United States
Environmental Protection
Agency

Industrial Environmental Research
Laboratory
Research Triangle Park NC 27711

Research and Development

EPA-600/S8-84-007 Apr. 1984

EPA

Project Summary

Protocol for the Collection and Analysis of Volatile POHCs Using VOST

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This document provides a state-of-the-art operating protocol for sampling and analysis of volatile organic constituents of flue gas from hazardous waste incinerators or other similar combustor systems using the Volatile Organic Sampling Train (VOST). It is intended to be used for guidance by personnel of the regulatory groups, personnel associated with engineering R&D, and the regulated community.

The VOST is designed to extract and concentrate volatile organic compounds (boiling point $\leq$ 100°C) from stack gas effluents. The concentrated organics are analyzed by procedures chosen to be compatible with the VOST in order to obtain flue gas concentration levels. This information is necessary to perform destruction and removal efficiency (DRE) calculations for incinerator operations. The results of laboratory evaluation and field use of the VOST have shown that the VOST provides sufficient sensitivity to permit calculation of a DRE equal to or greater than 99.99 percent for volatile organics present in the waste feed at 100μg/g.

The VOST is directly applicable to organic compounds with boiling points of 30°C to 100°C. Many organic compounds with boiling points less than 30°C or with boiling points in the 100°C to 150°C range may also be collected and analyzed by this method. Field application of the VOST for compounds with boiling points outside the 30°C-100°C range should be attempted only after laboratory evaluation of the collection and recovery efficiencies of the specific compounds.

The document is presented in two parts. Part A describes the key components of the train, the procedures for preparation of the sorbent materials, and procedures for sample collection using the VOST. Part B describes the procedures for analysis of VOST sorbent cartridges for volatile principal organic hazardous constituents (POHCs) using purge-trap-desorb gas chromatography/mass spectrometry (P-T-D GC/MS). Quality control procedures are presented in both Sections A and B.

This Project Summary was developed by EPA's Industrial Environmental Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Summary

The Resource Conservation and Recovery Act (RCRA) requires that owners/operators of facilities which treat hazardous waste by incineration ensure that the incinerators are operated in a manner which does not endanger human health or the environment. The Code of Federal Regulations, Title 40, Part 264, requires that a destruction and removal efficiency of 99.99 percent be achieved for each POHC designated in the Trial Burn Permit. The DRE standard implicitly requires sampling and analysis to quantify POHCs in the waste feed material and stack gas effluent. The "Sampling and Analysis Methods for Hazardous Waste Combustion" manual (methods manual) provides information on methods that are

applicable for collection and analysis of POHCs in process streams of hazardous waste incinerator units.

The methods manual identifies three possible methods for the collection of volatile organics (those with boiling points of ≤100°C). The methods include bag, glass bulb, and the VOST. Evaluation of the bags and bulbs indicates that these sampling systems are subject to a number of technical problems. The most important of these problems is the inadequate sensitivity for POHCs present in low concentrations.

The VOST provides increased sensitivity to low-level concentrations of volatile POHCs due to its ability to concentrate the gaseous effluent. The results of laboratory evaluation and field application of the VOST have shown that it provides sufficient sensitivity to permit calculation of a DRE equal to or greater than 99.99 percent for volatile POHCs which are present in the waste feed at 100μ g/g.

The methods manual identifies the VOST as a suitable sampling system for volatile organics and includes a paper describing the VOST. A detailed protocol was not included in the methods manual due to the fact that this is outside the scope of the document.

The purpose of this protocol is to provide a standard operating procedure to users of the VOST in the collection and analysis of samples for volatile POHCs in the gaseous effluents of hazardous waste incinerators or gaseous effluents of hazardous waste co-fired combustion processes. The protocol is presented in two parts. Part A describes the key components of the VOST train, and the procedures for sample collection using VOST. Part B describes the procedures for analysis of VOST sorbent cartridges for volatile POHCs using purge-trap-desorb gas chromatography/mass spectrometry (P-T-D GC/MS).

Part A—Sampling

The VOST is directly applicable to organic compounds with boiling points of 30°C to 100°C. Many organic compounds with boiling points below 30°C and between 100°C and 150°C may also be effectively collected and analyzed using the method However, laboratory evaluation of the collection and recovery efficiencies for compounds with boiling points outside the 30°C to 100°C range is required prior to field sampling.

A schematic diagram of the principal components of the VOST is shown in Figure 1 The gas stream is filtered through a glass wool plug in a quartz or glass lined probe. The gas is then cooled to 20°C by passage through a water cooled condenser to facilitate collection of volatile organic POHCs on a pair of sorbent cartridges. Liquid condensate is collected in an impinger placed between the two sorbent cartridges. The first sorbent cartridge contains approximately 1.6 g of Tenax, and the second cartridge contains approximately 1 g of Tenax followed by 1 g of petroleum based charcoal, 3:1 by volume. Isolation valves provide for purging the probe with stack gas prior to sampling and for leak checking the VOST

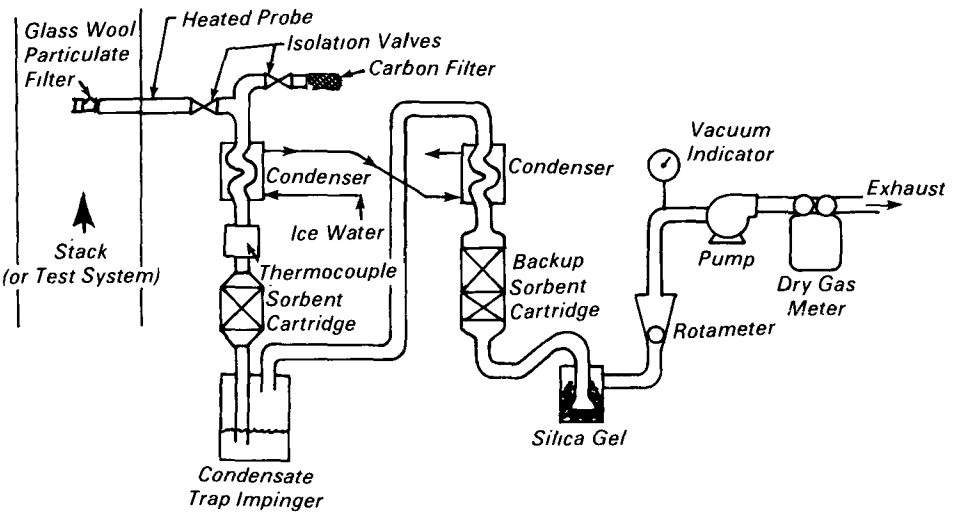

Figure 1. Schematic of Volatile Organic Sampling Train (VOST).

Laboratory data have been developed for a concentration range of 0.1 to 100 μg/m^3 (0.1 to 100 ng/L) for selected volatile POHCs. The concentration range of 100μg/m^3 to 500μg/m^3 (100 ng/L to 500 ng/L) is not expected to pose problems. Although the upper end of the range of applicability is limited by breakthrough of volatile POHCs through the sorbent cartridges used to collect the sample, the range is also limited by analytical constraints. Caution should be exercised in using the VOST to collect samples from a stack gas stream present at high concentrations, 500μg/m^3 (500 ng/L) or greater. The analytical detector may overload during analysis resulting in invalid data. It would be appropriate to collect a reduced sample volume to alleviate the problem. Analytical problems such as peak interferences due to peak tailing or instrument overloading need to be evaluated prior to field sampling.

The VOST is designed to provide six stack gas samples over a 2-hour sampling period. The samples are collected at a sampling flow rate of 1 L/min, using a VOST equipped with a temperature controlled quartz or glass-lined probe.

An alternate set of operating conditions allows for reduced sample flow rate. Operation of the VOST under these conditions has been referred to as SLOW-VOST. This method is used to collect 5 L of sample (0.25 L/min for 20 minutes) or 20 L of sample (0.5 L/min for 40 minutes) on each pair of sorbent cartridges. The SLOW-VOST should be considered for sampling organic compounds with boiling points below 30°C. Fewer pairs of cartridge samples may be required for the SLOW-VOST with a minimum of three sorbent cartridges samples collected.

Part B—Analysis

The method of analysis for volatile POHCs collected on VOST sorbent cartridges is thermal desorption followed by purge-trap-desorb gas chromatography/mass spectrometry (P-T-D GC/MS). Much of the analytical method is described in EPA Method 624. Since the majority of gas streams sampled using the VOST will contain a high concentration of water, the P-T-D analytical method was chosen to minimize the effect of water on the analytical system.

A schematic diagram of the analytical system is shown in Figure 2. Sorbent cartridges are spiked with internal standards and thermally desorbed into the P-T-D system by heating to 180°C for 10 minutes. Sample cartridges may be desorbed in pairs. However, if the analyte concentrations are anticipated to be sufficiently high to saturate the GC/MS

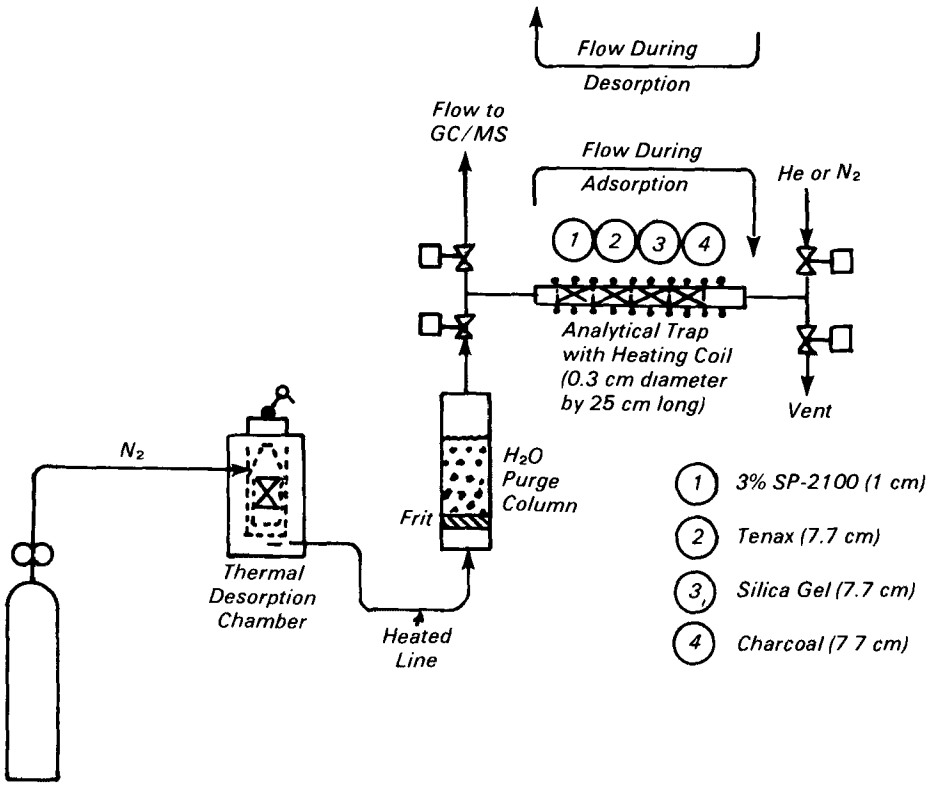

Figure 2. *Schematic diagram of trap desorption/analysis system.*

detector, when desorbed in pairs, consideration should be given to individual analysis of cartridges. The desorbed components pass into the bottom of the water column, and are purged from the analytical trap into the GC/MS. The volatile POHCs are separated by temperature programmed gas chromatography and detected by low resolution mass spectrometry. The quantities of volatile POHCs collected on the sorbent cartridges are calculated using the internal standard technique.

The sensitivity of the analytical method for a particular POHC depends on the level of interferences, the level of background contamination on blank sorbent cartridges, and the purgeability of the POHCs from water. The target detection limit for the method is 0.1μg/m^3 in stack gas. This corresponds to 2 ng adsorbed on a single pair of Tenax and Tenax/charcoal cartridges. The desorption of multiple pairs of cartridges onto a single pair for subsequent analysis may be required to achieve the detection goal of 0.1μg/m^3. Most uses of the VOST have not required this level of detection.

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The complete report, entitled "Protocol for the Collection and Analysis of Volatile POHCs Using VOST," (Order No. PB 84-170 042; Cost: $10.00, subject to change) will be available only from:

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650

The EPA Project Officer can be contacted at:

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