



Project Summary

Validation of the Volatile Organic Sampling Train (VOST) Protocol

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The measurement of volatile organic emissions from a hazardous waste incinerator is one of the more difficult source testing problems. Specific compounds called principal organic hazardous constituents (POHCs) are to be identified and quantified at levels of 0.5 to 100 ppb in hot, wet incinerator exhaust gas, which may also contain high particulate and acid levels. The Volatile Organic Sampling Train (VOST) Protocol which describes the practices used by laboratories making these measurements allows for several alternative designs and operating procedures. Because its use is currently being recommended by regulatory agencies to measure emissions for compliance determinations, the VOST Protocol was subjected to a methods validation study.

The VOST Protocol validation program consists of two phases: a laboratory validation and a field test validation. The laboratory validation examined the results of sampling six different POHCs at two different concentration levels, two tube configuration designs, two moisture levels and other procedural variations. The field test validation determined the expected precision and recovery results when spiking the gas stream of a hazardous waste incinerator with five specific POHCs. Recommendations relative to method improvements, quality assurance measures and other aspects of VOST sampling and analysis are also discussed in the full report.

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 two separate volumes (see Project Report ordering information at back).

Introduction

The Code of Federal Regulations, Title 40, Part 264, requires that a destruction and removal efficiency (DRE) of 99.99 percent be achieved for each principal organic hazardous constituent (POHC) designated in the Trial Burn Permit (1). The calculation of DRE requires sampling and analysis to quantify POHCs in the waste feed material and stack gas effluent. The manual entitled *Sampling and Analysis Methods for Hazardous Waste Combustion* provides information of applicable methods for collection and analyses of POHCs in process streams of hazardous waste incinerator units (2). *Protocol for the Collection and Analysis of Volatile POHCs using VOST* (VOST Protocol) describes the Volatile Organic Sampling Train (VOST) used to measure POHCs in the stack gas effluent (3).

Specific POHCs may be identified and quantified at low levels of 0.5 to 100 ppb in hot, wet incinerator exhaust gas, which may also contain high particulate and acid levels. The VOST has already been used to collect a significant amount of background emission data, and it is currently being recommended by regulatory agencies as the means to measure emissions for compliance trial burns; therefore, the EPA considered the validation of the VOST Protocol to be an essential research objective. The validation program consisted of two phases: laboratory validation studies (Phase I) (4) and field test validation studies (Phase II) (5). This

summary describes the experimental program to evaluate as many of the acceptable practices as possible under controlled conditions in the laboratory and under typical incinerator conditions in the field. The results of the laboratory phase of the validation studies led to certain recommendations about specific VOST procedures to be followed during field testing. An estimate of recovery (accuracy) and precision for the VOST Protocol under field conditions is also described.

Laboratory Validation Studies

Experimental

Six volatile organic compounds were selected for use in the VOST laboratory evaluation. These compounds are listed in Table 1. This table shows the boiling points and incinerability of the compounds. Two of these compounds, carbon tetrachloride and chloroform, were included because of their expected frequent designation as POHCs in hazardous waste incinerator trial burns. Benzene was included because of the effect its historically high background levels has had on Tenax, which is the primary sample collection medium of the VOST. Tetrachloroethylene (TCE) was included in the study to test recovery of a compound with a boiling point (121°C) near the high range of the protocol (approximately 100°C). Trichlorofluoromethane (TCFM) was selected because its low boiling point (24°C) challenged the recovery of the VOST near its low range (approximately 30°C). The final compound studied in this investigation was vinyl chloride (VC). VC was included even though its boiling point (-12°C) made it unlikely to be quantitatively recovered by the VOST. Except for TCFM, mixtures of these compounds at the ppb level were readily available as Group 1 gases through the Quality Assurance Division's (QAD) gas cylinder audit development program (6). The QAD's Group 1 gases were developed and certified by the National Bureau of Standards (NBS) and, therefore, concentrations of these mixtures could be traced to NBS for use in determining VOST recovery. A separate cylinder containing only TCFM and nitrogen was obtained, and the concentration of this cylinder was similarly traced to an NBS standard.

Test gas atmospheres for the laboratory study were generated by mixing in a dilution system gases from a cylinder of the five Group 1 gases (approximately 75 ppb each component, balance nitrogen),

Table 1. List of Compounds Selected For VOST Protocol Validation

Compound	Boiling point, ^b °C	Incinerability ^a ranking		Comments
		ΔH^c	T 99.99/2 ^d	
Tetrachloroethylene	121	15	1	Group 1 compound; ^e potential recovery problem
Benzene	80	282	23	Group 1; historical Tenax blank problem
Carbon tetrachloride	77	4	2	Group 1; frequently selected as a POHC
Chloroform	61	10	44	Group 1; common lab solvent.
Trichlorofluoromethane	24	1	NL ^f	Lower limit of acceptable boiling point range
Vinyl chloride	-12	58	26	Group 1; potential break-through problem

^a A ranking of 1 is the most difficult to incinerate.

^b The general target range in the VOST Protocol is 30° to 100°C.

^c Based on the heat of combustion table for 283 RCRA Appendix VIII constituents.

^d Based on the temperature required to achieve 99.99 percent destruction at a residence time of two seconds (list of 55 compounds compiled by J. J. Cudahy of IT Enviroscience, September 1983).

^e Group number refers to the QAD gas cylinder audit program.

^f Not listed.

a cylinder of approximately 75 ppb TCFM in nitrogen, and a cylinder of ultra-pure zero air. Test gas concentrations of approximately 15 and 0.5 ppb were generated. These two concentrations were considered to be within the range of normal hazardous waste incinerator POHC concentrations.

A quad (four-train) VOST was used to collect four samples simultaneously from a manifold purged with test gas of known levels of target compounds. Figure 1 shows a schematic of the quad VOST equipment in the laboratory evaluation. Figure 2 shows a more detailed description of a single VOST. Two types of VOST sample collection tubes are specified in the VOST Protocol. Figure 3 shows the two tube configurations in detail. The term "ST" is used here to designate the suspended tube design. This tube is conventionally used in ambient air sampling with Tenax. The other design is designated "ND" for neck-down tube. The VOST Protocol defines the ND as the inside/inside tube configuration and the ST as the inside/outside tube configuration. Since one of the goals was to determine which design to use in field work, the quad VOST laboratory experiments were conducted with two ST-type trains and two ND-type trains.

The sorbent traps were all prepared from one lot of Tenax and one lot of

charcoal and were prepared and conditioned as specified in the protocol. Preparation and conditioning of the traps were performed in a separate area of the laboratory in which no solvents are handled or stored. The exact handling of each trap during preparation and conditioning activities was documented in a log book.

All sorbent traps were analyzed in the same manner and in accordance with the protocol. Each sampled trap was spiked with an internal standard, thermally desorbed with organic-free helium gas bubbled through organic-free water and collected on an analytical sorbent trap. After the sample desorption steps, the analytical sorbent trap was rapidly heated and the carrier gas flow was reversed so that the effluent flow from the analytical trap was directed into the GC/MS. The volatile organics were separated by temperature-programmed gas chromatography and detected by low-resolution mass spectrometry. The mass of volatile compounds was calculated by the internal standard technique.

Unless specifically designated otherwise, all samples were analyzed within one week of collection.

Results and Conclusions

In the course of performing the laboratory evaluation, several parameters were

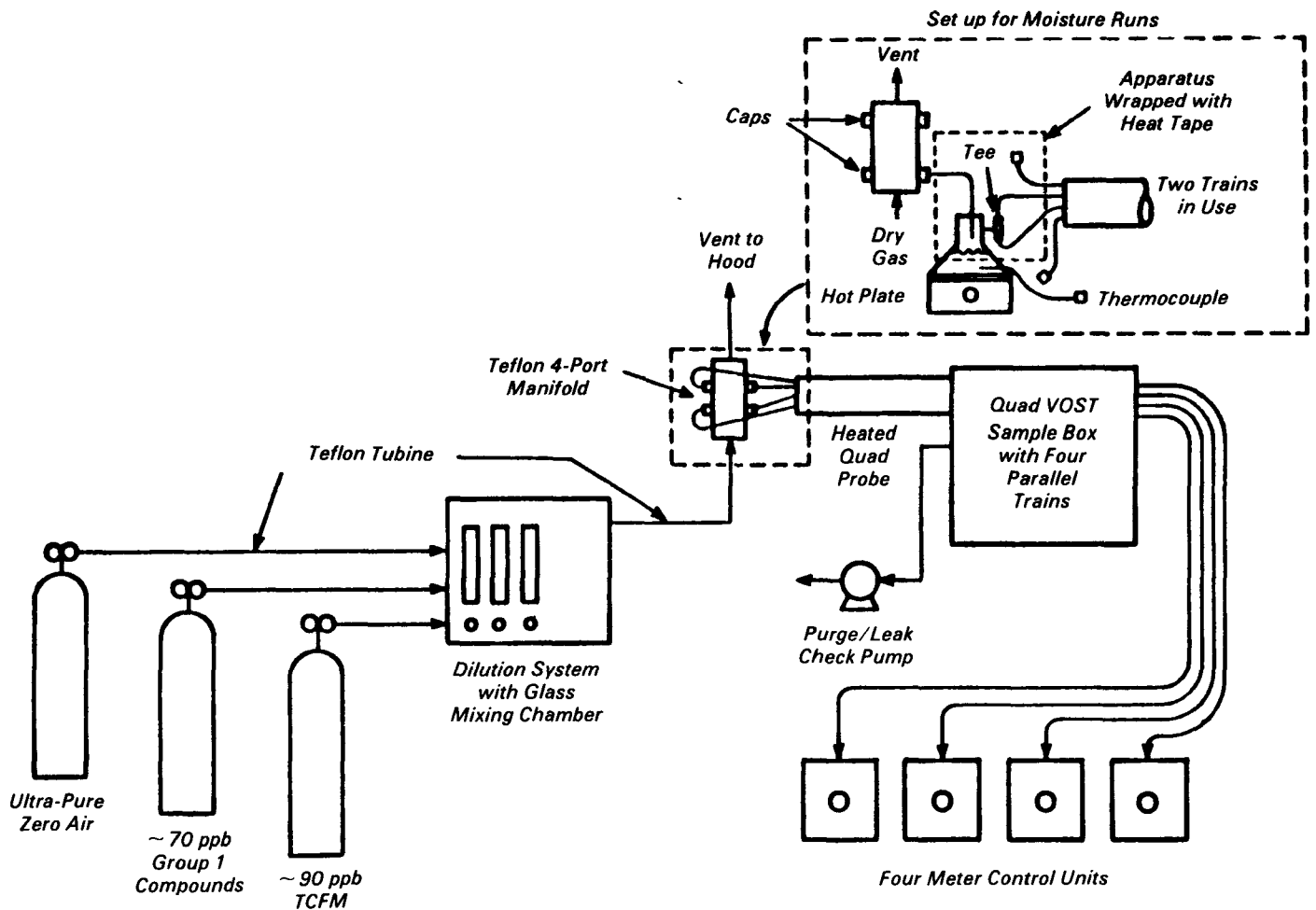


Figure 1. Schematic of sampling arrangement for laboratory evaluation.

studied (sorbent tube design, moisture level in the sample gas, and sample holding time).

The effects of tube design and moisture level varied from one component to the next. The following discussion describes the individual component data presented in Table 2, which was summarized from eight quad VOST runs.

The sample means are simple averages of paired run data; standard deviation are pooled from paired run data. The percent relative standard deviation (RSD) values represent the pooled standard deviations expressed as a percent of the means. The percent expected value (EV) is the mean measured concentration expressed as a percent of the concentration of the target compound in the sampled gas stream. The statistical significance of the difference between the paired measurement results for each type of sampling train

was determined by analysis of variance (ANOVA) for the dry test runs. The statistical significance of any difference between train types for the wet runs was determined by use of the t-test. Differences between train-types were determined to be statistically significant if the respective test indicated less than a 10 percent probability that the difference was due to chance.

All samples for quad Runs Q1, Q2, and Q3 were analyzed in the laboratory within less than one week after sample collection. Samples from quad Run Q4 were analyzed two weeks after sample collection and those from quad Run Q5 were analyzed five weeks after collection. These data are shown in Table 3. All of these samples were collected during the same week of sampling and at the same nominal test conditions, 15-ppb concentration of target compounds in a dry gas

stream. The measured concentration data were analyzed statistically by ANOVA to determine if the difference between results obtained at the three sample holding times was significant. For example, the average concentration of VC for Run Q5 (five-week holding time) was compared with the average concentration of VC for Run Q4 (two-week holding time) and for Runs Q1, Q2, and Q3 (holding time less than one week). If the difference between the concentration was significant, the effect of holding time was significant.

There was an overall tendency toward a decrease in the reported concentration of each target compound as the time between sample collection and sample analysis was increased. The value obtained for any compound on either type of trap, except VC on ST traps, was the highest when analysis was completed within one week. This apparent effect of

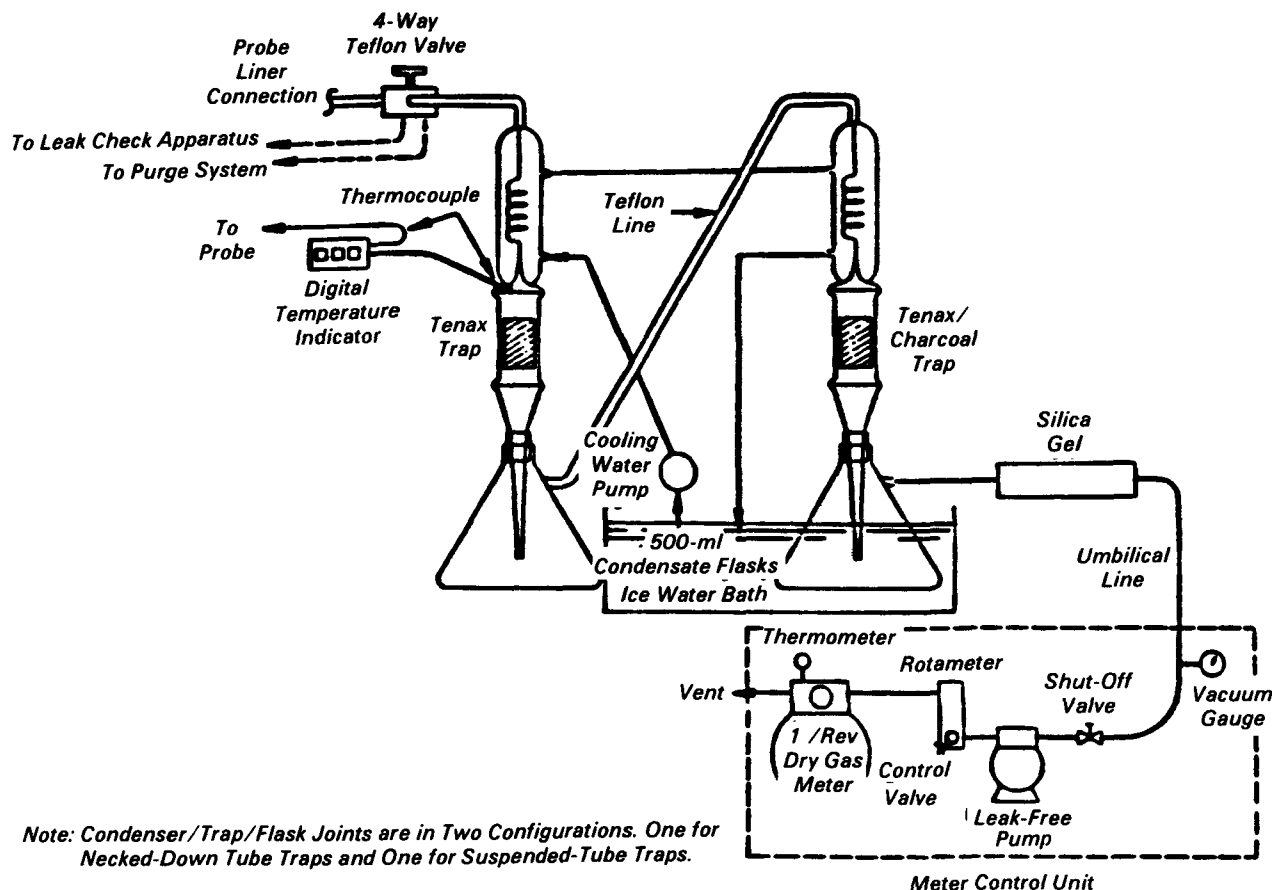


Figure 2. Schematic of single volatile organic sampling train (VOST).

sample holding time was statistically significant for VC and TCFM, the two lowest-boiling compounds studied.

The results of these laboratory tests indicate that the VOST precision and component percent recovery depend on the target compound and on the type of sorbent trap used. In some cases, the precision and percent recovery may also be affected by the moisture content of the sample stream and the amount of compound collected on the sorbent trap, which is a function of the concentration in the sample stream, the sampling rate, the sample volume, and the sampling time.

The precision and accuracy for the ND and the ST types of sorbent traps varied in certain cases. For example, of the three compounds, chloroform, carbon tetrachloride, and TCE, only carbon tetrachloride showed differences in precision and percent recovery between the ND and ST traps. For carbon tetrachloride, the ST traps also showed percent recovery differences between wet and dry condi-

tions. The precision reflected by the pooled relative standard deviation of the replicate pairs ranged between 10 and 20 percent and also indicated that the ND traps were superior to the ST traps.

Field Test Validation Studies Experimental

The Phase II field test validation of the VOST Protocol was conducted at the hazardous waste incinerator operated at EPA's Combustion Research Facility (CRF) near Jefferson, Arkansas. This incinerator is a pilot-scale rotary kiln type with an afterburner and caustic scrubber for emission control. Figure 4 shows a schematic of the sample point location in the process. The primary purpose of the validation was to determine the expected precision and accuracy of the protocol at a hazardous waste incinerator for certain specific POHCs. The POHCs for the field study were the same as in the laboratory study except vinyl chloride was not included because of its poor recovery. A

series of VOST runs was conducted on the incinerator stack gas with a four-train (quad) sampling assembly. Each quad run consisted of four samples collected simultaneously at the same point in the gas stream. A portion of the stack gas was spiked with the desired amount of target compounds by using mass flow controllers and a compressed gas cylinder containing approximately 150 ng/liter of each target compound. The amount of spike gas used for the tests represented less than 10 percent of the sample volume. All samples were collected at the nominal protocol conditions of 1 liter/minute for 20 minutes. Spike levels provided a minimum of 200 ng of each target compound (approximately 1-5 ppb concentration of target compound).

For recovery determinations, eight test runs were made with the quad sampling train. Figure 5 shows a schematic of the recovery test equipment in which two of the trains in each quad run were spiked individually. The other two trains in each quad run were not spiked so that back-

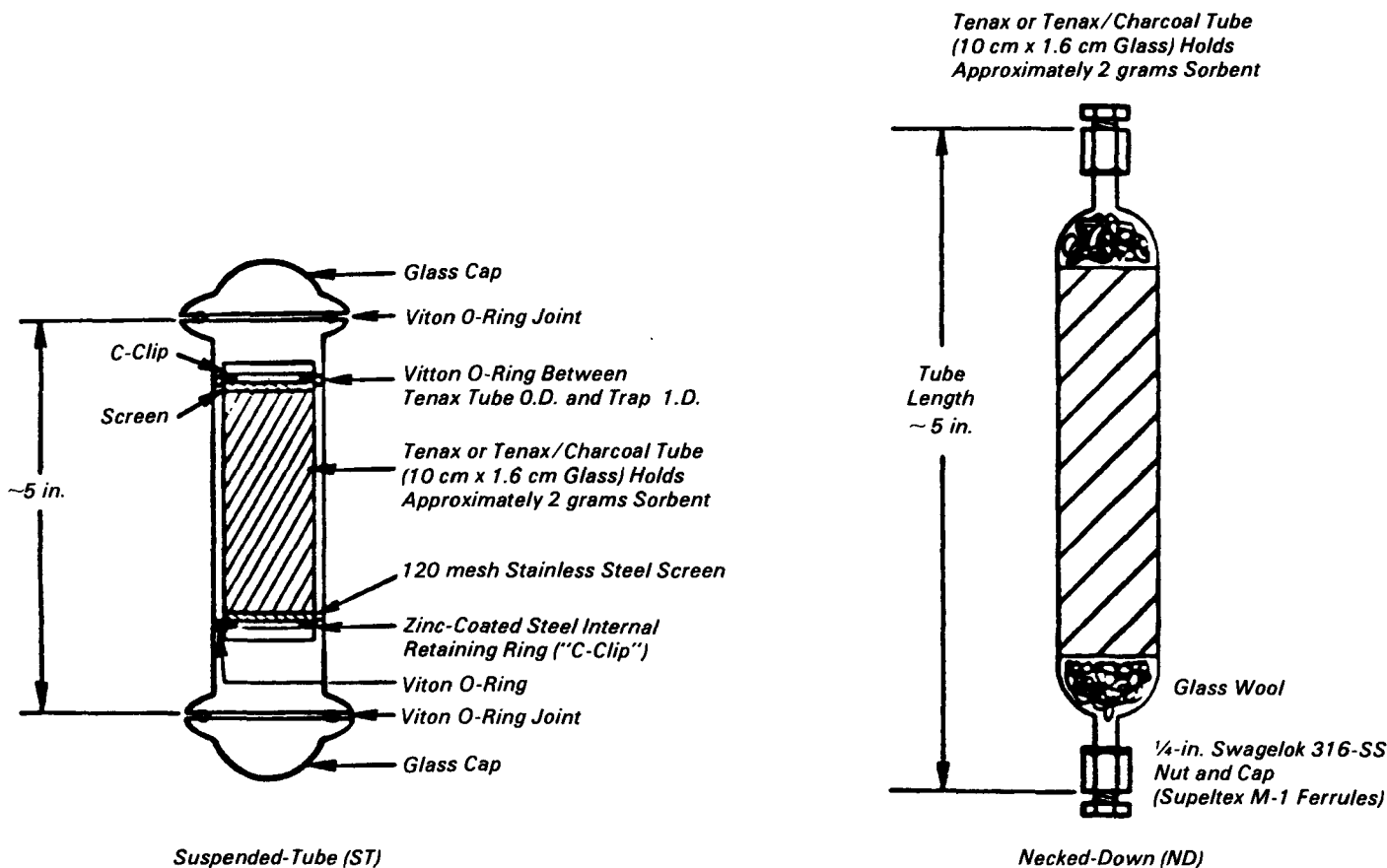


Figure 3. Sorbent trap configurations.

ground levels could be measured. The average background level of each target compound in the two unspiked samples was used to correct the amount of target compounds detected in each of the spiked samples. The amount of the target compounds recovered from the spiked trains (corrected for background) was used to estimate the level of recovery (accuracy) obtained by the VOST Protocol during the test series.

For precision estimates, seven test runs were made with the quad sampling train. Figure 6 shows a schematic of the precision test equipment in which a portion of the stack gas was extracted from the duct and spiked with the desired amount of target compounds so that all four trains in a given quad run were exposed to the same concentration of target compounds. Overall, VOST precision was estimated by pooling the relative standard deviations of the seven quad runs.

The 15 quad VOST runs were made during three separate field trips. During each trip, tests were conducted while the incinerator was burning commercially

available solvents (all of which were listed in the Resource Conservation and Recovery Act (RCRA) Appendix VIII) but none of the target compounds. This was done to minimize the background level of target compounds in the stack gas compared with the 200-ng spiking level. Two of the recovery runs (Runs 4 and 5) were made with 1,2-dichloroethane (DCE) as the feed solvent. All of the other tests were made with a feed solvent mixture referred to as Soup 2. This mixture consists of approximately 28 percent by weight each of nitrobenzene, 1,2,4-trichlorobenzene, and acetonitrile, and the balance made up by toluene. Propane was fired as an auxiliary fuel to maintain system temperatures.

All sampling times were 20 minutes, and between 20.1 and 21.3 liters were collected during each run (at metered conditions). These conditions corresponded to average sampling rates between 1.00 and 1.06 liters/min. The gas temperature at the exit of the primary condenser averaged between 5.6° and 19.7°C, and the maximum temperatures were all less than 20°C.

All sample preparation, conditioning, and analysis procedures were in accordance with the February 1984 VOST Protocol and the minor modifications as recommended by the results of Phase I. To eliminate any problems associated with sample holding time, all samples were analyzed within one week of the field collection.

As a part of the field study, laboratory blanks, trip blanks, and field blanks were taken. Laboratory and trip blanks remained sealed until analysis. Their analysis showed less than 2 ng of any POHC except for benzene which was more variable, ranging up to 14 ng. Benzene has been reported as an inherent contaminant in the Tenax sorbent. The recommended procedure for taking field blanks is to install them in the sampling train during the leak check procedure. This ensures that the blanks are exposed in the same manner as the actual sample tubes. Only chloroform was found in significant quantities in the field blanks during the first field test. During this test high levels of chloroform were

Table 2. Summary of Precision, Accuracy, and Train-Type Comparison Results^a

Nominal test conditions	Parameter	Vinyl chloride		Trichloro-fluoro-methane		Chloroform		Carbon tetra-chloride		Benzene		Tetrachloro-ethylene	
		ND	ST	ND	ST	ND	ST	ND	ST	ND	ST	ND	ST
0.5 ppb, dry ^b	\bar{X} , ng/liter	0.60	0.17	2.22	2.37	1.90	1.94	2.72	2.28	2.87	2.55	3.69	3.91
	σ^c , ng/liter	0.09	0.01	0.31	1.42	0.08	0.11	0.14	0.26	1.04	1.13	0.03	1.79
	% RSD ^c	15.0	5.9	13.9	55.9	4.2	5.7	5.1	11.4	36.2	44.3	0.8	45.8
	% EV ^d	42	12	78	83	83	85	85	71	175	155	103	109
	Significant difference? ^e		Yes		No		No		Yes		No		No
0.5 ppb, wet ^f	\bar{X} , ng/liter	0.53	0.36	2.74	2.53	2.02	1.84	2.71	1.63	2.48	2.01	3.79	3.55
	σ , ng/liter	0.13	0.09	0.17	1.0	0.12	0.27	0.39	0.32	0.75	0.03	0.15	0.40
	%RSD	24.5	25.0	6.2	39.5	5.9	14.7	14.4	19.6	30.2	1.5	4.0	11.3
	% EV	38	26	97	89	89	81	85	51	153	124	107	100
	Significant difference? ^g		Yes		No		No		Yes		No		No
15 ppb, dry ^h	\bar{X} , ng/liter	38.55	5.25	63.79	46.78	79.73	73.03	88.23	75.55	57.03	53.01	130.2	117.1
	σ , ng/liter	2.89	2.18	3.47	7.93	18.68	2.19	24.64	3.10	15.12	2.20	32.02	10.51
	%RSD	7.5	41.5	6.3	17.0	23.4	3.0	27.9	4.1	26.5	4.2	24.6	9.0
	% EV	91	12	76	56	116	106	92	78	116	107	121	108
	Significant difference? ^e		Yes		Yes		No		No		No		No
15 ppb, wet ⁱ	\bar{X} , ng/liter	20.32	19.98	64.47	33.84	73.34	65.31	86.46	53.43	52.64	49.35	143.4	139.2
	σ , ng/liter	2.10	3.71	4.55	6.62	2.89	6.12	1.13	2.39	1.10	3.45	4.16	3.51
	%RSD	10.3	18.6	7.1	19.6	3.9	9.4	1.3	4.5	2.1	7.0	2.9	2.5
	% EV	48	47	75	39	107	95	90	56	107	100	133	129
	Significant difference? ^g		No		Yes		Yes		Yes		No		No

^a Includes data from quad Runs Q1 through Q3 and Q6 through Q8 and paired Runs W1 through W8. No data are corrected for blank values.

^b Quad Runs Q6 through Q8, including all data except Run Q7, Sample No. L.5ST112 values for all compounds.

^c Standard and relative standard deviations are pooled from paired run data.

^d Percent of expected value = $(X/EV) \times 100$, where EV is the average for applicable runs taken from the summary table of dilution system data and expected concentrations in ng/liter.

^e Difference between train types based on analysis of variance at 10% probability level.

^f Paired Runs W5 through W8, including all data. The moisture content of the sampled gas stream was approximately 30%.

^g Based on t-test statistic at 10% probability level, difference between train types.

^h Quad Runs Q1 through Q3, including all data except Run Q2, Sample No. L15ND321 values for vinyl chloride and trichlorofluoromethane.

ⁱ Paired Runs W1 through W4, including all data. The moisture content of the sampled gas stream was approximately 30%.

present in the gas stream. During the following two tests no high POHC levels were found in the field blanks. The use of field blanks is recommended as a way of identifying possible contamination problems.

The sampling train also collects a condensate fraction. Since the POHCs chosen for this evaluation were not especially water soluble, several runs of condensate were grouped together for analysis. These composite samples showed less than one percent of the POHC compounds present in the condensate.

Results and Conclusions

Precision of the VOST method was determined by calculating the pooled rela-

tive standard deviation from the valid quad runs as described in the experimental section. As shown in Table 4, results for certain of the target compounds were invalidated in some of the samples. Precision run 6 (Run P6) was voided because the final dry gas meter readings were not recorded properly. Sample P1-4 (Precision Run 1, Train 4) was voided because the end of Tenax/charcoal trap was broken and it could not be connected to the analytical system in the normal manner. One of the sorbent traps in sample P3-4 and in sample P5-3 was broken in the field, and these samples were not analyzed. The internal standards were loaded improperly for sample P4-4. The GC/MS filament was flickering throughout the analysis of sample P8-3.

None of these results were used in the precision calculations.

The remaining data yielded sufficient information about precision of the VOST method. Table 4 also shows the calculated percent relative standard deviation (%RSD) for each of the test components. These %RSD data show that the VOST method is capable of attaining precision estimates of less than 5%. These are levels much better than expected and not attained in other tests where process and POHC component quality control may not be possible.

Accuracy of the VOST method was defined by the percent recovery of the spike compounds. The spiking procedure is described in the experimental section.

Table 3. Summary of Sample Holding Time Results^a

Sample holding time ^b (weeks)	Parameter	Vinyl Chloride		Trichloro-fluoromethane		Chloroform		Carbon tetrachloride		Benzene		Tetrachloro-ethylene	
		Trap type	ND	ST	ND	ST	ND	ST	ND	ST	ND	ST	ND
1 ^c	\bar{X} , ng/liter	38.55	5.25	63.79	46.78	79.73	73.03	88.23	75.55	57.03	53.01	130.2	117.1
	σ^d , ng/liter	2.89	2.18	3.47	7.93	18.68	2.19	24.64	3.10	15.12	2.20	32.02	10.5
	% RSD ^d	7.5	41.5	6.3	17.0	23.4	3.0	27.9	4.1	26.5	4.2	24.6	9.0
2 ^e	\bar{X} , ng/liter	15.21	7.28	47.56	31.16	70.72	67.74	75.55	72.33	53.04	47.80	116.0	99.5
	σ , ng/liter	2.11	0.23	4.29	2.25	0.76	9.19	0.38	6.29	3.31	2.65	2.19	7.50
	% RSD	13.9	3.2	9.0	7.2	1.1	13.6	0.5	8.7	6.2	5.5	1.9	7.5
	Difference, ^f %	-61	39	-25	-33	-11	-7	-14	-4	-7	-10	-11	-15
5 ^g	\bar{X} , ng/liter	10.70	6.42	50.02	29.60	70.42	68.26	81.86	70.29	50.68	50.20	126.1	112.2
	σ , ng/liter	5.64	5.03	0.40	4.64	6.02	5.20	3.09	3.27	1.28	1.02	20.93	12.30
	% RSD	52.7	78.3	0.8	15.7	8.5	7.6	3.8	4.7	2.5	2.0	16.6	11.0
	Difference, ^f %	-72	22	-22	-37	-12	-7	-7	-7	-11	-5	-3	-4
Significant difference ^h		Yes ⁱ		Yes		No		No		No		No	

^a All samples collected at a nominal target compound concentration of 15 ppb in a dry gas stream. All data were used from Runs Q1 through Q5 except for Run Q2. Sample No. L15ND121 values for vinyl chloride and trichlorofluoromethane. No data are corrected for blank values.

^b Time between sample collection and analysis. All samples were stored in refrigerated cans containing charcoal.

^c Runs Q1 through Q3.

^d Standard and relative standard deviations are pooled from paired run data.

^e Run Q4.

^f Difference between mean concentrations at indicated holding time and initial values.

$$\% = \frac{\bar{X}_i - \bar{X}_{initial}}{\bar{X}_{initial}} \times 100$$

^g Run Q5.

^h Difference between holding times based on analysis of variance at 10% probability.

ⁱ The effect of sample holding time is significant for the ND train but not for the ST train.

All recovery tests were made when the incinerator was burning the compound mix described as Soup 2 except for Runs 4 and 5 which were made during the incineration of DCE. The tests (DCE only) showed high amounts of chloroform (640 to 965 ng) and carbon tetrachloride (65 to 212 ng) in the unspiked samples relative to the amounts in the spiked samples (approximately 200 ng). Otherwise (using Soup 2), the amounts of target compounds detected in the unspiked background samples were less than approximately 50 ng. Values for TCFM were generally less than 12 ng. Values for TCE were generally less than 9 ng. Values for chloroform, carbon tetrachloride, and benzene generally ranged between 15 and 50 ng. The amounts detected in the spiked samples ranged between 180 and 354 ng.

As shown in Table 5, the results of certain target compounds were invalidated for some of the samples, and for specific target compounds in others. Sample R4-3-US (Run 4, Train 3, Unspiked) was voided because one of the

sorbent traps was broken after the internal standard was loaded. Results for sample R5-4-US were not used because the Tenax/charcoal trap was connected backward during analysis. The results for chloroform in sample R1-3-S and results for TCFM in sample R5-3-S were voided because the mass spectrometer detector was interrupted during integration of these peaks. Recovery values for R4 and R5 for chloroform and carbon tetrachloride were voided because of the high background level relative to the spike amounts as described earlier.

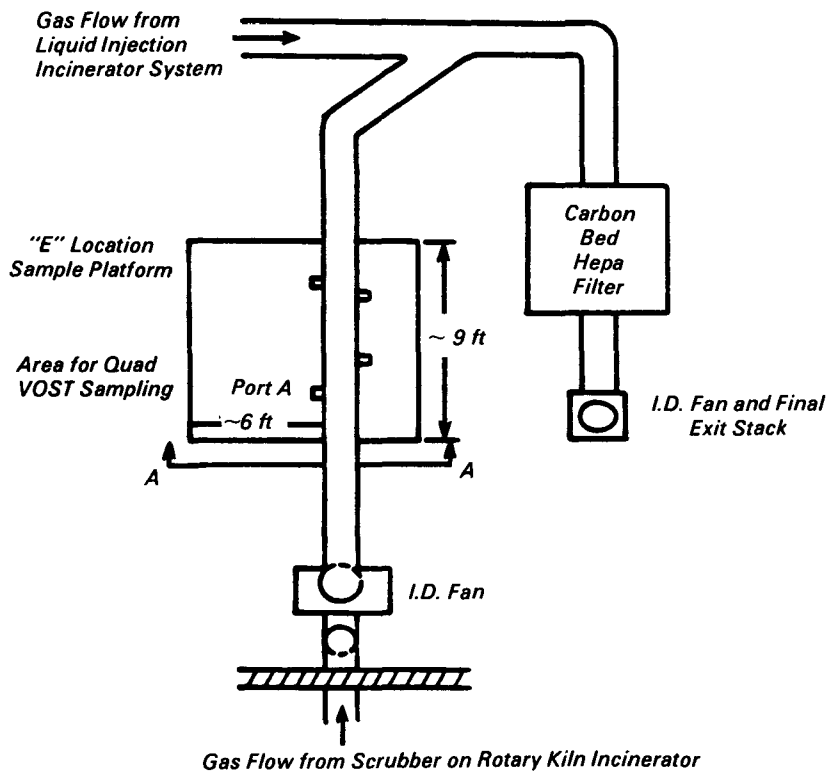
The average of the two unspiked samples was used as a measure of the component background levels. Because the other two sampling trains were spiked separately, they are treated as independent samples and provide two independent estimates of sample recovery. The mean recovery is reported as a percentage.

Table 5 also shows the calculated average recoveries (\bar{X}) and standard deviation (SD) of the POHCs. The average recovery was higher than 90%, with two

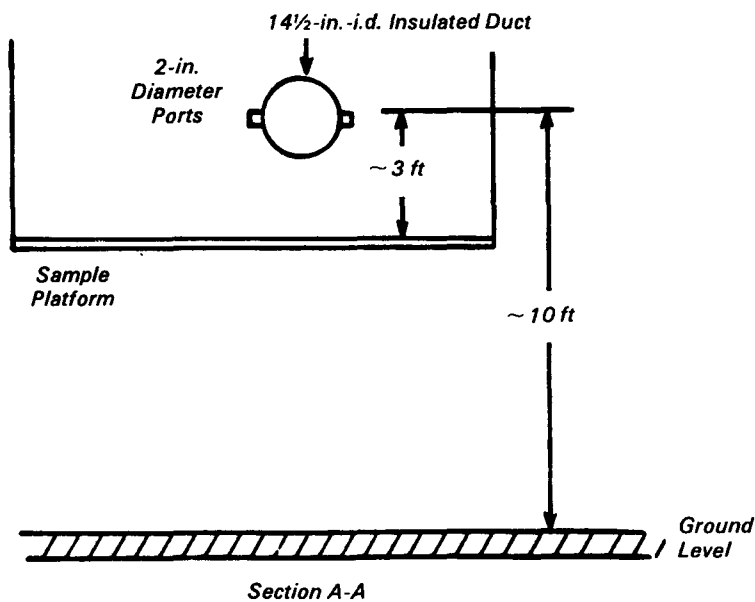
components (chloroform and TCE) showing a positive bias of approximately 20%. However, as a general conclusion both the laboratory and field data show that the VOST when carefully applied is capable of providing both precise and accurate measurements of volatile organics found at low parts-per-billion gas concentration.

References

- Code of Federal Regulations, Title 40, Part 264 (1980).
- Harris, J. C., et al., "Sampling and Analysis Methods for Hazardous Waste Combustion," EPA 600/8-84-002, February 1984.
- Hansen, E. M., "Protocol for the Collection and Analysis of Volatile POHCs Using VOST," EPA 600/8-84-007, March 1984.
- Validation of the Volatile Organic Sampling Train (VOST) Protocol - Laboratory Phase, EPA.
- Validation of the Volatile Organic Sampling Train (VOST) Protocol - Field Validation Phase, EPA.



Plan View



Section A-A

Figure 4. Schematic of "E" location sampling site at CRF.

6. Jayanty, R. K. M., et al., "Evaluation of Parts-per-billion Organic Cylinder Gases for Use as Audits During Hazardous Waste Trial Burn Tests," APCA 35 No. 11, November 1985.

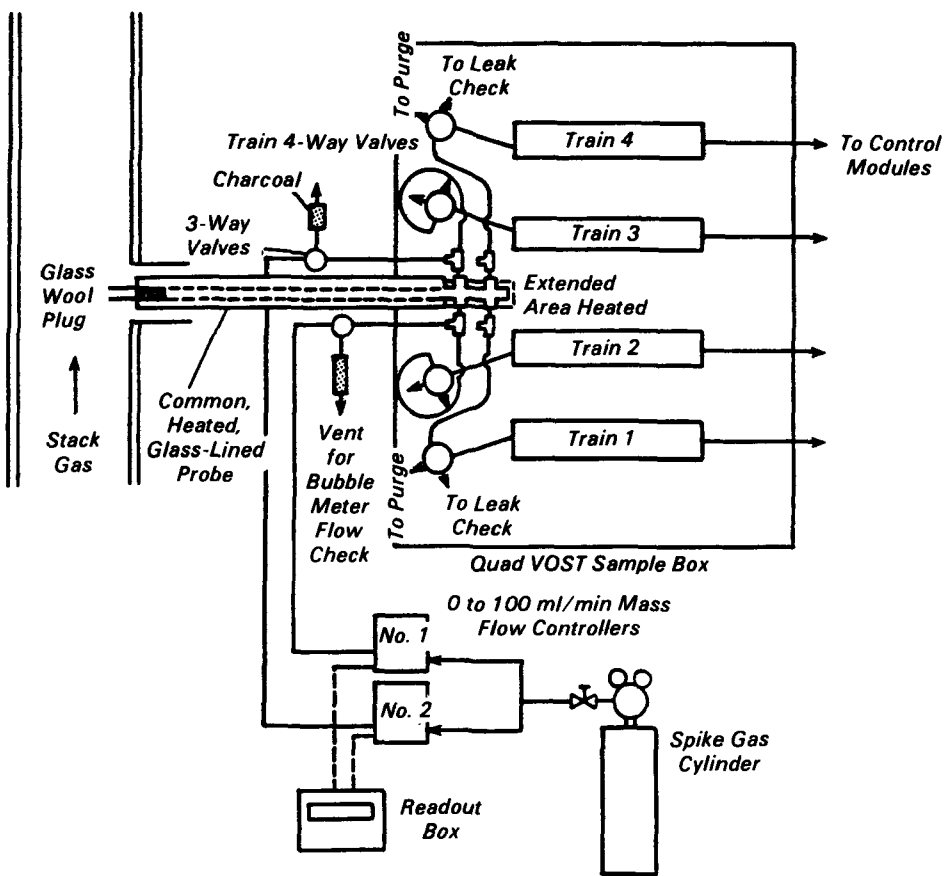


Figure 5. Schematic of sampling trains and spiking apparatus for recovery (accuracy) quad runs.

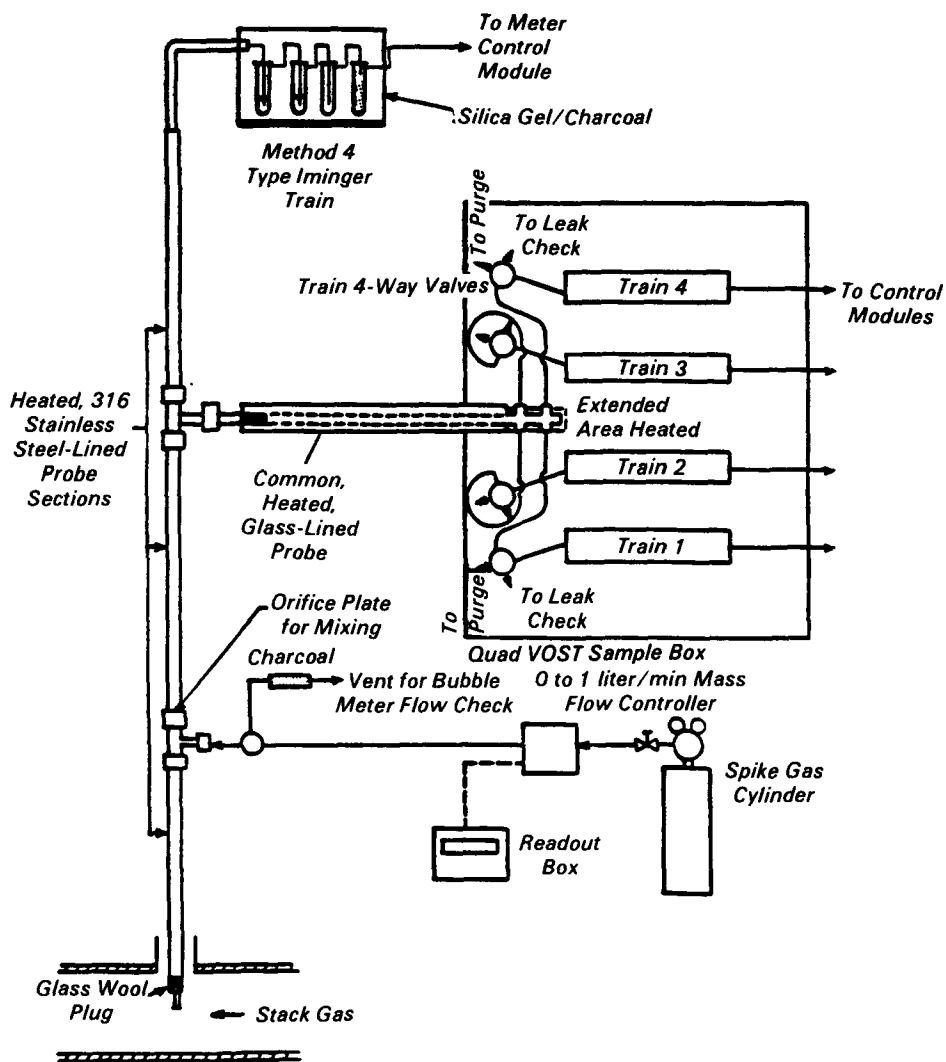


Figure 6. Schematic of sampling trains and spiking apparatus for precision quad runs.

Table 4. Summary Results of Each Precision Run

Value	Precision Test Number								Summary Results
	P-1	P-2	P-3	P-4	P-5	P-6	P-7	P-8	
POHC = Trichlorofluoromethane									
<i>n</i>	= 3	4	3	3	3	—	4	3	<i>n</i> = 23
\bar{X} (ng/1)	= 14.82	16.55	16.18	17.50	14.36	—	10.01	10.56	\bar{X} = 14.1 ng/1
SD (ng/1)	= 0.77	0.66	0.45	0.18	0.64	—	0.58	0.97	SD = 0.7 ng/1
RSD (percent)	= 5.19	4.02	2.76	1.01	4.48	—	5.78	9.18	RSD = 4.6 %
POHC = Chloroform									
<i>n</i>	= 3	4	3	3	3	—	4	3	<i>n</i> = 23
\bar{X} (ng/1)	= 17.68	18.68	18.06	20.04	15.42	—	12.35	13.56	\bar{X} = 16.4 ng/1
SD (ng/1)	= 0.32	0.44	0.62	0.54	0.39	—	0.47	0.60	SD = 0.4 ng/1
RSD (percent)	= 1.78	2.34	3.44	2.70	2.52	—	3.77	4.45	RSD = 3.0 %
POHC = Carbon tetrachloride									
<i>n</i>	= 3	4	3	3	3	—	4	3	<i>n</i> = 23
\bar{X} (ng/1)	= 20.16	20.74	21.46	22.36	17.62	—	11.07	11.61	\bar{X} = 17.7 ng/1
SD (ng/1)	= 0.78	0.57	0.58	0.85	0.71	—	0.25	0.51	SD = 0.6 ng/1
RSD (percent)	= 3.87	2.75	2.69	3.82	4.04	—	2.22	4.37	RSD = 3.5 %
POHC = Benzene									
<i>n</i>	= 3	4	3	3	3	—	4	3	<i>n</i> = 23
\bar{X} (ng/1)	= 19.99	20.03	19.56	21.65	15.10	—	13.56	14.30	\bar{X} = 17.7 ng/1
SD (ng/1)	= 0.18	0.34	0.78	0.69	0.17	—	0.57	0.24	SD = 0.5 ng/1
RSD (percent)	= 0.91	1.68	4.00	3.18	1.09	—	4.18	1.71	RSD = 2.7 %
POHC = Tetrachloroethylene									
<i>n</i>	= 3	4	3	3	3	—	4	3	<i>n</i> = 23
\bar{X} (ng/1)	= 19.99	21.51	19.89	22.60	17.20	—	13.68	0.8 ng/1	\bar{X} = 19.99 ng/1
RSD (percent)	= 3.82	4.86	3.32	5.29	3.61	—	2.61	2.97	RSD = 4.1 %

Table 5. Summary Results of Each Recovery Run

Value	Recovery Test Number								Summary Results
	R-1	R-2	R-3	R-4	R-5	R-6	R-7	R-8	
POHC = Trichlorofluoromethane									
	Percent Recovered								
1 =	89.45	98.66	91.83	100.62	90.75	85.41	79.26	86.33	<i>n</i> = 15
2 =	105.63	89.96	94.56	108.17	—	90.67	75.14	109.21	\bar{X} = 93%
\bar{X} =	97.54	94.31	93.20	104.39	90.75	88.04	77.20	97.77	SD = 10%
POHC = Chloroform									
1 =	—	127.81	110.98	—	—	131.94	116.08	121.36	<i>n</i> = 11
2 =	126.31	129.27	116.68	—	—	132.18	133.26	156.78	\bar{X} = 127%
\bar{X} =	126.31	125.54	113.83	—	—	132.06	124.67	139.07	SD = 12%
POHC = Carbon tetrachloride									
1 =	106.06	110.64	104.26	—	—	103.87	94.32	102.52	<i>n</i> = 12
2 =	108.79	120.59	105.86	—	—	99.82	119.24	115.14	\bar{X} = 108%
\bar{X} =	107.43	115.62	105.06	—	—	101.84	106.78	108.83	SD = 8%
POHC = Benzene									
1 =	100.07	114.73	99.56	103.90	103.84	112.75	98.20	109.15	<i>n</i> = 16
2 =	110.54	104.07	100.53	108.20	109.65	117.74	115.20	104.35	\bar{X} = 107%
\bar{X} =	105.30	109.40	100.04	106.05	106.74	115.25	106.70	106.75	SD = 6%
POHC = Tetrachloroethylene									
1 =	107.84	123.35	110.80	124.00	114.55	125.90	119.36	125.65	<i>n</i> = 16
2 =	114.82	115.25	127.28	131.12	125.84	134.16	132.45	122.20	\bar{X} = 122%
\bar{X} =	111.33	119.30	119.04	127.56	120.20	130.03	125.90	123.92	SD = 8%

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