



## Project Summary

# Performance Audit Results for POHC: VOST and Bag Measurement Methods

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Gas cylinders containing a mixture of five organic compounds in a balance gas of nitrogen were used to conduct performance audits to evaluate the accuracy and precision of the sampling and analysis phases of the Volatile Organic Sampling Train (VOST) and bag measurement methods. The five compounds in the audit cylinders were: carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene. The audit cylinders contained compounds in two concentration ranges. A low concentration cylinder in the range of 7 to 90 ppb for each POHC was used for the evaluation of the VOST method. A high concentration cylinder in the range of 90 to 430 ppb for each POHC was used for the evaluation of the bag measurement method.

Thus far, four laboratories have participated in the VOST audits but only two laboratories have been involved in the bag audits. The objectives of the audit program, the audit procedures used, the laboratories audited, the performance audit results, and pertinent conclusions and recommendations are presented in the project report. The audit results indicate that laboratories can analyze POHC with accuracy to within the limits of  $\pm 50$  percent that are stated in the VOST protocol.

Compound stabilities have been determined through multiple analyses of the cylinders contents. Over the 4-month study period, the low ppb audit mixture of five organic compounds was found to be stable. It is recommended that a performance audit using these audit cylinders be conducted during each hazardous waste trail burn test

to assess and document the accuracy and precision of POHC measurements.

*This Project Summary was developed by EPA's Environmental Monitoring Systems 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).*

### Introduction

Under the regulations of the Resource Conservation and Recovery Act (RCRA) of 1976, the Administrator of the U.S. Environmental Protection Agency (EPA) has promulgated performance standards for owners and operators of hazardous waste treatment, storage, and disposal facilities. The RCRA regulations, which deal specifically with hazardous waste incineration, are described in the Code of Federal Regulations, Title 40 Parts 122, 264 and 265. Hazardous waste incinerators are tightly regulated under the Act. The determination of destruction/removal efficiency (DRE) for principle organic hazardous constituents (POHC) requires a reliable sampling and analytical method which may be used with confidence during the hazardous waste trial test burns. EPA is currently recommending a Volatile Organic Sampling Train (VOST) method for collection and analysis of volatile (bp 30-100°C) POHC emissions from hazardous waste incinerators (Ref. 1).

Research Triangle Institute (RTI) was directed by the Quality Assurance Division of EPA's Environmental Monitoring Systems Laboratory, Research

Triangle Park, North Carolina to conduct performance audits to evaluate the accuracy and precision of the sampling and analysis phases of the VOST and bag measurement methods. The main objectives of this audit program are: (1) to demonstrate that cylinder gases can be used to assess and document the accuracy and precision of POHC measurements during trial burns of hazardous waste, (2) to screen the sampling and analysis phases of the VOST/bag methods to determine the principle sources of measurement error, (3) to evaluate the proficiency of laboratories currently using the VOST or bag methods for POHC measurements, and (4) to determine the stability of the POHC at parts-per-billion (ppb) concentrations in gas cylinders. RTI used a multicomponent mixture of five organics in compressed gas cylinders as an audit material to evaluate the objectives of the program at four laboratories. The various activities performed during the study and the results obtained are discussed in the project report.

### Audit Procedures

Gas cylinders containing a mixture of five organic compounds in a balance gas of nitrogen were used as the audit material for this study. The five compounds in the audit cylinders were: carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene. The audit cylinders contained compounds in two concentration ranges. A low concentration cylinder in the range of 7 to 90 ppb for each POHC was used for the evaluation of the VOST method. A high concentration cylinder in the range of 90 to 430 ppb for each POHC was used for the evaluation of the bag measurement method. The audit cylinders were prepared and analyzed by the National Bureau of Standards (NBS) under an Interagency Agreement with EPA. After NBS analysis, the audit cylinders were sent to the Environmental Sciences and Engineering Unit at RTI. RTI analyzed the cylinders before and after conducting the VOST/bag audits to demonstrate the stability of the organic compounds during the audit

period. RTI analysis results were in close agreement with the NBS-measured concentrations (see Table 1). In addition, the NBS and RTI analyses showed the organic compounds to be stable over the entire study period (approximately 4 months).

Performance audits were conducted at four laboratories. The laboratories were: Battelle Columbus Laboratories; PEDCo Environmental, Inc.; Radian Corporation (formerly TRW Environmental Engineering Division); and Engineering Science, Inc. (Sampling was performed by Engineering Science and GC/MS analysis was performed by the Analytical and Chemical Sciences Unit at RTI). All four laboratories participated in the VOST audits but only two laboratories were involved in the bag audits. All participating laboratories were told the name and concentration range of each compound prior to the audit. The proposed audit sequence and general instructions for the sampling and analysis of volatile POHC from Quality Assurance (QA) audit cylinders were sent to each

Table 1. Summary of Audit Results

Activity Audited	Audit Gases	Audit Gas True Conc. and Stability			Lab A <sup>(6)</sup>			Lab B			Lab C <sup>(4)</sup>			Lab D <sup>(6)</sup>		
		NBS Conc. ppb	RTI <sup>(1)</sup> <sup>(2)</sup> <sup>(5)</sup>		Range (ppb)	Average <sup>(5)</sup>		Range (ppb)	Average <sup>(5)</sup>		Range (ppb)	Average <sup>(5)</sup>		Range (ppb)	Average <sup>(5)</sup>	
			10/83 ppb	12/83 ppb		ppb	% <sup>3</sup> Acc.		ppb	% <sup>3</sup> Acc.		ppb	% <sup>3</sup> Acc.		ppb	% <sup>3</sup> Acc.
VOST (Both sampling & analysis)	carbon tetrachloride	21	21.7	21.1	5.9-15.3	10.4	-50	24.5-30.4	26.6	27	22.0-24.0	23.0	10	15.0-21.0	18.0	-14
	chloroform	23	23.8	23.2	21.7-25.2	23.1		28.8-29.8	29.4	28	26.0-30.0	27.7	20	18.0-19.0	18.5	-20
	perchloroethylene	29	30.4	31.1	29.7-36.3	33.9	17	35.4-41.4	38.8	34	--NOT AUDITED--			29.0-36.0	32.5	12
	vinyl chloride	31	34.9	28.0	12.8-18.2	15.5	-50	7.8-13.0	10.5	-66	--NOT AUDITED--			24.0-32.0	28.0	-10
	benzene	18	19.4	20.0	22.9-24.1	23.5	31	22.9-24.8	23.7	32	15.0-17.0	16.3	-9	19.0-19.0	19.0	6
VOST Cartridges analysis only	carbon tetrachloride	21	21.7	21.1	10.4-14.9	12.7	-40	22.6-24.0	23.3	11	22.0-27.0	23.7	13	19.0-20.0	19.5	-7
	chloroform	23	23.8	23.2	23.6-25.0	24.3	6	25.8-27.0	26.4	15	26.0-38.0	30.3	32	18.0-19.0	18.5	-20
	perchloroethylene	29	30.4	31.1	37.5-38.5	38.0	31	30.9-35.8	33.4	15	--NOT AUDITED--			33.0-36.0	34.5	19
	vinyl chloride	31	34.9	28.0	13.1-20.8	16.4	-47	7.7-21.4	14.6	-53	--NOT AUDITED--			32.0-39.0	35.5	15
	benzene	18	19.4	20.0	25.4-27.0	26.2	46	21.5-23.4	22.5	25	21.0-21.0	21.0	17	17.0-19.0	18.0	0
BAG (Both sampling & analysis)	carbon tetrachloride	141	148	149	222-230	225	60	121-124	123	-13						
	chloroform	120	117	117	160-163	161	34	99-103	101	-16						
	perchloroethylene	126	128	136	153-181	165	31	70-73	71	-44	--NOT AUDITED--			--NOT AUDITED--		
	vinyl chloride	154	161	158	86-126	104	-33	96-105	101	-34						
	benzene	133	136	143	147-167	157	18	90-101	97	-27						
Direct Gas Analysis by GC Only	carbon tetrachloride	141	148	149	223-252	238	69	131-135	133	-6						
	chloroform	120	117	117	169-176	173	44	115-117	116	-3	--NOT AUDITED--			--NOT AUDITED--		
	perchloroethylene	126	128	136	97-104	101	-20	116-126	121	-4						
	vinyl chloride	154	161	158	87-136	118	-23	101-107	104	-33						
	benzene	133	136	143	160-180	173	30	117-119	118	-11						

<sup>1</sup> NBS (Gas and Particulate Division) and RTI (Environmental Sciences and Engineering unit) values were obtained by direct GC analysis.

<sup>2</sup> RTI values are shown to illustrate cylinder gas stability.

<sup>3</sup> % accuracy =  $\frac{\text{Lab average value} - \text{NBS value}}{\text{NBS value}} \times 100$

<sup>4</sup> Analysis by GC and not GC/MS.

<sup>5</sup> Each concentration for RTI and all the laboratory values are an average of two or three analyses.

<sup>6</sup> Analyses were performed 2 to 3 weeks after sample collection.

laboratory prior to the actual audit. The audits were conducted by R.K.M. Jayanty and J.A. Sokash, RTI, at the four laboratory sites. The sampling and analysis procedures used by the respective laboratories are summarized in the report.

## Summary of Results

### *Volatile Organic Sampling Train (VOST) Measurements*

Audits were conducted at four laboratories to evaluate the combined sampling and analysis accuracy and precision of the VOST method. In addition, accuracy and precision of the analytical component were also evaluated. Each laboratory collected three pairs of samples using the entire VOST system and three pairs of samples on VOST organic sorption cartridges only from the VOST audit gas cylinder. All samples were collected from the audit cylinder at a flow rate of 0.5 L/min for 20 min for a total volume of approximately 10L. The collected samples were analyzed by three of the laboratories (Laboratories A, B, and D—see Table 1) using thermal desorption, purge and trap and gas chromatography/mass spectrometry (GC/MS). One laboratory (Laboratory C), however, analyzed the collected samples by gas chromatography using PID/Hall detectors. Laboratory C analyzed only three organics (carbon tetrachloride, chloroform and benzene). All four laboratories analyzed at least one pair of VOST (Tenax-Tenax/charcoal) cartridges as blanks. The analysis results reported by the laboratories for each individual measurement of the five compounds and their percent accuracy relative to the NBS-determined concentrations are tabulated and included in the project report. A summary of all VOST audit results including laboratory accuracy is shown in Table 1. From Table 1, the range of percent accuracy for the entire VOST, in general, is found to be within the  $\pm 50$  percent accuracy limits reported in the VOST protocol (Ref. 1). The only exceptions are the vinyl chloride and carbon tetrachloride results from Laboratory A and vinyl chloride results from Laboratory B, which were outside the expected accuracy limits described in the VOST protocol. Similar ranges of percent accuracy were found for directly-loaded VOST cartridge samples. Blank concentrations for the five compounds from the Tenax and Tenax/charcoal cartridges were found to be  $<1.0$  ppb under laboratory conditions.

### *Bag Measurements*

Audits were conducted at two laboratories (Laboratories A and B) to evaluate

the combined sampling and analysis accuracy and precision for bag measurements. In addition, the accuracy and precision of the analytical component only was evaluated. Each laboratory collected audit gas in a bag three times for analysis. Between each analysis the bag was evacuated, filled with dry nitrogen, and then analyzed to show the absence of POHC memory effects. Each bag sample was analyzed in triplicate by direct injection of the sample from the bag into the gas chromatograph. The audit gas was also analyzed three times directly from the audit cylinder. A summary of the percent accuracies for each compound for both bag measurement (sampling plus analysis) and direct analysis only are shown in Table 1. In general, the range of percent accuracy was within  $\pm 50$  percent.

## Conclusions and Recommendations

Cylinder gases containing low ppb audit mixture of five organic compounds (carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene) have been used successfully for performance audits on VOST and bag methods to assess the accuracy and precision for the combined sampling and analysis of POHC. Four laboratories may not be representative of the type of results that currently could be expected from all laboratories conducting POHC

measurements during trial burns. However, the audit results indicated that laboratories can analyze POHC with accuracy to within the limits of  $\pm 50$  percent that are stated in the VOST protocol. The audit results also indicate that the analysis phase and not sampling is the principal source of error for both the VOST and bag methods. Blank concentrations for VOST cartridges were found to be less than 1 ppb for each of the five organics under laboratory conditions. Over the 4-month study period, the low ppb audit mixture of five organic compounds was found to be stable. It is recommended that a performance audit using EPA audit cylinders be conducted during each hazardous waste trial burn test to assess and document the accuracy and precision of POHC measurements. In addition, an audit may be useful prior to the trial burn to evaluate the proficiency of the source test organization for POHC measurements.

## Reference

1. Protocol for the Collection and Analysis of Volatile POHC Using VOST, August 1983. Copy available from Technical Support Office, Industrial Environmental Research Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

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*The complete report, entitled "Performance Audit Results for POHC: VOST and Bag Measurement Methods," (Order No. PB 84-187 889; Cost: \$8.50, subject to change) will be available only from:*

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