTICAL GUIDE - TRIAL BURNS FOR HAZARDOUS  
WASTE INCINERATORS

by

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## NOTICE

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

## FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of solid and hazardous wastes. These materials, if improperly dealt with, can threaten both public health and the environment. Abandoned waste sites and accidental releases of toxic and hazardous substances to the environment also have important environmental and public health implications. The Hazardous Waste Engineering Research Laboratory assists in providing an authoritative and defensible engineering basis for assessing and solving these problems. Its products support the policies, programs, and regulations of the Environmental Protection Agency, the permitting and other responsibilities of State and local governments and the need of both large and small businesses in handling their wastes responsibly and economically.

The manual concentrates on those aspects of a trial burn that are the most important and those that are potentially troublesome. The manual contains practical explanations based on experience of Midwest Research Institute (MRI) and others in conducting trial burns and related tests for EPA. It includes the comments of several industrial plant owners and operators. It is directed mainly to incinerator operators, those who may conduct the actual sampling and analysis, and those who must interpret trial burn results. It will also be useful for regulatory personnel and others that need to understand trial burns.

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## ABSTRACT

The manual concentrates on those aspects of a trial burn that are the most important and those that are potentially troublesome. The manual contains practical explanations based on experience of Midwest Research Institute (MRI) and others in conducting trial burns and related tests for EPA. It includes the comments of several industrial plant owners and operators. It is directed mainly to incinerator operators, those who may conduct the actual sampling and analysis, and those who must interpret trial burn results. It will also be useful for regulatory personnel and others that need to understand trial burns. Potential trouble spots that have been encountered are: (1) trial burns frequently take more time and effort than an operator anticipates; and (2) failure to meet the trial burn requirements.

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## SECTION I

### INTRODUCTION

On May 19, 1980, the U.S. Environmental Protection Agency (EPA) published regulations under the authority of the Resource Conservation and Recovery Act (RCRA) for hazardous waste incinerators. These regulations require that new and existing incinerators adequately destroy hazardous organic compounds and maintain acceptable levels of particulate and chloride emissions. Owners and operators of incinerators are required to demonstrate the performance of the facility by means of a trial burn. As a consequence, industry and control agency personnel have become involved in planning for, conducting, and interpreting the results from trial burns. This manual is written to assist those individuals in their efforts.

The manual concentrates on those aspects of a trial burn that are the most important and those that are potentially troublesome. The manual contains practical explanations based on experience of Midwest Research Institute (MRI) and others in conducting trial burns and related tests for EPA. It includes the comments of several industrial plant owners and operators. It is directed mainly to incinerator operators, those who may conduct the actual sampling and analysis, and those who must interpret trial burn results. It will also be useful for regulatory personnel and others that need to understand trial burns.

One of the major objectives was to make this Guide readily usable. For that reason, the discussion is brief and avoids dwelling on detail. A question and answer format is used to relate the material to operator concerns. Each subsection begins as a question that could well be posed by an incinerator operator who needs to conduct a trial burn. The narrative following each question provides answers to the question or provides information pertinent to the question. For each question, the most important considerations are discussed, and potential trouble spots are identified.

This Guide addresses multiple components of the trial burn process including planning and preparation, sampling and analysis for the trial burn, process monitoring during the trial burn, and data reduction and reporting. The Guide does not directly address the preparation of the Trial Burn Plan, but it does address some planning aspects that affect Trial Burn Plan preparation and subsequent interpretation of the trial burn results.

The remainder of the Guide is divided into three sections. Section II presents an overview of the trial burn process and requirements. Section III discusses planning for the trial burn. Section IV discusses conducting the trial burn and reducing and reporting data from the trial burn.

## SECTION II

### OVERVIEW OF A TRIAL BURN

This section summarizes different aspects of the trial burn. It describes the trial burn process and requirements for the trial burn. Basic information is provided to help answer four questions:

- A. What does a trial burn involve?
- B. What types of sampling and analyses are typically involved?
- C. What skills, equipment and facilities are needed to conduct the trial burn?
- D. What are the major cost factors associated with a trial burn?

#### A. WHAT DOES A TRIAL BURN INVOLVE?

When an incinerator operator is faced with the need to perform a trial burn, the first questions that come to mind are: "What do I do for a trial burn?" and "What does the trial burn do to me?." From the operators' perspective, the key trial burn considerations are the regulatory limits that must be achieved, the permit conditions that result from the burn, and the extent of sampling and analysis activities required. Potential trouble spots that have been encountered are: (1) trial burns frequently take more time and effort than an operator anticipates; and (2) failure to meet the trial burn requirements. Each of these considerations is discussed below.

##### 1. Regulatory Limits

The trial burn provides regulatory agencies with data that will allow them to issue an operating permit. Consequently, the trial burn is directed to testing the plant to show that it achieves the RCRA limits, under the desired plant operating conditions. Those RCRA limits are:

- Destruction and removal efficiency (DRE) > 99.99% for all subject principal organic hazardous constituents (POHCs).
- Particulate emission < 180 mg/dscm (corrected to 7% O<sub>2</sub>).
- Hydrogen chloride (HCl) emissions < 4 lb/hr, or > 99% removal efficiency.

In addition to the above standards, state permit officials may add their own individual trial burn and permit conditions to the federal standards.

## 2. Permit Conditions

From the operator's standpoint, operating conditions imposed by a permit need to allow the plant to incinerate the types and quantities of waste they expect to handle, at the necessary feedrates, and within an acceptable range of operating conditions. That is, the permit conditions need to provide the plant with the desired flexibility, within limits that are reasonably achievable. Based on the trial burn results, the operating permit may specify certain criteria such as:

- No wastes may be incinerated which contain any Appendix VIII<sup>a</sup> compound having a higher heating value (HHV) below that of the most difficult to incinerate POHC used in the trial burn.
- Maximum concentration of certain POHCs in waste feed.
- Maximum waste feedrate, and/or maximum total heat input rate.
- Maximum air feedrate, or maximum flue gas velocity.
- Minimum combustion temperature.
- Maximum carbon (CO) monoxide content of stack gas.
- Maximum chloride (Cl) and ash content of waste feed.

Additional criteria are discussed in Reference 1.

The trial burn involves testing at conditions that meet the plant's operating needs while meeting the three RCRA limits. It may be necessary to test at more than one operating condition in order to satisfy all those needs. For example, it might be difficult to achieve a high heat input rate (i.e., design heat input rate) with a waste feed that contains desired high levels of Cl and ash. These factors are discussed more fully as a part of planning activities in Sections III-B and III-C.

## 3. Sampling and Analysis Activities

Each test run in the trial burn includes sampling of the waste feeds and the stack effluent. These samples are then split into a series of subsamples to be analyzed for POHCs, Cl, HHV, ash, etc. The subsamples are then analyzed for the subject POHCs by rather complex methods that include analyses by gas chromatography/mass spectrometry (GC/MS). Analysis results, along with waste feedrates and stack gas flow rates measured during each run, are used to calculate the DREs. Usually, samples of ash and scrubber waters are also taken and analyzed for the subject POHCs. Although not required by RCRA, regulatory agencies may impose other additional sampling and/or analysis requirements. More detail on sampling and analysis procedures is included in Section II-B.

For any trial burn, at any one set of operating conditions (and waste feed characteristics), EPA documents recommend three replicate runs. However, it may be acceptable to make three or more runs with each run done at different conditions or with different waste feed characteristics. In this

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<sup>a</sup> Federal Register, Wednesday, May 20, 1981, Vol. 46, No. 97, 40 CFR 261, Appendix VIII, p. 27477.

regard, there appear to be differences in what is acceptable from case-to-case, so plans must be approved by the responsible regulatory agencies before the trial burn. The Trial Burn Plan should specify the number of runs and the test conditions for each run.

An important thing to remember in planning for three or more runs is that the quantities of waste required are substantial. Each run may require 4 to 8 hr of plant operating time. It is probably best to also burn the same, or very similar, wastes during nontest periods (i.e., at night) in order to maintain reasonably steady conditions over the test period. The total trial burn period can require a rather large quantity of the specified waste(s). Those quantities are also specified as part of the Trial Burn Plan.

#### 4. Trial Burn Time Requirements

A major factor in performing a trial burn is time. Many steps are involved in the trial burn sequence of events listed in Table 1. Some of the steps have time limits dictated under RCRA. For others, adequate time must be allowed. For example, the many samples obtained in a trial burn, and the complexity of POHC analysis, make it desirable to allow 1-1/2 months to complete the analyses and another half month to prepare a detailed report of all results. General guidelines for time requirements are included in Table 1. In specific instances greater amounts of time may be required. If an operator is unfamiliar with trial burns, consultation with other operators, consultants or agency personnel early in the process can provide more exact estimates of time requirements for specific situations.

In addition to the time required to adequately prepare and conduct the trial burn, time is also required for preparation of the Part B Permit Application. Frequently the applicant will be working on trial burn preparations and responding to letters and comments on the RCRA permit simultaneously.

#### 5. Assessing Potential Performance Problems

Probably the most important question faced by the operator is "Will I pass?" (i.e., meet the RCRA requirements). The trial burn can be designed to include several different operating conditions including some where potential incinerator performance problems are minimized. Another alternative selected by some plants is to conduct an unofficial preliminary "miniburn" (i.e., one run) prior to the actual trial burn. This miniburn provides some indication of the results that can be expected, but it must be done at least 2 months before the scheduled trial burn in order to complete all analyses, evaluate the results, and make whatever changes are required.

#### B. WHAT TYPES OF SAMPLING AND ANALYSIS ARE TYPICALLY REQUIRED?

The primary objectives of the sampling and analysis (S&A) program are: (a) to quantify POHC input and output rates to determine whether DRE requirements are met; (b) to measure input and output rates of chloride; and

TABLE 1. TIME FACTORS INVOLVED IN A TRAIL BURN

- 
- 
- Notification to submit Part B application.
  - Evaluate all conditions at which plant desires to be permitted (1 month).
  - Prepare trial burn plan and submit to EPA (required 6 months after notification).
  - Prepare responses to EPA on any questions or deficiencies in the trial burn plan (1 month).
  - Make any additions or modifications to plant that may be necessary (1 to 3 months).
  - Prepare for trial burn.
    - \* Prepare for all S&A, or select S&A contractor (2 to 3 months).
    - \* Select date for trial burn, in concert with S&A staff or contractor (completed 1 month prior to test).
    - \* Notify all appropriate regulatory agencies (1 month).
    - \* Obtain required quantities of waste having specified characteristics.
    - \* Calibrate all critical incinerator instrumentation (2 weeks).
  - Conduct trial burn sampling (1 week).
  - Sample analysis (1 to 1-1/2 months).
  - Calculate trial burn results (1/2 month).
  - Prepare results and requested permit operating conditions for submittal to EPA (1/2 to 1 month).
  - Obtain operating permit.
- 
-

(c) to determine stack effluent particulate concentrations. The two most important considerations are selecting the S&A "matrix" (i.e., selecting the streams to be sampled and analytes to be measured) and identifying appropriate S&A methods. Specific problems which can be encountered are adverse stack conditions, sample train sealing problems, and the need for specialized S&A methods. Each of these factors is discussed briefly below and in more detail in Sections III-F and G and IV-A and B.

## 1. Selecting the S&A Matrix

The main focus of the sampling activities is collection of the waste feed and the stack effluent samples, the latter being the most complex. Usually, the ash and scrubber waters are also sampled and analyzed. The main focus of the analysis activities is on the POHCs. The stack S&A also includes determination of HCL and particulate emissions, but these methods are relatively simple compared to those for POHCs. A discussion of sampling and analysis needs can also be found in References 1, 2, and 3.

Overall, the S&A typically required consists of the following, as a minimum.

- Obtain representative samples of each waste feed stream to the incinerator. Analyze those samples for the selected POHCs, and for HHV, Cl, and ash. (Remember that the input rate of each waste feed must also be determined in order to compute the POHC input rate which is used in the calculation of DRE.)

To achieve a "representative" waste feed sample, liquid waste feeds are often sampled once every 15 min and composited in each run. Solid waste feeds must also be sampled using the best practical method of obtaining representative samples of each type of solid waste used in the trial burn.

- Sample stack emissions to determine stack gas flowrate, HCL, particulate concentration, and to determine concentration of POHCs.

## 2. Identifying S&A Methods

An example of S&A methods that could be specified for a trial burn is shown in Tables 2 and 3. These tables identify the main references that are available on recommended S&A methods, particularly Refs. 2 and 3. These documents contain valuable information but do take considerable time to understand. They are best utilized by personnel experienced in S&A methods. These references are the best sources to identify the methods that can be used in a Trial Burn Plan. However, experience helps a great deal in selecting the most appropriate of the available recommended methods.

Determination of stack gas flow rate and particulate emissions is done according to the conventional stack sampling method commonly referred to as Method 5 (M5). This method encompasses EPA Methods 1-5 and is defined in detail in 40 CFR Part 60, Appendix A. HCL emissions are sampled by modifying the Method 5 train to include a caustic impinger.



TABLE 2. SAMPLING METHODS AND ANALYSIS PARAMETERS

Sample	Sampling frequency for each run	Sampling method <sup>a</sup>	Analysis parameter <sup>b</sup>
1. Liquid waste feed	Grab sample every 15 min	S004	V&SV-POHCs, Cl <sup>-</sup> , ash, ult. anal., viscosity, HHV
2. Solid waste feed	Grab sample of each drum	S006, S007	V&SV-POHCs, Cl <sup>-</sup> , ash, HHV
3. Chamber ash	Grab 1 sample after all 3 runs are completed	S006	V&SV-POHCs, EP toxicity
4. Stack gas	Composite	MM5 (3 hr)	SV-POHCs, particulate, H <sub>2</sub> O, HCl
	Three pair of traps, 40 min each pair	VOST (2 hr)	V-POHCs
	Composite in Tedlar gas bag	S011	V-POHCs <sup>c</sup>
	Composite in mylar gas bag	M3 (1-2 hr)	CO <sub>2</sub> and O <sub>2</sub> by Orsat
	Continuous (3 hr)	Continuous monitor	CO (by plant's monitor)

<sup>a</sup> VOST denotes volatile organic sampling train  
MM5 denotes EPA Modified Method 5  
M3 denotes EPA Method 3  
SXXX denotes sampling methods found in "Sampling and Analysis Methods for Hazardous Waste Combustion."<sup>3</sup>

<sup>b</sup> V-POHCs denotes volatile principal organic hazardous constituents (POHCs).  
SV-POHCs denotes semivolatile POHCs.  
HHV denotes higher heating value.

<sup>c</sup> Gas bag samples may be analyzed for V-POHCs, only if VOST samples are saturated and not quantifiable.

TABLE 3. EXAMPLE ANALYTICAL PROCEDURES

Sample	Analysis parameter	Sample preparation method	Sample analysis method
1. Liquid waste feed	V-POHCs	8240	8240
	SV-POHCs	8270	8270
	Cl	-	E442-74
	Ash	-	D482
	HHV	-	D240
	Viscosity	-	A005
2. Solid waste feed	V-POHCs	8240	8240
	SV-POHCs	8270	8270
	Cl	-	D-2361-66 (1978)
	Ash	-	D-3174-73 (1979)
	HHV	-	D-2015-77 (1978)
3. Ash	V-POHCs	8240	A101
	SV-POHCs	P024b, P031	A121
	Toxicity	-	C004
4. Stack gas			
a. MM5 train			
Filter and	Particulate	M5	M5
probe rinse	SV-POHCs	P024b, P031	A121
Condensate	Cl	-	325.2
	SV-POHCs	P021a	A121
XAD resin	SV-POHCs	P021a	A121
Caustic impinger	Cl	-	325.2
b. VOST	V-POHCs	-	A101
c. Tedlar gas bag	V-POHCs	-	A101 <sup>a</sup>
d. Gas bag	CO <sub>2</sub> , O <sub>2</sub>	-	M3 (Orsat)
e. Cont. monitor	CO	-	Continuous monitor

Note: Four-digit numbers denote methods found in "Test Methods for Evaluating Solid Waste," SW-846.<sup>2</sup>

Numbers with prefixes of A, C, and P denote methods found in "Sampling and Analysis Methods for Hazardous Waste Combustion."<sup>3</sup>

Method No. 325.2 (for Cl) is from "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1979.<sup>4</sup>

Numbers with prefixes D and E denote methods established by the American Society for Testing and Materials Standards (ASTM).

M3, M5 refer to EPA testing methods found in the Federal Register, Vol. 42, No. 160, Thursday, August 18, 1977.<sup>5</sup>

<sup>a</sup> Tedlar gas bag samples will be analyzed for V-POHCs, only if VOST samples are saturated and not quantifiable.

Sampling of stack effluent for POHCs, in order to determine DRE, may require from one to three separate methods (or more), depending on the number of POHCs to be quantified and their characteristics (e.g., volatile or semivolatile), and on the detection limits that are required to prove a DRE of 99.99%. These methods are:

1. Modified Method 5 (MM5) - for semivolatile POHCs.
2. Volatile Organic Sampling Train (VOST) - for volatile POHCs.
3. Gas bags - for volatile POHCs.
4. Special methods - for certain POHCs which cannot be sampled with any of the above methods.

Semivolatile POHCs commonly require use of MM5. This one sampling train, shown in Figure 1, provides for determination of particulate, HCl, and the SV-POHCs. However, the probe rinse must be evaporated and the filter desiccated to determine particulate. Thereafter, these components can be extracted, and combined with extracts of the XAD resin and the condensate, for analysis by GC/MS to determine SV-POHCs. A small aliquot of the condensate must be removed before extraction to quantify Cl<sup>-</sup> in the condensate, as well as in the caustic.

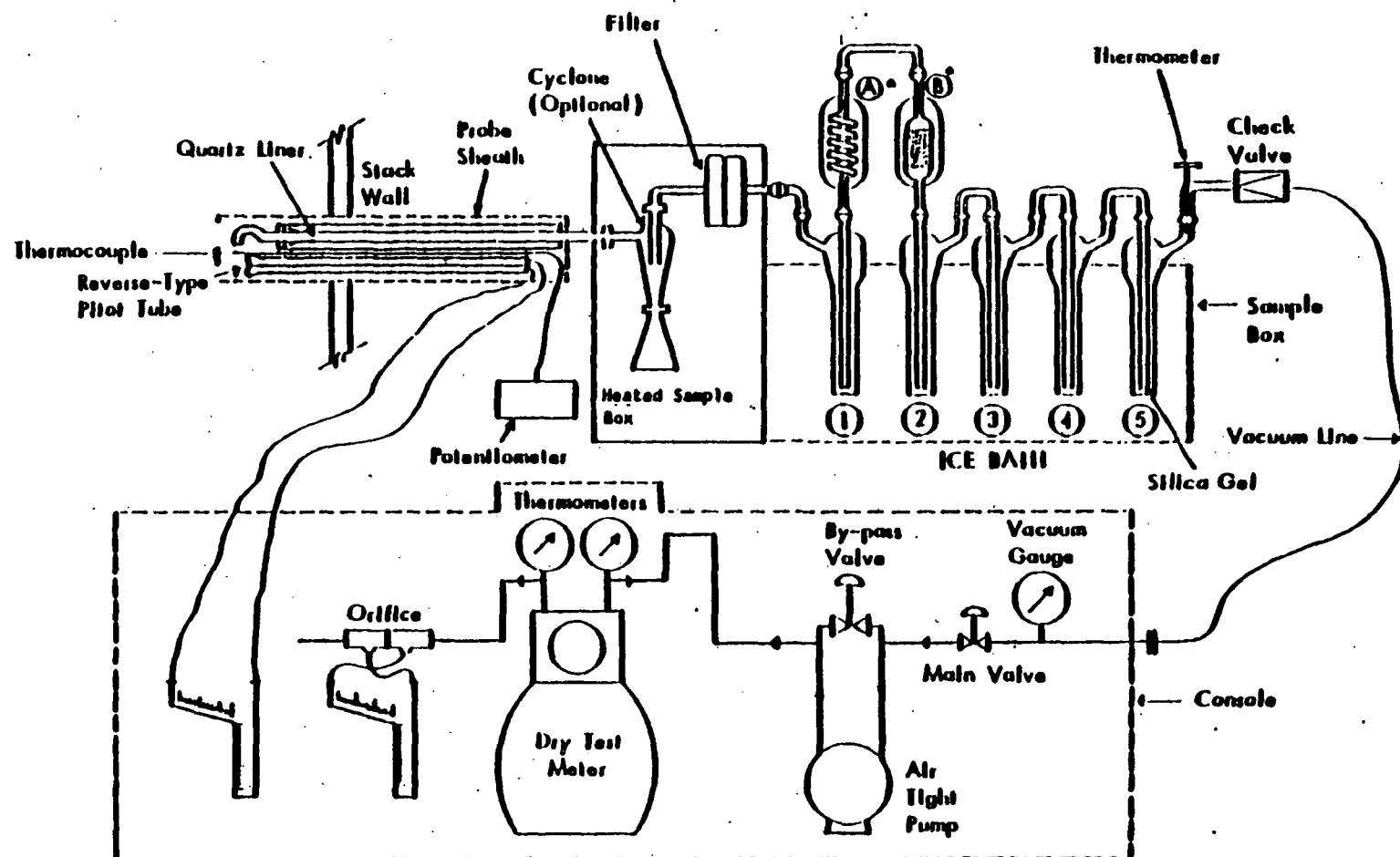
A diagram of the VOST train commonly used for sampling volatile POHCs is shown in Figure 2. This train, unlike M5 or MM5, does not involve traversing the stack. However, the VOST preparation and analysis procedures are quite complex. Those interested in the detailed procedures should refer to Ref. 6.

### 3. Adverse Stack Sampling Conditions

Adverse stack sampling conditions are frequently encountered at hazardous waste incinerators. Problems that have been encountered include cyclonic flow, very high temperature stacks (1600° to 1800°F), and high moisture content (saturated with H<sub>2</sub>O at 150°F with droplet carryover). These potential problems should be considered during planning, and appropriate actions should be taken. More complete discussions of cyclonic flow and moisture are included in Section IV-B-2.

### 4. Sample Train Sealing Problems

Both the VOST method, and available guidance on the MM5 method, state that no grease be used on any of the connections in the train (i.e., ball-joints). Teflon or Viton O-rings have been used in VOST, and in MM5, to provide adequate seals without use of grease. Added care must be taken to ensure leak-check integrity of the sampling trains, with some added risk that a test may have to be repeated if any sampling train fails the post-test leak check.



- ① Modified Greenburg-Smith, Reversed, Empty
- ② Greenburg-Smith, 50 ml of Double Distilled In Glass  $H_2O$
- ③ Greenburg-Smith, 100 ml of 0.1 N KOH
- ④ Modified Greenburg-Smith Empty
- ⑤ Modified Greenburg-Smith,  $SiO_2$

- (A) Condenser
- (B) XAD Resin Cartridge
- \* Ice Water Jacket

Figure 1. Modified Method 5 sampling train (MM5)

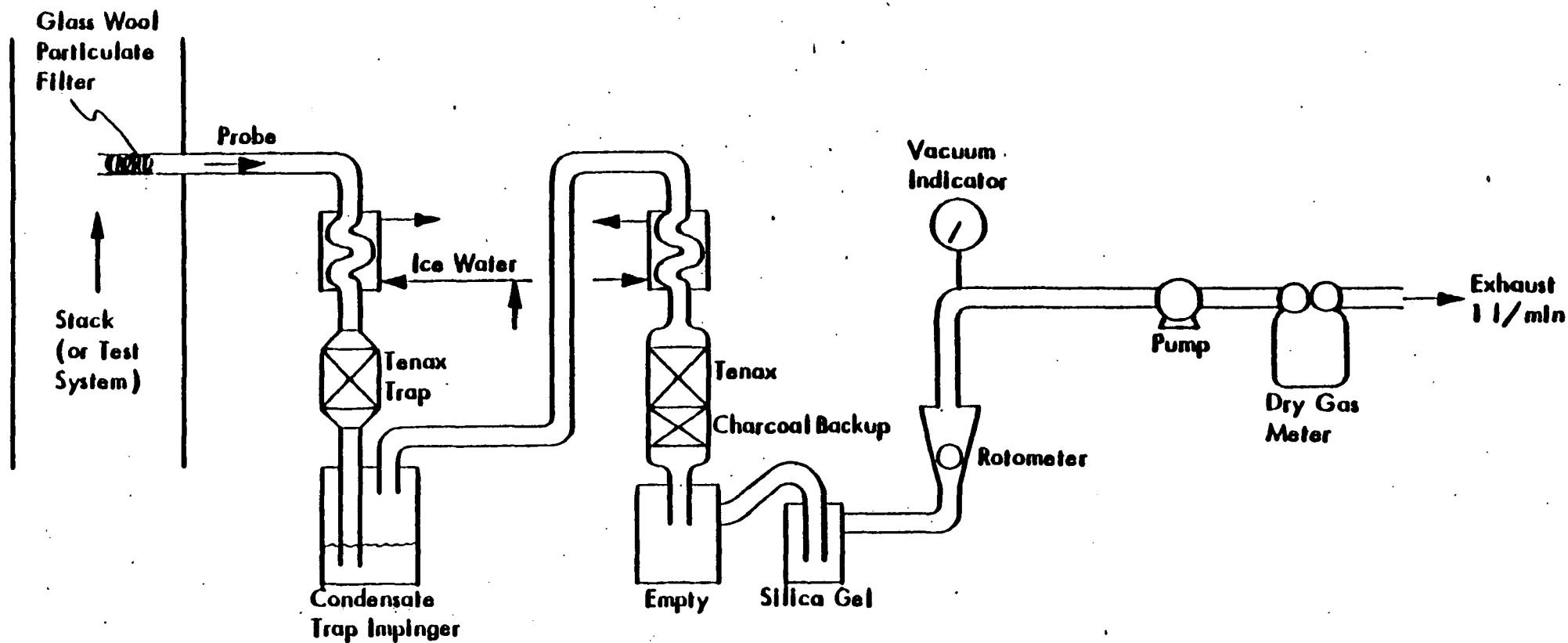


Figure 2. Volatile organic sampling train (VOST).

## 5. Need for Specialized Methods

Although the majority of POHCs are sampled with either VOST or MM5, specialized sampling methods must be used for some POHCs. Those POHCs which require specialized methods are identified in Appendix B of Ref. 3. This reference should be consulted during the planning stage to assure that proper methods are used.

### C. WHAT SKILLS, EQUIPMENT, AND FACILITIES ARE NEEDED TO CONDUCT A TRIAL BURN?

The incinerator facility operator is responsible for conducting the trial burn. The facility must provide the types and quantities of waste needed, and operate the plant during the trial burn at the conditions under which they desire to be permitted. However, the specialized sampling and analyses required in a trial burn are beyond the capability of most facilities. A facility that has most of the necessary capabilities still may decide to use an experienced contractor because of the specialized nature of the methods and the fact that the trial burn may be only a one time need. The important considerations in the decision are facilities and equipment requirements and staffing needs. Each of these factors, plus a brief consideration of contractor selection, are addressed below.

#### 1. Facilities and Equipment

Whether the operator uses a contractor or their own staff for the trial burn S&A, certain capabilities are required. The facilities and equipment that are usually necessary are shown in Table 4.

#### 2. Staffing Needs

Decisions regarding use of in-house or contractor expertise to conduct a trial burn also depend on staffing needs. At a minimum, the trial burn staff should be knowledgeable in stack sampling methods, have experience in analysis of low concentrations of organics in complex matrices, and be familiar with calculating and reporting trial burn results. Knowledge of process monitoring is also helpful. A detailed discussion of the number and capability of S&A personnel required is included in Section III-E.

#### 3. Selecting a Contractor

Trial burn procedures are relatively new, and are much more sophisticated than a normal EPA Method 5 test for particulate emissions. There are about 10 to 20 organizations in the United States who have trial burn S&A experience, and probably several more who are capable of doing so. If a facility would like to know who to contact for S&A service, they should make inquiries at state and federal regulatory agencies or contact other incinerator facilities who may have already conducted trial burns.

TABLE 4. CAPABILITIES NECESSARY FOR TRIAL BURN SAMPLING AND ANALYSIS

- 
1. Sampling equipment for solid waste feeds (especially drummed wastes).
  2. Stack sampling equipment, usually including the following:
    - EPA Method 5 equipment, and all associated test equipment (e.g., EPA Methods 1, 2, and 3).
    - Method 5 equipment adaptable to Modified Method 5 (greaseless) and associated XAD resin preparation, extraction, and analysis facilities.
    - Volatile Organic Sampling Train (VOST) Equipment with at least 18 pairs of VOST traps. Also, all facilities needed for preparing, checking, and analyzing the traps.
    - Gas bags and associated sampling equipment (see Figure 7, Reference 3).
    - Field laboratory equipment for sample recovery.
  3. Facilities for analyzing all samples, including:
    - Laboratories containing relevant safety equipment such as hoods and equipped with sample preparation equipment including Soxhlet extractors, separatory funnels, continuous extractors, blenders, Sanifiers or other equipment to homogenize waste feed samples, sodium-sulfate drying tubes, Kuderna-Danish glassware, etc.
    - Equipment for preparing VOST traps to allow simultaneous heating and purging of the traps. Ideally the traps should be prepared and stored in an organic-free laboratory.
    - All required compounds to prepare calibration standards and surrogate recovery spiking solutions.
    - Computerized GC/MS instrumentation.
    - Established QA procedures for assessing precision and accuracy of analytical methods.
  4. Knowledge and preferably experience in all of the sampling and analysis methods and calculation/reporting of results.
  5. Process monitoring experience, especially quantification of waste feed-rates and documentation of plant operating conditions.
-

#### D. WHAT ARE THE MAJOR COST FACTORS ASSOCIATED WITH A TRIAL BURN?

The three major cost components of the trial burn are planning and preparation, sampling and analysis and quality assurance (QA). Each of the components must be included in trial burn budgets.

##### 1. Planning and Preparation

One of the first cost factors for a trial burn is preparation of the Trial Burn Plan, including preparing responses to questions and additional information requested by the regulatory agencies.

The second cost factor is plant additions and modifications needed to comply with RCRA regulations (see Section III-A). These may include CO monitors, waste feed flow monitors, etc., and stack sampling ports and scaffolding needed for the trial burn.

A third cost factor is associated with acquiring/storing the types and quantities of wastes necessary for the trial burn (see Section III-C).

##### 2. Sampling and Analysis

The major cost factor is the sampling and analysis required by the trial burn. In general, this cost usually ranges from \$30,000 to \$150,000, depending on the number of runs, the number of samples to be taken in each run, and the analysis required on each sample.

The number of runs to be conducted in the trial burn is one of the major cost factors. EPA recommends three replicate runs at each operating condition to be tested. Thus, a minimum of three runs are usually done. If two operating conditions are involved, then six runs may be necessary. In some cases, an array of operating conditions are tested, with only one run at each condition.

In each run, all influent and effluent streams are usually sampled and analyzed as described earlier (see Table 2). The number of these samples and the complexity of their analysis obviously affects the cost. Ordinarily, the field sampling activity, including all preparation for sampling, accounts for one-fourth to one-third of the S&A cost. Analysis of samples usually accounts for one-third to one-half the cost, with the remaining costs for data reduction, calculations, and reporting of results.

##### 3. Quality Assurance

Analysis costs, especially for the POHCs, are a rather large cost factor, partly because analytical QA activities typically include:

- Replicate analysis of some samples.
- Analysis of samples spiked with POHCs or surrogates.
- Analysis of blanks.



- Analysis of calibration standards.
- Analysis of blind audit samples.

All of the above can easily involve analysis of twice the number of samples actually taken. All these samples then must be multiplied by the number of analyses to be performed on each sample. Some QA is an essential part of a trial burn, but excessive QA can rapidly increase the cost.

#### 4. Estimating the Costs

Determining the cost for a trial burn is highly site dependent. In general, the trial burn costs will depend on the following factors, as discussed in the preceding sections:

- Number of runs
- Number and type of waste feed samples
- Number of effluent samples
- Number of different analyses performed on each sample
- Complexity of the QA/QC plan
- Modifications required to prepare facility for trial burn

The normal range for a trial burn sampling and analysis program conducted by an outside contractor is \$30,000-\$150,000+. This range does not include plant modifications, preparations on-site for the test, or preparation of the permit application and trial burn plan.

The breakdown of costs for a trial burn is roughly: one-third for the field sampling program; one-third for sample analysis and project QA/QC; and one-third preparation, engineering calculations and reporting. These are rough estimates and frequently the analysis portion of the program can involve as much as half of the total cost.

In summary, the sampling and analysis part of a trial burn is costly, and each time another run, another sample, or another analysis is added to the test plan the cost will rise. Each such addition needs to be carefully considered in order to hold costs at the lower end of the range.

### SECTION III

#### PLANNING FOR A TRIAL BURN

The probability for success of a trial burn is enhanced by good planning. The major objectives of the planning process are: (a) to select trial burn conditions that provide the plant adequate operating flexibility; (b) to assure that the trial burn will be conducted in a manner acceptable to regulatory agencies; and (c) to make the trial burn cost effective. Key questions addressed during planning are:

- A. What equipment/instrumentation is the incinerator required to have?
- B. How should operating conditions be selected?
- C. How should trial burn POHCs be selected?
- D. What types and quantities of waste are needed and how can they be prepared?
- E. How many runs are necessary and how long is each run?
- F. How many people are needed and with what experience?
- G. How are POHC sampling methods selected?
- H. What detection limits are required for the sampling and analysis methods?
- I. What QA/QC needs to be done?
- J. How is it best to plan for the possibility that trial burn results may be outside RCRA requirements?

#### A. WHAT EQUIPMENT/INSTRUMENTATION IS THE INCINERATOR REQUIRED TO HAVE?

The incinerator is required by RCRA to have the equipment/instrumentation shown in Table 5. The regulatory agencies also may require monitoring of other important operating parameters (e.g., scrubber water flow rates, venturi scrubber  $\Delta P$ , etc.). Minimum or maximum levels for each parameter may be specified in the operating permit. Analysis of waste feeds may also be required if the operating permit stipulates limitations on HHV, Cl, or ash content of waste feed.

TABLE 5. INCINERATOR EQUIPMENT/INSTRUMENT REQUIREMENTS FOR TRIAL BURN

- 
- 
- Equipment to maintain particulate emissions below 0.08 grains/dscf.
  - Equipment to maintain 99% HCl removal or HCl emissions below 4 lb/hr.
  - Equipment that provides 99.99% DRE on POHCs.
  - Stack test ports and scaffolding.
  - Valves, taps, etc., for sampling all waste feeds, liquid effluents, ash, etc.
  - Equipment to maintain noncyclonic flow in stack when testing.
  - Continuous CO monitor.
  - Continuous waste feed flow monitor.
  - Continuous monitor for combustion gas velocity or air input rate.
  - Continuous combustion temperature monitor.
  - Automatic interlock system to shut off waste feed under the following situations.
    - a. Low combustion temperature.
    - b. High CO concentration.<sup>a</sup>
    - c. High combustion airflow to incinerator or high combustion gas velocity.
- 
- 

<sup>a</sup> Established based on trial burn results or state statutory limitations.

## B. HOW SHOULD THE OPERATING CONDITIONS BE SELECTED?

Operating conditions for the trial burn are selected to provide the plant with operating flexibility. Important considerations are the key operating parameters that affect permit conditions and the use of pretests or miniburns to help establish those conditions while meeting RCRA requirements (e.g., DRE).

### 1. Operating Parameters that Affect Permit Conditions

The operating conditions selected for the trial burn must represent the worst case conditions under which the incinerator may expect to operate, and therefore needs to be permitted to operate. The conditions selected may include any or all of the following:

- Waste containing hardest-to-burn POHC (lowest HHV).
- Highest concentrations of all POHCs selected.
- Maximum waste feedrates.
- Maximum combustion airflow rate (minimum residence time).
- Maximum CO level in stack gas.
- Minimum combustion temperature.
- Minimum HHV of waste.
- Maximum thermal input (Btu/hr).
- Minimum O<sub>2</sub> level in stack gas.
- Maximum Cl content of waste feed.
- Maximum ash content of waste feed.
- Minimums or maximums on other operating conditions (e.g., venturi scrubber ΔP, scrubber water flow rate and pH).

Obviously, it is very difficult to achieve all of the above at any one set of operating conditions. In fact, some of the conditions are almost direct opposites (e.g., maximum airflow rate but minimum O<sub>2</sub> in stack gas).

The first six items in the above list are probably the most important and may be achievable in one set of operating conditions that also include some of the other conditions. If so, one trial burn (three runs) at those conditions may suffice. If not, additional runs that include the other conditions may be necessary. Of course, operating conditions which result in permit conditions most favorable to each individual facility will have to be determined on a case-by-case basis.

The major problem with the worst-case conditions is that they maximize the chance of failure (not meeting RCRA requirements). Since the plant wants to pass, the exact conditions must be carefully selected, balancing operating needs against increasing chance of failure. Plant operating experience is very important in these decisions.

## 2. Use of Pretest or Miniburns

Preliminary testing and miniburns can be extremely valuable in helping to select operating conditions for the actual trial burn. The following types of miniburns may be useful.

The hardest to burn POHC, at high concentration, can be used in a miniburn that is conducted at the lowest temperature and the highest CO level. If the results show a DRE exceeding 99.99%, then it is likely that 99.99% DRE will be achieved regardless of any other operating conditions.

At high Cl input rates, a well designed scrubber will not usually fail 99% removal even at minimum conditions. A pretest could verify that presumption.

Achieving the particulate limit causes problems more frequently than does achieving DRE. A pretest with EPA Method 5 will help identify any problems and help in selecting conditions for the trial burn. The pretest can also uncover specific sampling and analysis problems that may not be readily apparent.

Mist carried over from a recirculating scrubber solution or alkaline scrubbers can have a drastic impact on particulate emission measurements, especially if the scrubbers are not equipped with efficient mist eliminators. It may be advisable to conduct a preliminary particulate test, well in advance of the actual trial burn, to identify possible problems.

For existing plants, any of the above pretesting could be done prior to submitting the Trial Burn Plan for approval. For new plants, pretesting will have to be part of the approved Trial Burn Plan.

## C. HOW SHOULD TRIAL BURN POHCs BE SELECTED?

POHCs for the trial burn should be selected during development of the trial burn plan. The selection is in conformance with the regulatory approach laid out in the Guidance Manual for Hazardous Waste Incinerator Permits (Reference 1). In addition to the regulatory criteria, the following two considerations should be taken into account: (1) maximum flexibility of operating conditions under the permit; and (2) ease of sampling and analysis during the trial burn.

Currently the regulation requires that a DRE of 99.99% be demonstrated for the selected POHCs.

In addition, the following limits will result from the selection of POHCs for the trial burn:

- Appendix VIII compounds in any subsequently burned waste feed must be present in concentrations lower than the POHC in highest concentration during the trial burn.
- Appendix VIII compounds in any subsequently burned waste must have a heat of combustion ranking higher than the POHC with the lowest heat of combustion during the trial burn. (Heats of combustion for all Appendix VIII compounds have been determined and can be found in Reference 1).

Because of these limits, the POHCs chosen for trial burn testing must include the Appendix VIII compounds in the waste feed, usually the compounds in the highest concentration and with the lowest heat of combustion. "Appendix VIII" refers to the Appendix of the hazardous waste regulations which lists compounds which are considered hazardous (see 40 CFR 261 Appendix VIII).

It is important that the Appendix VIII compound present in highest concentration in any proposed waste feed be present in the feed during the trial burn at the maximum concentration expected, in order to obtain the necessary permit conditions. Likewise, it is important that the compounds with the lowest heat of combustion be present in the waste feed used during the trial burn at sufficient levels to determine 99.99% DRE (see Section II.H).

In selecting POHCs for a trial burn, sampling and analysis implications also must be considered. From this point of view, Appendix VIII compounds fall into three categories:

- Volatiles - compounds which can be sampled using the VOST (in certain cases other methods may be more appropriate, as discussed in Section III.H)
- Semivolatiles - compounds which can be sampled using the Modified Method 5 train
- Other - compounds which must be sampled using different techniques; special trains, colorimetric methods, etc. These include compounds which degrade easily in water or which have special interferences or are otherwise difficult to quantify using GC/MS analysis.

Ideally, all trial burn POHCs could be selected from either the volatile or semivolatile group. This minimizes the number of sampling trains used in the field and simplifies the analysis. If possible the "other" category should be avoided, because more specialized equipment may be needed, which will have to be cleared by permit reviewers in advance of the test, and may result in higher sampling and analysis costs. An additional consideration is to avoid POHCs which also might show up as products of incomplete combustion from the burning of the waste (e.g., chlorinated benzenes, ethanes, and methanes).

All of these considerations must be taken into account when selecting POHCs for a trial burn. One solution which has been used at incinerators which hope to burn a wide variety of wastes, is spiking of a low heat of combustion compound (e.g., carbon tetrachloride or perchloroethylene) in significant concentrations (5-10%) into the waste feed.

D. WHAT TYPES AND QUANTITIES OF WASTE ARE NEEDED AND HOW CAN THEY BE PREPARED?

The response addresses calculation of waste quantities, assuring adequate supplies of waste by type, and preparation of wastes. Specific problems addressed include mixing of synthetic or spiked wastes and time requirements.

1. Quantities of Waste

The quantity of waste required is dependent on the waste feedrate to be used during each run, the number of runs, and the duration of each run. Waste feedrate and number of runs are selected by the incinerator operator, and are specified in the Trial Burn Plan. The sampling time required in each run is usually 3 to 4 hr plus 1 hr to line out the unit before start of testing, and 1 to 2 hr for contingencies (plant operating problems or sampling problems). Considering these, a quantity of waste sufficient for 8 hr of operation should be available for each run. If the trial burn involved only three runs, at one set of operating conditions, then waste sufficient for 24 hr of operation should be available.

2. Types of Waste

Sufficient quantities of waste must be available for each type of waste feed that is used. Each type of waste must have all the specific characteristics that are required to meet selected operating conditions. For example, the waste to be burned during a trial burn might include both continuous feeding of an organic liquid and intermittent feeding of drummed solids. Each of these wastes must meet certain specifications selected for the trial burn, including POHC concentrations, heating value, Cl and ash content, etc.

3. Waste Preparation

Three methods can be used to prepare the required quantities of wastes possessing the correct characteristics. These three methods pertain mainly to POHC characteristics but may be used to achieve any of the necessary characteristics. The three methods are:

- a. Use actual wastes,
- b. Use synthetically prepared wastes, or
- c. Continuously spike POHCs into the waste during the trial burn.

Method (a) usually is desirable, if it is possible to acquire actual wastes that have the necessary characteristics or to achieve those characteristics by blending of actual wastes. Method (b) usually involves using actual wastes mixed with purchased chemical compounds (i.e., POHCs). Method (c) is similar to method (b) except that it applies mainly to continuous liquid feeds, with the purchased chemical(s) continuously pumped into this feedline.

#### 4. Mixing

All of the above three methods require that the waste feeds be well mixed, but mixing is especially important for methods (b) and (c). Lack of good mixing for any waste feed can, and has, caused problems in trial burns. For method (c) the trial burn may involve continuous spiking of POHCs (pure components or mixtures thereof) into a liquid waste feed line. When this is to be done, a connection (1/4 or 1/2-in. valve) must be provided, with another sample tap located further downstream. It is also highly advisable for the plant to install an inline mixer between these two connections to help ensure that the "spiked" components are well mixed with the waste and that the samples collected are representative of this mixture.

#### 5. Time Requirements

Another important factor in waste preparation is time. The quantities of waste involved can be rather large, and it may take several weeks to acquire sufficient quantities of wastes to prepare a homogeneous batch with the proper characteristics. Storage space for these "special" wastes, over some time period, can impact normal plant operations. Finally, some additional time may be needed to sample and analyze the wastes to be sure they have the necessary characteristics.

Adequate time also must be allowed for numbering, weighing, and sampling of drummed solids before the trial burn. Since the number of drums may exceed 300, the problem of weighing and sampling initially may not be realized. Also, samples of drummed solids must be representative of those drums used in each run. Representative samples may be obtained by sampling each drum during each run, or by "staging" the drums to be used in each run and sampling them prior to the trial burn.

#### E. HOW MANY RUNS ARE NECESSARY AND HOW LONG IS EACH RUN?

This question was discussed in Sections II-A-3 and II-D-2. Additional points offered as guidance are:

- Each run will require at least 2 to 4 hr. It is best to plan only one run per day, except in special cases when sampling is less complex than usual. Quite often, when an incinerator operator hears that the sampling time required for each run is 3 to 4 hr, it is assumed that the sampling crew can do two runs each day. However, the sampling crew has about 2 hr of work in preparing for each run and at least 2 hr of work after each run is completed to recover, label, and package each sample. In many instances a variety of problems do occur, both in plant operations and in sampling, so that one 3 to 4 hr run may involve a 12 to 16 hr day for the sampling crew. The most reasonable assumption is that one run can be completed each day.

EPA recommends three runs at each set of operating conditions to be tested.



- If several sets of operating conditions are to be tested, regulatory agencies may allow fewer than three runs at each condition.
- Conducting more than six runs may not be cost effective.

#### F. HOW MANY PEOPLE WILL BE NEEDED, WITH WHAT EXPERIENCE?

Personnel required for sampling during the trial burn usually number between 5 and 10, depending on the complexity of the sampling. A typical example, reflecting the sampling plan shown earlier in Table 2, is presented in Table 6.

The personnel list in Table 6 is only an example. In some cases one person can do multiple jobs depending on sampling frequency and complexity, and physical layout of sampling locations. Also, quite often the crew chief performs one of the sampling activities, again depending on complexity of the sampling activity. Plant personnel may perform the process monitoring. However, the data should be separate from any normal plant operating log, and usable in the Trial Burn Test Report.

#### G. HOW ARE POHC STACK SAMPLING METHODS SELECTED?

A general procedure to identify the appropriate POHC stack sampling methods is outlined in Table 7. When both volatile and semivolatile POHCs are present, both MM5 and VOST are needed. Analyses performed on these samples must provide the necessary detection limits for the POHCs, as mentioned in Tables 7 and 8.

#### H. WHAT DETECTION LIMITS ARE REQUIRED FOR THE SAMPLING AND ANALYSIS METHODS?

##### 1. Waste Feed Detection Limits

Analyses of POHCs in waste feeds must be capable of detecting the expected concentrations, which usually are above 10,000 ppm (1%). But, it is desirable that the detection limit be 100 ppm (commonly achieved by recommended analytical techniques). A POHC at this concentration or above may be considered (under RCRA) to be "significant."

##### 2. Stack Gas Detection Limits

Detection limits required for POHCs in stack gases are discussed in Table 8. The rule-of-thumb that can be used in most cases is:

$$100 \text{ ppm in waste feed} = 1 \text{ } \mu\text{g}/\text{m}^3 \text{ in stack gas at 99.99\% DRE}$$

This equation can be used to estimate stack gas concentrations for any waste feed concentration (i.e., 2,000 ppm in waste = 20  $\mu\text{g}/\text{m}^3$  in stack gas, at 99.99% DRE).

TABLE 6. A TYPICAL EXAMPLE OF SAMPLING PERSONNEL REQUIRED

Job	Number of personnel	Experience required
1. Sample liquid feed (once every 15 min)	1	Technician with sampling experience and safety training.
2. Drum solid sampling and recording (once every 5-10 min)	1	Technician with sampling experience and safety training.
3. Sampling ash and scrubber waters every 1/2-1 hr	1	Technician with safety training.
4. Stack sampling MMS	2	Experienced console operator and technician for probe pushing.
VOST	1	Experience with VOST operation.
5. Process monitor to record operating data every 1/4-1/2 hr and determine waste feed-rates	1	Engineer or other person experienced in plant operations and trial burn requirements.
6. Field laboratory	1	Experienced chemist for check-in and recovery of all samples, and preparation of sampling equipment for each run.
7. Crew chief	<u>1</u>	Person experienced in all aspects of trial burn sampling to direct all activity and solve problems that may occur.
Total	9	

TABLE 7. PROCEDURE FOR IDENTIFYING NECESSARY STACK SAMPLING METHODS

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Step 1. Determine Whether Each POHC is a Volatile or Semivolatile Compound

Volatile compounds are generally those that have boiling points below 130°C. Most can be sampled with VOST or gas bags. The best way of determining the sampling method needed is to refer to Appendix B of Ref. 2. If the POHC shows sampling by "particulate/sorbent" then MM5 is required. If it shows "sorbent" or "gas bulb" then VOST or gas bags will be the sampling method. Regardless of whether or not a POHC is a volatile or semivolatile, some POHCs require special sampling methods as indicated in Appendix B of Ref. 2 (e.g., formaldehyde).

Step 2. Estimate Concentration of Each POHC in the Stack Gas, Assuming a DRE of 99.99%

Estimation of the concentration of each POHC requires some knowledge or approximation of POHC concentration in waste feeds, waste feed-rates, and stack gas flowrates. Using that information, concentrations of each POHC in the stack gas can be estimated, for an assumed DRE of 99.99% (see example calculation in Table 8).

For semivolatile POHCs, MM5 is suitable for any stack gas concentration above 1  $\mu\text{g}/\text{m}^3$ .

For volatile compounds, VOST should be used when stack gas concentrations fall within the range of 1 to 100 ng/L. However, if the estimated stack gas concentration exceeds 100 ng/L, then gas bags should also be used and analyzed in the event that the VOST sample concentrations saturate the GC/MS data system.

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TABLE 8. EXAMPLE CALCULATION FOR ESTIMATING POHC CONCENTRATION  
IN STACK GAS, AT DRE OF 99.99%

Basis:

Waste feed flowrate approximately 4 gpm  
Density of waste approximately 9 lb/gal  
POHC concentration in waste feed is near minimum significant level  
of 200 ppm (200 ppm = 0.000200 g POHC/g feed)

Stack gas flowrate unknown but total heat input to incinerator is  
approximately  $30 \times 10^6$  Btu/hr with 100% excess air

Calculation:

Rule of thumb (applies in most, but not all cases):

Each 100 Btu of heat input produces about 1 dscf of flue gas at 0%  
excess air, or 2 dscf of dry flue gas at 100% excess air

1. Flue Gas Flowrate:

$$\left( 30 \times 10^6 \frac{\text{Btu}}{\text{hr}} \right) \left( \frac{\text{hr}}{60 \text{ min}} \right) \left( \frac{2 \text{ dscf}}{100 \text{ Btu}} \right) = 5,000 \text{ dscf/min}$$

$$\left( 5,000 \frac{\text{dscf}}{\text{min}} \right) \left( \frac{1 \text{ m}^3}{35.3 \text{ ft}^3} \right) = 142 \text{ m}^3/\text{min}$$

2. Waste Feedrate:

$$\left( 4 \frac{\text{gal.}}{\text{min}} \right) \left( \frac{9 \text{ lb}}{\text{gal.}} \right) \left( \frac{454 \text{ g}}{\text{lb}} \right) = 16,300 \text{ g/min}$$

3. POHC Input Rate:

$$\left( 16,300 \frac{\text{g feed}}{\text{min}} \right) \left( 0.000200 \frac{\text{g POHC}}{\text{g feed}} \right) = 3.26 \text{ g POHC/min}$$

4. POHC Stack Output Rate, at 99.99% DRE:

$$(3.26 \text{ g/min}) (1.0 - 0.9999) = 0.000326 \text{ g/min}$$

5. POHC Concentration in Stack Gas (at 99.99% DRE):

$$\frac{0.000326 \text{ g/min}}{142 \text{ m}^3/\text{min}} = 0.0000023 \frac{\text{g}}{\text{m}^3} = 2.3 \text{ } \mu\text{g/m}^3 = 2.3 \text{ ng/L}$$

Some POHCs may require special sampling/analysis methods or may show low recovery efficiencies for MMS samples. Therefore, each case must be considered separately to ensure that the detection limit for the methods used are low enough to quantify those specific POHCs at the concentrations expected in the stack at 99.99% DRE. Consultation with analytical chemists, or with the authors and EPA project officer given in Ref. 3 can be most valuable in this regard.

### 3. High Concentration of Volatile POHCs

Since the GC/MS analytical techniques for MMS and VOST samples can easily achieve a detection limit of  $1 \mu\text{g}/\text{m}^3$ , there is usually no problem. However, high stack concentrations of some volatile POHCs may exceed the range for VOST samples (i.e., that saturate the GC/MS). Those samples require use of gas bags in order to determine if 99.99% DRE was or was not achieved. The gas bags are analyzed by transferring a small volume of sample onto a VOST trap prior to GC/MS analysis. For example, 5 L may be taken for analysis. This quantity is 4 times less than the quantity sampled by VOST under normal sampling conditions.

#### I. WHAT QA/QC NEEDS TO BE DONE?

It is important in planning the trial burn to stipulate exactly what QA will be done and to know why it is needed. Some QA activities may be desirable but are not essential in specific cases. Blanket statements that "full QA" will be employed in the trial burn are not definitive, and excessive QA can drastically increase costs. An example list of basic QA for a trial burn is given in Table 9. Preliminary discussion of QA procedures with the responsible regulatory agency is recommended prior to submittal of the Trial Burn Plan.

One example of a QA activity that may be specified without adequate thought is "chain-of-custody." The number of samples collected in a trial burn normally numbers 100 to 300. Adherence to chain-of-custody procedures for all of these samples requires considerable time and effort, with its associated cost impacts. Unless there is reason to believe that sample results will be a part of some judicial proceedings, chain-of-custody procedures on the samples may be an unnecessary added cost when traceability procedures would suffice.

#### J. HOW IS IT BEST TO PLAN FOR THE POSSIBILITY THAT TRIAL BURN RESULTS ARE OUTSIDE RCRA REQUIREMENTS?

There is always the haunting possibility that the Trial Burn results may show failure to meet one or more of the RCRA requirements. This result is more likely when the trial burn is conducted under "worst case" conditions at which the plant wants to be permitted to operate.

A miniburn and other preliminary testing (e.g., Method 5) can help identify problems before the trial burn, and, after modifications, avoid failure during the trial burn. Another alternative is to conduct runs at two sets of operating conditions. One set would be worst case, while the

TABLE 9. EXAMPLE QA FOR A TRIAL BURN

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- All equipment used in S&A activities should have written calibration procedures. Procedures and documentation of the most recent calibration should be available.
  - Traceability procedures (not necessarily chain-of-custody) should be established to ensure sample integrity.
  - A GC/MS performance check sample should be analyzed each day prior to sample analysis. If results are outside acceptable limits, samples should not be run.
  - All samples from at least one run should be analyzed in triplicate to assess precision.
  - A minimum frequency of check standards (5% is suggested) should be used with each sample batch. Analysis of actual samples should be suspended if check standards are outside of the desired range.
  - Blank samples should be analyzed to assess possible contamination and corrective measures should be taken as necessary. Blank samples include:
    - Field blanks - These blank samples are exposed to field and sampling conditions and analyzed to assess possible contamination from the field (a minimum of one for each type of sample preparation or the number specified by the appropriate method).
    - Method blanks - These blank samples are prepared in the laboratory and are analyzed to assess possible laboratory contamination (one for each lot of samples analyzed).
    - Reagent and solvent blanks - These blanks are prepared in the laboratory and analyzed to determine the background of each of the reagents or solvents used in an analysis (one for each new lot number of solvent or reagent used).
  - Field audits and laboratory performance and systems audits may be included in some cases. Cylinders of audit gases for volatile POHCs are available from EPA.
  - A minimal level of calculation checks (e.g., 10%) should be established.
- 
-

other set could be conditions that increase the chance of passing and that the plant could tolerate for continued operation. The latter would be less desirable and would not be cost efficient if the plant passed under worst case conditions. Therefore, it may be possible to test only under worst case conditions. If the plant fails the RCRA requirements, then perhaps the contingency plan could be to request a variance and retest under the other set of operating conditions, as soon as possible after the results from the first test are available.

## SECTION IV

### CONDUCTING THE TRIAL BURN

Many questions arise when preparing for and conducting a trial burn. Some questions which may be asked are:

- A. What is involved in preparing for the tests?
- B. What is involved in conducting the actual sampling?
- C. What is involved in analysis of samples?
- D. How are sampling and analysis data converted to final results?
- E. How are the data and results usually reported?

Each of the above questions are broad and cover many specific items. Subsequent sections of this manual will attempt to address these questions by discussing specific areas that are most important and areas that may cause problems. In formulating answers, it has been assumed that the incinerator operator has obtained all necessary approvals of the Trial Burn Plan and is preparing to implement that plan. At that point, the realities of the test come to the forefront and answers are needed to many questions like those discussed below.

#### A. WHAT IS INVOLVED IN PREPARING FOR THE TESTS?

Preparations for the test are numerous; several of the most important items are scheduling, sampling crew activities, equipment preparation and calibration, facility readiness, process data, data sheets and labels, and safety precautions. One potential problem that should be addressed during preparation is how to coordinate with observers during the trial burn.

##### 1. Schedule

Many scheduling problems can occur if the lead time required is not anticipated. These include:



<u>Item</u>	<u>Time Often Required</u>
Make additions or revisions to Trial Burn Plan	Varies
Acquire all wastes needed	Varies
Select test contractor	3 months before test
Pretest site visit by contractor	1 month before test
Notify all regulatory agencies	1 month before test
Install necessary sampling access	complete 1 week before test
Test contractor begins preparation	2-3 weeks before test
Numbering, weighing all drum solid feed	2-3 days before test
Conduct test	3+ days
Sampling equipment teardown	1 day after last test
Analysis of samples	1-1½ months after test
Report of results from contractor	2 months after test
Submit results to EPA	3 months after test

If a miniburn or other preliminary testing is involved, it should be done at least 2 months before the Trial Burn, which would increase some of the required time intervals shown above.

## 2. Sampling Crew

If a sampling/analysis contractor is used, that contractor will have to make crew selections and assignments at least 2 to 3 weeks prior to the test. Many of those crew members and the analytical personnel will perform the complex activities for equipment preparation and calibration, preparation of all absorbent traps (MM5 and VOST) and special cleaning of all sampling containers. Other logistical arrangements for transporting equipment and personnel to the site must also be made. For these reasons, a firm date for the test should be established, in concert with the S&A contractor, at least 1 month prior to the test.

## 3. Equipment

The large amount of sampling equipment needed for a Trial Burn is usually surprising to the operator. A list of some of this equipment is shown in Table 10. What that list does not show is the detailed preparation procedures for much of the equipment. For example:

TABLE 10. EXAMPLE LIST OF SAMPLING EQUIPMENT AND SUPPLIES TYPICALLY USED

Method 5 - particulate train

Console with pump heaters  
 Sample box with pump heaters  
 Umbilical-sampler hookup (gooseneck)  
 Umbilical cords and adapter (elec.)  
 Probe tips (4/set)  
 Probes (type-SS, glass)  
 Extra quartz inserts  
 Extra glass inserts  
 "S" type pitot (ft-5)  
 Rails - dexangle  
 Port-rail clamps (collar size 4")  
 Probe-support  
 Sample box guide attachment  
 Potentiometer  
 Spare thermocouples  
 Intercom (with cable)  
 Manometer, inclined  
 Manometer "U"  
 5-impinger foam inserts (for sample box)  
 Digital pyrometer  
 Umbilical thermocouple adapter  
 Submersible pumps  
 Latex tubing for condensers  
 Glass tape (high temp)  
 Console supply briefcase

Method 5 - glassware

Aluminum cases  
 Impingers  
 "U" connector  
 90° connector  
 Cyclone  
 Filter holder with frit  
 Cyclone bypass, 90°  
 Teflon sleeves (45/50)  
 2-liter impinger bottle  
 Condenser  
 XAD's  
 U-tubes  
 2 L bottle foam inserts  
 Socket flask - 500 ml  
 Plastic caps for probe ends  
 Miscellaneous clamps, gaskets,  
 stoppers

Method 4 - moisture train

Probe  
 Midget impinger  
 Midget connectors  
 Glass wool  
 Silicone grease  
 Micrometer valve  
 Vacuum pump  
 Vacuum gauge  
 Impinger box/ice bath  
 Dry gas meter w/thermometer  
 Spring clips  
 Vacuum tubing  
 Stopwatch

Integrated gas train

Grab sample  
 Probe  
 Squeeze bulb  
 Gas bags  
 Integrated gas bag train  
 Probe  
 Midget impingers  
 Micrometer valve  
 Pump  
 Rotameter  
 Box w/bag insert  
 Bag  
 Pitot tube (S-type)  
 Inclined manometer  
 "U" manometer (H<sub>2</sub>O)  
 Vacuum gauge  
 Purge fitting  
 Miscellaneous tubing  
 Glass wool  
 Sealant  
 Analysis  
 Orsat analyzer  
 Spare Orsat parts

VOST equipment

Teflon line  
 Rotameter

TABLE 10. (continued)

VOST equipment (concluded)

Spare 602 polymer  
Probes  
Dry gas meter  
Manifold  
VOST - glassware and fittings  
  Condenser  
  Fittings  
  Impinger  
  Teflon tubing  
  Glass stopcock  
  Glass tapered joint  
Spare glassware  
Teflon sleeves for glassware  
Tenax traps  
Clean coolers

Fyrite O<sub>2</sub> and CO<sub>2</sub>

■ Fyrite (21% scale)  
CO<sub>2</sub> Fyrite (20% scale)  
Sample pumping line consisting of:  
  - hose from probe to filter  
  - filter  
  - aspirator bulb w/check valve  
  - rubber connector tip  
Spare parts  
  Filtering yarn  
  Diaphragm  
  Gaskets  
Spare chemicals  
Orsat - stand  
O<sub>2</sub> buret  
CO<sub>2</sub> buret  
Manifold  
Graduated buret  
Leveling bottle w/tubing  
Orsat sampler  
Mylar bags

Continuous monitors

Conditioning manifold  
CO analyzer  
CO analyzer  
O<sub>2</sub> analyzer

Recorder  
Nitrogen cylinder  
Air cylinder (THC)  
H<sub>2</sub>/N<sub>2</sub> cylinder (THC)  
Calibration gases  
Gas regulators  
Teflon tubing  
Ascarite trap  
Silica gel trap  
Silica gel  
Ascarite  
Heated lines  
Controller  
Variacs  
Probe filter  
Parts box

Safety equipment item

Hard hats  
Hard hat liners  
Safety shoes  
Safety glasses - goggles  
Slip on shields for glasses  
Ear plugs  
Neutralizer  
Ear protectors  
Face masks (full face respirators)  
Dust respirators  
Climbing belt and lanyard  
Gloves high temperature  
Rain gear  
First aid kit  
Water jug  
Saf-T-Lok  
Fire extinguisher  
Face shield  
Viton gloves  
Jumpsuits  
Restricted area sign  
Black and yellow ribbon  
Tarps  
No smoking sign  
Respirators  
Cartridges - organic vapors and acids  
Eyewash bottle  
Fire blanket

TABLE 10. (continued)

Computer and associated items

Hardware -  
 Pocket computer  
 Printer cassette interface  
 Cassette recorder  
 Cassette to interface connecting cable  
 Ribbon cartridge  
 Printer paper  
 Level II basic reference manual  
 Extra blank cassette  
 Software  
 Program printout copies

Laboratory-general equipment

Lab tool box  
 Oven  
 Kimwipes  
 Chix wipes  
 Wash bottles  
 Glass wool  
 Barometer  
 Plastic for lab floor  
 Graduated cylinders  
 Pipettes  
 Funnel glass  
 Beakers  
 Thermometer (0-125)°C  
 Bulbs for disposable pipettes  
 Brushes and soap  
 pH paper  
 Ultrasonic cleaner  
 Triple beam balance w/weights  
 Ramrods with brushes  
 Wash tub  
 Filter holder clamps

Laboratory-chemicals

## General -

Orsat chemicals:  
 3 oz Oxorbent (O<sub>2</sub>)  
 3 oz Cosorbent (CO)  
 3 oz disorbent (CO<sub>2</sub>)  
 3 oz burette solution

Distilled-deionized H<sub>2</sub>O double-distilled  
 B and J Acetone (1 pt/part. Tests) gal.  
 Silica gel (2 run/lb) (large can)  
 Spare Fyrite chemicals  
 Particulate (Method 5)  
 Filter paper (glass fiber)  
 Sample bottles (glass)  
 B and J methanol  
 60 ml poly bottles w/caps  
 0.1 n KOH

General items

Spare hardware equipment  
 Electrical tool box  
 FM 2-way radio  
 Heat gun  
 Sample labels  
 Ice bags  
 Lab notebooks  
 Air tank for blow-back  
 Portable welding unit  
 Label tape and dispenser  
 Data sheets  
 SOP's  
 Traceability sheets  
 Timers for consoles  
 Rubber bands  
 Clipboards  
 Paper clips  
 Scotch tape  
 Masking tape

- Calibrate all MM5 consoles.
- Condition and check all VOST traps.
- Clean and pack all MM5 resin traps (XAD).
- Preweigh all MM5 filters.
- Clean all glassware and sample bottles.
- Purchase special reagents and solvents.
- Modify equipment for special sampling situations.
- Reserve time for use of analytical instrumentation.
- Collect and pack all necessary field laboratory equipment.

In many cases, the complexity of the sampling plan or lack of available plant facilities requires provision of a large field trailer for the samples and sampling equipment. This trailer also serves as the field laboratory.

#### 4. Facility Readiness

Facility readiness is critical to conducting the test as scheduled. Checking operational readiness of the incinerator and its components, including critical instrumentation (especially flow meters), is vital and should be done early enough to correct any problems identified. When the plant is operated under worst case conditions, unanticipated problems often occur. The plant should be operated under test planned conditions prior to the tests to minimize costly delays during the test.

Other facility readiness needs are identified during the pretest site survey. This survey will identify most of the sampling needs, especially those related to the stack sampling ports and sampling platform, which can require some installation work by the plant. Frequently the contractor will need to rent a trailer to be used on site for sample workup and storage. A suitable location for the trailer should be identified during the survey. The survey also will identify other needs such as electrical supply requirements. (These requirements are usually much larger than the plant expects.) The survey should be conducted at least 1 month prior to the test to allow time for modifications to the facility.

Facility readiness also includes preparation of all the wastes to be used in the tests. Waste preparation is especially important for drummed solid waste. It is highly desirable to have all drummed waste on site at least a week prior to the test and have the drums arranged in a staging area in the order that they will be used. These preparations will facilitate numbering, weighing, and sampling of each drum. Drum preparation can require considerable time and effort on the part of the plant operating staff and the sampling crew.

#### 5. Process Data

Process data recorded during a Trial Burn is of equal importance with the sampling activity, for two reasons:

- Process data are necessary for computation of DRE.
- Some process readings recorded during the Trial Burn may, and probably will, become the limits specified in the operating permit (e.g., minimum combustion chamber temperature).

The average feedrate of each waste and fuel input stream must be determinable for each run by flowmeter, tank level change, drum weights, etc. Those feed rates, and analysis results for waste feed samples are used to compute thermal input rate (BTU/hr) and POHC input rate. These parameters are used to calculate DRE.

Three steps for process monitoring are recommended:

1. Before the test, determine what process data must be taken and reported to EPA.
2. Record all possible data, to help identify any problems during the test, or in the test results.
3. Before the test, establish the acceptable range for each critical operating parameter.

Item 3 above is not as simple as it may appear. For example, combustion chamber temperatures for the Trial Burn might be  $2000^{\circ} \pm 50^{\circ}\text{F}$ . Questions then arise as to what if the temperature range is not maintained at all times during the test:

- Is sampling to be interrupted if the temperature goes outside the established range?
- How long can the temperature be outside the range, or how far outside the range, before ordering an interruption in sampling?
- If sampling is interrupted, how long must the temperature be back within range before sampling can be restarted?

This one example demonstrates the complexity of questions that frequently arise. These questions should be anticipated and guidance developed before the Trial Burn to assure trial burn operating conditions that meet plant needs. There is often precious little time to make those decisions when the questions are faced during a test.

## 6. Data Sheets and Labels

Preparation of all data sheets and sample labels that will be needed for the test is important.

Many different data sheets are needed for a Trial Burn as shown Table 11. The units of measure must be shown for every item on every data sheet. Too often, data are taken (i.e., numbers recorded) without showing the units of measure (e.g.,  $^{\circ}\text{F}$ , gal/min, etc.). Instrument factors (e.g.,  $\text{Rdg} \times 100 = ^{\circ}\text{F}$ ) should be noted during trial burn preparation to assure that data are accurately compiled. Data sheets may be a better record than copies of strip charts, since the latter do not show units of measure or multiplication factors and are often difficult to interpret for other reasons.

All data sheets should be prepared before the test to ensure that all necessary data are recorded. Specific assignments should be made as to who

TABLE 11. EXAMPLE LIST OF DATA FORMS

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Traverse Point Locations  
Preliminary Velocity Traverse  
Method 5 Data Sheets  
Isokinetic Performance Work Sheet  
M5 Sample Recovery Data  
Integrated Gas Sampling Data (Bag)  
Orsat Data Sheet  
VOST Sampling Data

Drum Weighing Record  
Drum Sampling Record  
Liquid Waste Feed Sampling Record  
Fuel Oil Sampling Record  
Drum Feed Record

Process Data (Control Room)  
Miscellaneous Process Data (In-Plant)  
Tank Level Readings

Log of Activities

Ash Sampling Record  
Scrubber Waters Sampling Record  
Sample Traceability Sheets  
GC/MS Data Calculation Sheets

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Note: Units of measure must be shown for each item on each data sheet.

is responsible for each sheet. The data to be recorded can be identified during the pretest survey, and the data forms prepared thereafter.

Sample labels should also be prepared prior to the test. Labels can be prepared most efficiently using computer printed labels like the example shown in Figure 3. Replicate labels which each contain all essential information including a unique sample number for each sample are printed. Replicate labels are needed in order to place one on the sample container, one on the outside of the final packaged sample, one in the field laboratory log book and leave the fourth on the sheet of labels. The last label provides a quick way of checking which samples were taken, since some changes may be made in the field. Blank labels are also always provided for such changes or additional samples that may be taken.

Preparing the computerized labels requires careful thought to identify each and every specific sample that will be taken during each run, including all replicates and blanks. Label preparation also helps identify all the sizes and types of sample containers that will be needed, how they must be prepared, and the number of each that is required (including spares). This activity often shows that 50 to 100 individual samples (and labels) will be involved in each run. Given this magnitude of samples, preprinted labels with specific sample names and a consistent numbering system should be prepared before the Trial Run to help avoid confusion and errors that can occur if labels are prepared later in the field.

## 7. Safety Precautions

Most plants and sampling crews utilize common safety equipment such as safety glasses, steel-toed shoes and hard hats. However, an outside contractor's sampling crew needs to be made aware of all plant safety requirements and any special hazards that may exist, especially with regard to particularly toxic components in the feed streams or the stack effluent (e.g., high CO levels).

Sampling personnel need to be instructed on any special safety equipment and procedures for liquid waste sampling or sampling of any other hazardous waste feeds. The need for protective equipment such as specific types of gloves, goggles, respirators, chemical resistant suits, etc., should be established early enough in the planning stage so that the sampling crew can prepare for their use. Once at the test site, plant personnel must ensure that the sampling personnel are informed of the plant's safety procedures, especially if they could impact on the test program (e.g., evacuation of the sampling area caused by a process upset in an adjoining portion of the plant).

One special note about safety for sampling of drummed wastes is encountering "bulging" drums. A bulging drum can, of course, indicate pressure buildup in the drum. If a bulging drum is encountered in the course of drum sampling, only experienced plant personnel should attempt to open it. Furthermore, a drum can be under pressure, even if it is not bulging. It is recommended that plant personnel be assigned to assist the sampling crew and be responsible for opening all drums to be sampled.



RUN # 1                    101 Liq. Organic Waste Feed  Proj. #            DATE: Plant Name	RUN # 1                    101 Liq. Organic Waste Feed  Proj. #            DATE: Plant Name	RUN # 1                    101 Liq. Organic Waste Feed  Proj. #            DATE: Plant Name
RUN # 1                    102 Aqueous Waste Feed  Proj. #            DATE: Plant Name	RUN # 1                    102 Aqueous Waste Feed  Proj. #            DATE: Plant Name	RUN # 1                    102 Aqueous Waste Feed  Proj. #            DATE: Plant Name
RUN # 1                    103 Kiln Ash Sluice Water  Proj. #            DATE: Plant Name	RUN # 1                    103 Kiln Ash Sluice Water  Proj. #            DATE: Plant Name	RUN # 1                    103 Kiln Ash Sluice Water  Proj. #            DATE: Plant Name
RUN # 1                    104 Liquid Scrubber Effluent  Proj. #            DATE: Plant Name	RUN # 1                    104 Liquid Scrubber Effluent  Proj. #            DATE: Plant Name	RUN # 1                    104 Liquid Scrubber Effluent  Proj. #            DATE: Plant Name
RUN # 1                    105 VOST Trap Pair #1, Tenax Proj. #            DATE: Plant Name	RUN # 1                    105 VOST Trap Pair #1, Tenax Proj. #            DATE: Plant Name	RUN # 1                    105 VOST Trap Pair #1, Tenax Proj. #            DATE: Plant Name
RUN # 1                    106 MMS Caustic Solution  Proj. #            DATE: Plant Name	RUN # 1                    106 MMS Caustic Solution  Proj. #            DATE: Plant Name	RUN # 1                    106 MMS Caustic Solution  Proj. #            DATE: Plant Name

Figure 3. Example of computer labels.

## 8. Observers

The operator frequently does not realize until a test starts that trial burns bring out everyone with a vested interest and even those who are just interested. Observers may include regulatory authorities, extra operating and maintenance personnel, responsible plant management, and many others who otherwise are seldom present. They all usually congregate in the control room. These "observers" usually have reason to be present, but their numbers can create problems.

Observers want to ask questions and to have lengthy discussions with the plant operators. Some of this may be necessary, but it can divert the operators' attention from their primary function and responsibility. Similarly, the observer may want to ask questions of the sampling crew at times when they must give their full attention to their sampling responsibilities. Also, suggestions made by "observers" to operators or samplers are sometimes interpreted as a directive to change how they are doing something.

To help avoid the above problems, the following should be done prior to the trial burn:

- Instruct all operating personnel and samplers not to make any changes unless directed to do so by their supervisor or other designated individuals.
- Require that each observer minimize discussion or interference with operators or samplers during busy periods, especially during test periods.
- Assign one plant person as the primary contact for all observers, and request that the observers direct questions and comments to that person first.
- Since most observers are interested in what is being done and how it is being done, have descriptive material available and, if needed, make arrangements for them to discuss the test plan and sampling/analysis methods with appropriate personnel at appropriate times before or after the actual test periods.

### B. WHAT IS INVOLVED IN CONDUCTING THE ACTUAL SAMPLING, AND WHAT ARE THE PROBLEMS THAT MAY OCCUR?

The main factors involved in the actual sampling for a trial burn are:

- Equipment setup
  - Sampling train setup
  - Setup waste feed sampling

- Preliminary testing
  - Velocity traverse
  - Cyclonic flow check
  - Moisture measurements
- Actual testing
  - Waste feed sampling
  - Process monitoring and determination of waste feed rates
  - Sampling of ash and scrubber waters, etc.
  - Stack sampling
  - Sample recovery
  - Labeling and sample packaging/storage
  - Preparation of equipment for next run
- Equipment dismantling and packing

Brief discussions of the above items and procedures to avoid problems that may occur, are presented below.

### 1. Equipment Setup

#### Sampling Train Setup--

The first job of the sampling crew after arriving at the facility is unloading and setting up of equipment (usually into a field laboratory trailer) including setup for the stack sampling. Setup on the stack for MM5 is usually the most difficult step. First, relatively heavy equipment (40 to 80 lb) must be moved up to the sampling platform. Second, support rails or a monorail must be installed to allow the MM5 train to traverse the stack. These rails often must extend outward from the port a distance equal to the stack diameter plus about 2 ft. The rails must be rigidly secured to support the sampling train over its entire length (~ 8 ft) and allow free movement with no interfering objects (e.g., guard rails). The problem usually encountered is the lack of means to support the rail, especially at the outer end which may extend 4 to 8 ft further out than the platform. (Some platform designers assume that a 6-ft diameter stack can be sampled from a 2-ft wide platform). Also, the sampling ports are almost always at about the same level as the platform guard rail, so part of the guard rail must be removed to provide clearance for the sample box, if not done earlier.

Stack samplers have necessarily developed various means of supporting the rails, but each test site always requires something slightly different. A pretest survey may identify some modifications the plant can make to facilitate the stack setup, consistent with the design of the rail system to be used. But ideally, the width of the platform would be at least equal to the diameter of the stack plus 2 ft.

Another common problem encountered during stack setup is inadequate electrical outlets. As a minimum, at least four 110-v, 20-amp electrical outlets should be available on the stack sampling platform. If at all possible, these circuits should be dedicated to the test, without interference from other plant equipment.

### Setup for drummed waste sampling--

The equipment setup period may include numbering, weighing, and sampling of drummed wastes. Plant personnel will be needed to assist in this activity. Adequate time must be set aside for this work, and the plant needs to preplan the work so that the wastes are properly staged and equipment provided (scales, forklift, etc.). Preparation of drummed waste is done most efficiently with 2 or 3 person teams. Each team can process a drum in 2 to 5 min.

## 2. Preliminary Testing

### Velocity Traverse--

One of the first preliminary tests is a preliminary velocity traverse the stack and determination of stack moisture content. Values obtained are used to determine sampling train conditions, so the preliminary measurements need to be made at test conditions. This preliminary test will require additional time on the first test day.

### Cyclonic Flow--

Another preliminary test is a check for cyclonic flow in the stack, as required by EPA Method 1. Sampling under cyclonic flow conditions requires special equipment and methods. Alternatively, a flow straightener can be installed in the stack, as far upstream of the test ports as possible. This installation could involve considerable delay to design, fabricate, and install the flow straightener. Installation may require a plant shutdown. If there is any reason to suspect that stack flow may be cyclonic, then the plant should either have the flow checked or install a flow straightener well in advance of the tests to avoid the possibility of having to delay testing at the last minute.

### Moisture Measurements--

Another possible problem is high moisture content of the stack gases from wet scrubbers. For scrubber stack test, crews should determine moisture content at saturated conditions prior to the particulate run to ensure that the runs are conducted under isokinetic conditions.

## 3. Actual Testing

Sampling usually consists of taking representative samples of all influent and effluent streams, especially waste feeds and stack effluent, during each of three runs. Process monitoring, including collection of data needed to determine all waste feed rates, is also done during each run. When recording process data, common practice is to read and record the instrument readings once every 15 min, even if they are also continuously recorded. These manual readings provide a good written record for inclusion in test reports. The results reported should include notation of momentary excursions. Otherwise, the operating permit might not contain any allowance for those types of occurrences that are a part of normal plant operations.

During the test one person who knows the conditions under which sampling should be interrupted, and who is in radio contact with the stack

test crew at all times should be responsible for process monitoring. That person must notify the crew to interrupt sampling whenever deemed necessary, but especially when a serious process upset or a shutdown occurs during a test. Such transient conditions could have a drastic impact on the samples, so all sampling must be stopped immediately. In that event, sampling can be resumed after the desired plant operating conditions have been reestablished.

Immediately after each run, the sampling crew must recover all samples, properly label each, and package them for storage and shipment. Samples are usually double-bagged with protective wrapping and stored in coolers, many of which must be iced each day. After that work has been completed, all sampling equipment must be prepared for the next run. All this may sound relatively simple, but there are numerous problems that can occur. Some of these problems are listed in Table 12 and are briefly explained below.

TABLE 12. POTENTIAL PROBLEMS THAT  
MAY OCCUR DURING TESTS

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Plant operating problems
Determination of waste feed rates
Weather
Sampling equipment problems
High filter vacuum due to filter loading

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Plant operating problems during a run are not uncommon, and are due in part to the fact that the plant is being operated under "worst case" conditions. This scenario may cause operating conditions to go outside the specified range for the test, or upsets may occur to cause a plant shutdown. Such situations require interruption of all sampling until desired conditions are reestablished.

Any interruptions in sampling, for whatever reason, can have an impact on proper determination of waste feed rates, especially liquid wastes. This may not be much of a problem if the plant is equipped with reliable waste flow meters. But, if tank level changes are the basis for determining feed rate, then levels have to be read whenever the sampling is interrupted and again when it is restarted.

Another problem that can often occur during testing is bad weather. This can alter plant operating conditions, but usually it impacts the sampling activity. Heavy rain, lightning, or high winds are common conditions that will require interruption in sampling, since it is unsafe for the sampling crew to remain up on the stack unless it is be enclosed.

Most of the other types of problems that occur during a test relate to the stack sampling equipment. Equipment used to conduct stack sampling per Method 5 include pumps, heaters, thermocouples, etc., all of which are subject to mechanical failure. Another type of problem is high pressure drop across the sampling train, usually caused by material on the particulate filter used in the train. When this occurs, sampling must be interrupted for 30 to 60 min to change filters.

The most serious problems related to the stack sampling are failure to achieve prescribed sampling rate (i.e., isokinetic sampling) or failure to pass final leak checks. Either situation can invalidate a run, probably requiring that it be repeated. The plant must be aware that this can happen, requiring additional quantities of waste and additional time.

C. WHAT IS INVOLVED IN ANALYSIS OF SAMPLES AND WHAT ARE THE PROBLEMS THAT MAY OCCUR?

Even in a relatively simple trial burn, 100 to 200 samples may be acquired for analysis. Each sample will need to be analyzed for several different parameters (HHV, Cl, ash) and several analytes (POHCs). Numerous problems can occur in the analysis phase of a trial burn due to the complexity of sample analysis, the variety of sample types, and the detection limits required for some samples. Most frequently, these problems occur in the analysis of samples for POHCs. Some of the most common problem areas are:

- Sample check-in
- Analysis directive
- Sample inhomogeneity
- Analytical interferences
- Saturation of GC/MS detection instrumentation

Usually the analytical results are reported to the project leader who is responsible for using them to calculate final results (DRE) and to prepare a test report. The project leader first examines the analytical results. Potential problem areas which may appear in the data are:

- High blanks
- Poor precision for analysis of replicate samples
- Poor accuracy for recovery of surrogate recovery components spiked into the sample prior to analysis
- Nonconformance with "expected" results

Each potential problem area is discussed below.

## 1. Sample Check-in

Samples taken during a trial burn are usually brought to the analytical laboratory for transfer to an analytical task leader. At that point, when the samples are checked in and transferred, the project leader needs to cross-check that each sample taken in the field has arrived and is intact. Any missing samples can in this way be immediately identified and hopefully located. Also, any extra samples that have been taken can be identified. The sample information should be recorded in a bound laboratory record book (LRB) and sample traceability sheets should be used, even if strict chain of custody sheets are not used.

## 2. Analysis Directive

After completing sample check-in, the project leader should prepare a written directive for sample analysis specifying the following for each and every sample:

- Sample number and type of sample (e.g., 141 - waste feed).
- Notation of any safety or hazard considerations in handling/analyzing samples.
- Analysis parameters and analytes.
- Analysis methods for each parameter or analyte.
- Analysis sequence (where necessary).
- Indication of whether sample is to be analyzed in duplicate (or triplicate).
- Samples to be spiked with analytes and surrogate recovery compounds for determination of percent recovery efficiency.

The directive should also indicate expected concentrations, analysis priorities, and the schedule for reporting of analysis results.

Preparation of the above directive is complex but very important. It documents the analysis needs and will be very helpful to those who must perform the analyses. Most importantly, it helps assure that all the necessary analyses will be done as needed to satisfy the trial burn requirements.

The analysis sequence can be especially important for MM5 samples. If the same sampling train is used to collect particulate and POHC samples, the particulate filter must be desiccated and weighed prior to extraction for POHC analysis. Another example is analysis of a filter for POHCs and metals. Since one precludes the other, it would probably be necessary to cut the filter in half. Such examples are presented here to demonstrate the importance of written specifications for the sequence in which analyses must be done.

### 3. Sample Inhomogeneity

When analyses of samples are initiated, a problem often encountered is nonhomogeneity of samples, especially waste feed samples. Liquid waste feeds often separate during shipment/storage. Steps must be taken to homogenize samples before portions or aliquots are removed for analysis. Solid waste samples often present even more difficult problems that should be discussed with the analysis task leader.

### 4. Analytical Interferences

Waste feed samples are organically complex and usually contain many constituents other than POHCs. These other constituents may interfere with the POHC analysis, especially if the POHC concentrations are low in comparison to the other constituents. Some type of sample cleanup may be required.

### 5. Saturation of GC/MS Data System

Saturation of the GC/MS data system may occur for any type of sample, but especially VOST traps, VOST samples are analyzed by thermal desorption, and cannot be prescreened or diluted. If saturation occurs, gas bag samples must be analyzed. Saturation can be a complex problem if there are several volatile POHCs with a wide range in their anticipated concentrations. VOST results may be essential for determining DRE of some POHCs present at low concentrations in waste feeds, but those POHCs may be masked by high concentration of other POHCs in the VOST samples. Such problems are best avoided by careful preplanning, including selection of POHCs and/or their concentrations in the waste feeds.

### 6. High Blanks

High blanks may occur for any parameter or analyte, but probably more frequently for POHCs. Blank levels can be used to "correct" sample levels, but this is not possible if blank levels are near or exceed sample levels. If both the blanks and sample levels are high, causing the uncorrected sample value to yield a DRE below 99.99%, no useful information is obtained for that sample. Every precaution must be taken in the laboratory and in the field to prevent contamination of samples.

### 7. Poor Precision

The QA protocol usually requires triplicate analysis of critical samples from at least one run. Wide variability in these triplicate analysis results may occur if samples were not homogeneous, if the samples contain some interfering component, or if other problems occur with the analytical technique. In any case, the precision obtained provides an indication of the possible variability in results reported for each sample and analyte. For POHCs, knowledge of this variability may be quite important if the calculated DREs are close to 99.99% (e.g., 99.989%).



## 8. Recovery Efficiency

Another normal part of the QA/QC is spiking of samples with known amounts of POHCs and/or surrogates to determine recovery efficiency for the analysis method. The desired recovery range is normally 70 to 130%, but actual results may in some cases be much lower or much higher for any of several reasons. Sample results usually are not corrected based on recovery efficiency results, but knowledge of the recovery efficiency is important for the same reason as knowledge of the precision. Also, poor precision or poor recovery efficiency may indicate a need to reanalyze the samples.

## 9. Actual Versus Expected Results

In certain cases the project leader has some idea of what the analytical results should be. For example, the amount of POHC added into a waste feed tank might be known, so there is some expected concentration of the POHC in those samples. Usually the analytical results are in good agreement with expected results, but in some instances the analytical results may disagree with the expected value. The analyses and calculations then must be rechecked, and the QA/QC data scrutinized more closely for a clue. Mathematical errors are the most common cause of such problems, but other possible causes, such as a poorly mixed feed tank, cannot be overlooked.

## D. HOW ARE THE SAMPLING DATA AND ANALYSIS DATA CONVERTED TO FINAL RESULTS?

This section addresses the calculation methods used to convert laboratory data on organics, and field data on flow rates, into DRE numbers. The values necessary to calculate DREs, and how they are obtained, are listed in Table 13. A brief review of the method used to calculate DRE is presented at the end of this section. First, however, it is necessary to give attention is given to the areas of blank correction, significant figures, rounding of DREs, and the need to use "<" and ">" signs in reporting DRE data.

### 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 Improvement (ACS)<sup>7</sup> 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.

**TABLE 13. DATA NECESSARY FOR CALCULATING DRE**

Measured value	Example units	How value is obtained
Mass flow rate of feed	g/min	Measured during test or calculated from flow and density.
Volumetric flow of feed	L/min	Measured during test.
Density of feed	g/mL	Density analysis from lab.
Concentration of POHC in feed	µg/g	Analysis of waste feed samples
67 Total quantity of POHC in stack sample	µg	Reported by lab for each sample taken during test.
Volume sampled of stack gas	Nm <sup>3</sup>	For VOST this is found in the sample train data. For M5 this is found with the M5 train data. For gas bags this is reported as the volume analyzed by the laboratory.
Total quantity of POHC in blank samples	µg	Analysis of "blanks"
Volumetric stack flow rate	Nm <sup>3</sup> /min	Reported as result of pitot traverse with M5 train

Note: 1 µg = 10<sup>-6</sup> g  
 1 ng = 10<sup>-9</sup> g  
 1 µg/m<sup>3</sup> = 1 ng/L  
 Nm<sup>3</sup> = normal cubic meters = dry standard cubic meters

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.

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:

		<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>
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 different than the trip blanks use the field blank data only. If the blanks are not significantly different use all of the blank values.

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 (std. 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. 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:

$$\text{DRE} = 100\% - \text{Penetration}$$

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.

## 3. Rounding Off DRE Results

The rules on this are stated in the Guidance Manual for Hazardous Waste Incineration Permits:- ". . . if the DRE was 99.9880 percent, it could not be rounded off to 99.99 percent." 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%.)

## 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, alternate sampling methods should be used, based on preliminary estimates of the stack concentrations that may exist.

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.

#### E. HOW ARE THE DATA AND RESULTS USUALLY REPORTED?

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 Tables 14 through 25. Using part of the data in these tables, an example calculation of DRE is shown in Table 26.

TABLE 14. INCINERATOR OPERATING CONDITIONS<sup>a</sup>

Parameter	Run 1, 11/3/82	Run 2, 11/4/82	Run 3, 11/4/82
Organic waste flow rate, <sup>b</sup> kg/min (lb/hr)	3.76 (497)	4.01 (542)	4.50 (595)
Aqueous waste flow rate, <sup>b</sup> kg/min (lb/hr)	6.13 (811)	5.38 (712)	4.90 (648)
Heat input rate, GJ/hr (10 <sup>6</sup> Btu/hr)	8.37 (7.93)	9.01 (8.54)	10.50 (9.96)
Combustion chamber temp., °C (°F)	1053 (1925)	1066 (1950)	1094 (2000)
Calculated residence time, <sup>c</sup> sec	2.5	2.4	2.2
Stack height, m (ft)	11.6 (38)	11.6 (38)	11.6 (38)
Stack exit velocity, m/s (fpm)	10.7 (2,110)	10.3 (2,030)	11.3 (2,230)
Stack temperature, °C (°F)	810 (1490)	749 (1380)	766 (1410)

<sup>a</sup> Data collected by reading plant monitoring instruments at regular intervals. Values shown are averages for each run.

<sup>b</sup> Determined by measuring storage tank liquid levels at start and finish of each run.

<sup>c</sup> Calculated from chamber volumes and stack flow rates.

TABLE 15. CONCENTRATIONS OF POHCs IN WASTE FEEDS (µg/g)

	Aqueous waste			Organic waste		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
<b><u>Volatile POHCs</u></b>						
Carbon tetrachloride	< 2 <sup>a</sup>	< 2	< 2	6,400	6,000	4,700
Trichloroethylene	< 1	< 1	< 1	5,900	5,500	4,300
Benzene	< 3	< 3	< 3	2.7	260	140
Toluene	94	110	100	1,800	2,400	1,900
<b><u>Semivolatile POHCs</u></b>						
Phenol	42,000	34,000	b	4,200	1,000	b
Naphthalene	< 100	< 100	b	510	350	b

<sup>a</sup> Results reported as less-than values represent limits of detection.

<sup>b</sup> MMS sample voided for this run due to equipment problems. Therefore, the waste feed samples were not analyzed for semivolatile POHCs.

TABLE 16. CALCULATED INPUT RATES FOR POHCs  
IN WASTE FEEDS

	Input rates (g/min) <sup>a</sup>		
	Run 1	Run 2	Run 3
<u>Volatile POHCs</u>			
Carbon tetrachloride	24	25	21
Trichloroethylene	22	22	19
Benzene	0.010	1.1	0.63
Toluene	7.3	10	9.0
<u>Semivolatile POHCs</u>			
Phenol	270	180	b
Naphthalene	1.9	1.4	b

<sup>a</sup> Combined input rates for both waste feeds.

<sup>b</sup> Samples not analyzed for semivolatiles since MM5 sample was voided for this run.



TABLE 17. CONCENTRATIONS OF VOLATILE POHCs BY VOST IN  
STACK EFFLUENT (Not Blank Corrected), ng/L

	Run 1			Average
	1st Pair	2nd Pair	3rd Pair	
Carbon tetrachloride	2.3	0.47	0.57	1.1
Trichloroethylene	20	1.8	1.6	7.8
Benzene	2.2	2.3	2.2	2.2
Toluene	6.2	0.99	2.1	3.1

	Run 2			Average
	1st Pair	2nd Pair	3rd Pair	
Carbon tetrachloride	2.3	1.7	1.7	1.9
Trichloroethylene	17	1.8	1.0	6.6
Benzene	2.0	7.4	2.6	4.0
Toluene	21	7.5	4.3	11

	Run 3			Average
	1st Pair	2nd Pair	3rd Pair	
Carbon tetrachloride	3.1	0.58	0.45	1.4
Trichloroethylene	4.8	0.95	0.66	2.1
Benzene	6.0	7.1	6.2	6.4
Toluene	15	9.7	5.7	10

TABLE 18. VOST BLANK CORRECTION VALUES

	Average blank value (ng)	Standard deviation (ng)
<u>POHCs</u>		
Carbon tetrachloride	< 2	0
Trichloroethylene	< 1	0
Benzene	< 3	0
Toluene	3.7	1.8

TABLE 19. VOST SAMPLE VOLUMES  
(Dry Standard  
Liters)

Run no.	Pair no.	Volume (L)
1	1	18.4
1	3	18.1
1	6	17.5
2	1	18.4
2	3	18.5
2	6	18.5
3	1	18.9
3	3	18.9
3	6	19.0

TABLE 20. BLANK CORRECTION VALUES FOR  
SEMIVOLATILE POHCs

Compound	Blank correction value (µg)
<u>POHCs</u>	
Phenol	3.4
Naphthalene	6.0

TABLE 21. DESTRUCTION AND REMOVAL  
EFFICIENCIES (DREs)

Volatile compounds	VOST		
	Run 1	Run 2	Run 3
Carbon tetrachloride	99.99966	99.99942	99.99946
Trichloroethylene	99.9975	99.9977	99.99906
Benzene	a	99.972	99.914
Toluene	> 99.9973	99.9926	99.9916
Semivolatile compounds	MMS		
	Run 1	Run 2	Run 3
Phenol	99.9985	> 99.99996	> 99.9996
Naphthalene	99.96	99.98	99.986

<sup>a</sup> Waste feed concentration < 100 µg/g in this run.

TABLE 22. MODIFIED METHOD 5 TEST DATA

	Run 1, 11/3/82	Run 2, 11/4/82
Volume of gas sampled (Nm <sup>3</sup> , dry)	2.277	2.101
Sampling time (min)	140	140
Percent isokinetic	95.0	96.8
Moisture content (%)	15.8	13.0
Percent O <sub>2</sub> (dry)	10.5	10.8
Percent CO <sub>2</sub> (dry)	7.8	7.7
Stack flow rate (actual m <sup>3</sup> /min)	355	341
Stack temperature (°C)	809	749
Stack flow rate (Nm <sup>3</sup> /min, dry)	73	76
Particulate concentration		
(mg/dscm)	842	523
(gr/dscf)	0.367	0.228
(mg/dscm corrected to 7% O <sub>2</sub> )	1,125	719
(gr/dscf corrected to 7% O <sub>2</sub> )	0.491	0.313
(lb/hr)	8.1	5.2
Chloride emissions		
(g/min)	31.1	37.1
(lb/hr)	4.1	4.9

Note: Run 3 was voided by a broken probe liner.

TABLE 23. CONTINUOUS MONITORING DATA<sup>a</sup>

	Run 1, 11/3/82	Run 2, 11/4/82
Oxygen (%)		
Range	7.1-11.0	8.3-11.0
Average	9.4	10.5
Carbon dioxide (%)		
Range	7.2-10.6	7.2-8.1
Average	8.5	7.6
Carbon monoxide (ppm <sub>v</sub> )		
Range	< 1-5.8	< 1-5.3
Average	1.4	1.8
Total hydrocarbons (ppm <sub>v</sub> ) <sup>b</sup>		
Range	< 1	< 1
Average	< 1	< 1

<sup>a</sup> Concentrations on dry gas basis.

<sup>b</sup> Total HC reported as propane.

TABLE 24. GENERAL ANALYSIS OF AQUEOUS WASTE

Parameter	Run 1	Run 2	Run 3
Heating value, kJ/kg (Btu/lb)	1,800 780	1,720 730	1,550 660
% Chlorides	0.39	0.36	0.26
% Water	88.54	93.89	89.67
% Ash	0.70	0.78	0.74
Saybolt viscosity (sec)	28.9	28.2	29.5

TABLE 25. GENERAL ANALYSIS OF ORGANIC WASTE

Parameter	Run 1	Run 2	Run 3
Heating value, kJ/kg (Btu/lb)	34,140 14,680	34,390 14,800	37,110 15,960
% Chlorides	1.03	1.26	0.72
% Water	2.15	3.18	5.73
% Ash	1.53	2.13	2.36
Saybolt viscosity (sec)	32.1	30.1	30.3

TABLE 26. EXAMPLE DRE CALCULATION

The following is a sample calculation showing the method used to convert the analytical results to DREs for trichloroethylene in Run 2 using the VOST sample.

$$\text{DRE} = \frac{W_{\text{in}} - W_{\text{out}}}{W_{\text{in}}} \times 100$$

DETERMINE INPUT RATE ( $W_{\text{in}}$ )

$$W_{\text{in}} = \begin{array}{l} \text{(organic waste flow rate} \times \text{TCE concentration)} + \\ \text{Table 14} \qquad \qquad \qquad \text{Table 15} \\ \text{(aqueous waste flow rate} \times \text{TCE concentration)} \\ \text{Table 14} \qquad \qquad \qquad \text{Table 15} \end{array}$$

$$W_{\text{in}} = (4,010 \text{ g/min}) (5,500 \text{ } \mu\text{g/g}) + (5,380 \text{ g/min}) (< 1 \text{ } \mu\text{g/g}) = 22 \times 10^6 \text{ } \mu\text{g/min} = 22 \text{ g/min (Table 16)}$$

CALCULATE OUTPUT RATE ( $W_{\text{out}}$ )

$$\text{Stack flow rate} = 76 \text{ Nm}^3/\text{min (Table 22)}$$

$$\text{VOST concentration avg} = \frac{20 + 1.8 + 1.6}{3} = 7.8 \text{ ng/L (not blank corrected)}$$

(Concentration values taken from Table 17)

Blank correction

$$\text{VOST} = \frac{< 1 \text{ ng/sample (Table 18)}}{18.5 \text{ L/sample (Table 19)}} = < 0.05 \text{ ng/L}$$

$$\text{Blank corrected value} = 7.8 \text{ ng/L} - < 0.05 \text{ ng/L} = < 7.8 \text{ ng/L} = < 7.8 \text{ } \mu\text{g/m}^3$$

VOST output rate

$$\begin{aligned} \text{Mass flow} &= (< 7.8 \text{ } \mu\text{g/m}^3) (76 \text{ Nm}^3/\text{min}) (1 \times 10^{-6} \text{ g/} \mu\text{g}) \\ &= < 0.00059 \text{ g/min (corrected)} \end{aligned}$$

CALCULATED DRE

$$\begin{aligned} \text{DRE} &= \frac{22 \text{ g/min} - < 0.00059 \text{ g/min}}{22 \text{ g/min}} \times 100 \\ &= > 99.9973\% \text{ (Table 21)} \end{aligned}$$

## SECTION V

### REFERENCES

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16. ABSTRACT  The manual concentrates on those aspects of a trial burn that are the most important and those that are potentially troublesome. The manual contains practical explanations based on experience of Midwest Research Institute (MRI) and others in conducting trial burns and related tests for EPA. It includes the comments of several industrial plant owners and operators. It is directed mainly to incinerator operators, those who may conduct the actual sampling and analysis, and those who must interpret trial burn results. It will also be useful for regulatory personnel and others that need to understand trial burns. Potential trouble spots that have been encountered are: (1) trial burns frequently take more time and effort than an operator anticipates; and (2) failure to meet the trial burn requirements.		
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