
Fundamentals of Implementation of the Volatile Organic Sampling Train (VOST)

Sampling and Analytical Procedures

A. VOST Method Description and General Application Notes

The acronym VOST stands for Volatiles Organic Sampling Train. It operates as a non-isokinetic sampling train because volatile compounds have relatively low boiling points, and therefore are not expected to be condensed on particulate matter in stack gas effluents. Sampling representativeness is not dependent on sampling rate or velocity. A heated glass probe is inserted into the stack port, operated to sample stack gas for a minimum of two hours, and resin cartridge sets are changed out after the collection of 20 liters of stack gas has been collected on them. A single condensate sample is collected at the end of the run. Typically, four sets of resin cartridge samples are collected per run along with a single aqueous condensate sample. Three of the four sets are usually analyzed, while the 4th set serves as a backup set of samples providing a hedge against loss or breakage.

1. Method 0030 vs. Method 0031

Both of these methods are SW-846 VOST methods, but Method 0031 is the preferred approach for several reasons. Method 0031 is an updated and improved version of Method 0030. The newer method uses two Tenax[®] tubes instead of one. Method 0031 uses Anasorb 747[®] resin in the place of charcoal, and provides more complete information regarding the analysis of the aqueous condensate samples. The added Tenax[®] tube doubles the capacity of the resin bed for the capture of volatile analytes before breakthrough can occur. Since these two Tenax tubes are analyzed together, no additional analytical costs are realized. Criticism has been levied against this method because of the tenacious holding power of the Anasorb 747[®] resin toward the Method 8260 surrogate compounds. A remedy for this issue is recommended, and if followed, works well to reversibly yield up the surrogates during analysis. The resin tube construction is conducted such that 1/3 of the tube volume at the front end is filled with Tenax[®] followed by Anasorb 747[®]. The Tenax[®] captures and retains the higher molecular weight compounds like the surrogates while the highly volatile compounds with low molecular weights are captured on the Anasorb 747[®]. Note that this combination of resins is the same as the Method 0030 backup tube construction.

Figures 1 and 2 show the sampling train schematics for VOST Methods 0030 and 0031, respectively.

Applicable Methods*:

- Method 0030 - "Volatile Organic Sampling Train"
- Method 0031 - "Sampling Method for Volatile Organic Compounds (SMVOC)"
- Method 5041A - "Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST: Gas Chromatography/Mass Spectrometry Technique)"
- Method 8260B - "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)"

*Methods taken from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. SW-846 Method 0031, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

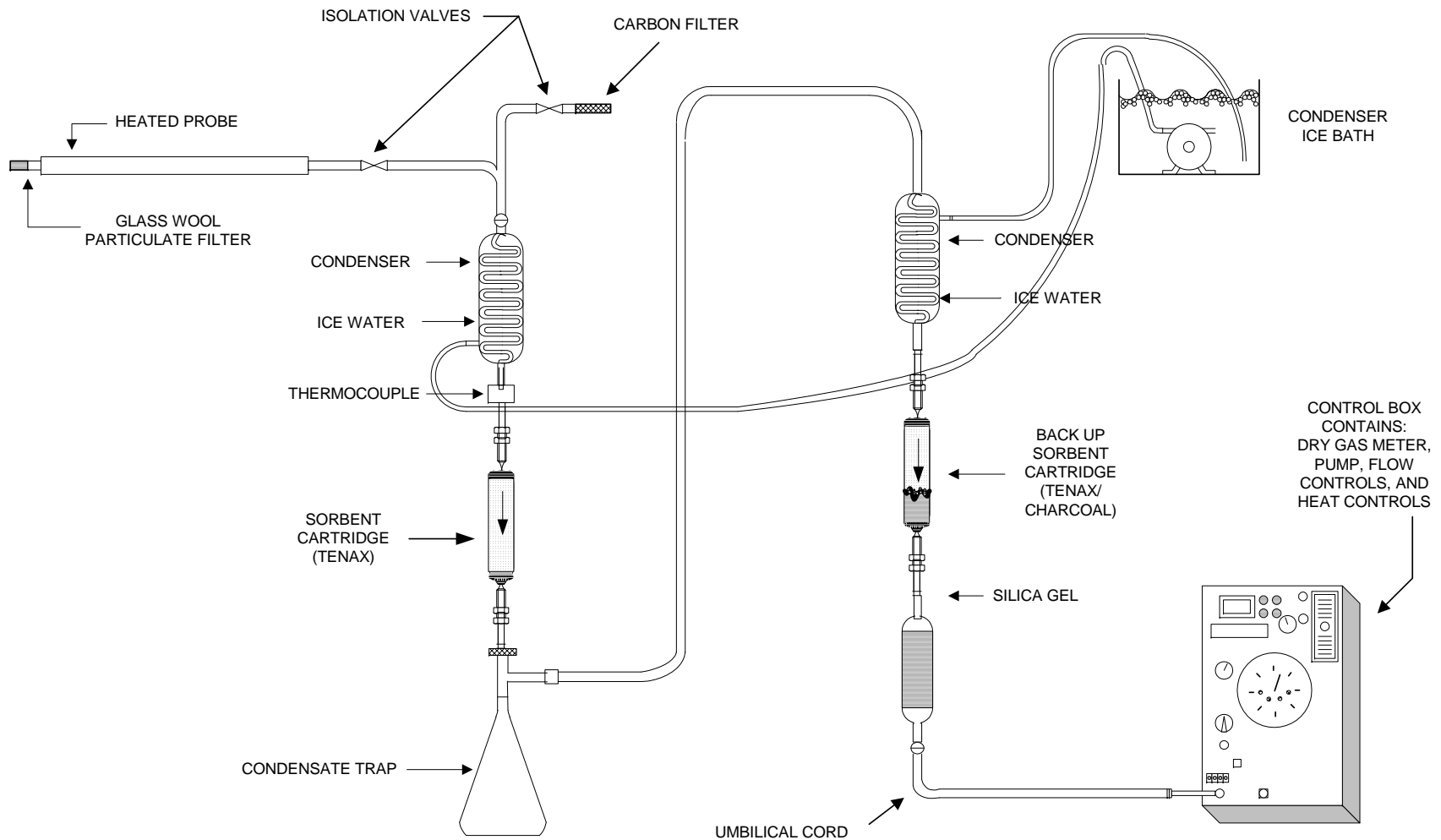


Figure 1. Schematic of Volatile Organic Sampling Train (VOST) Method 0030

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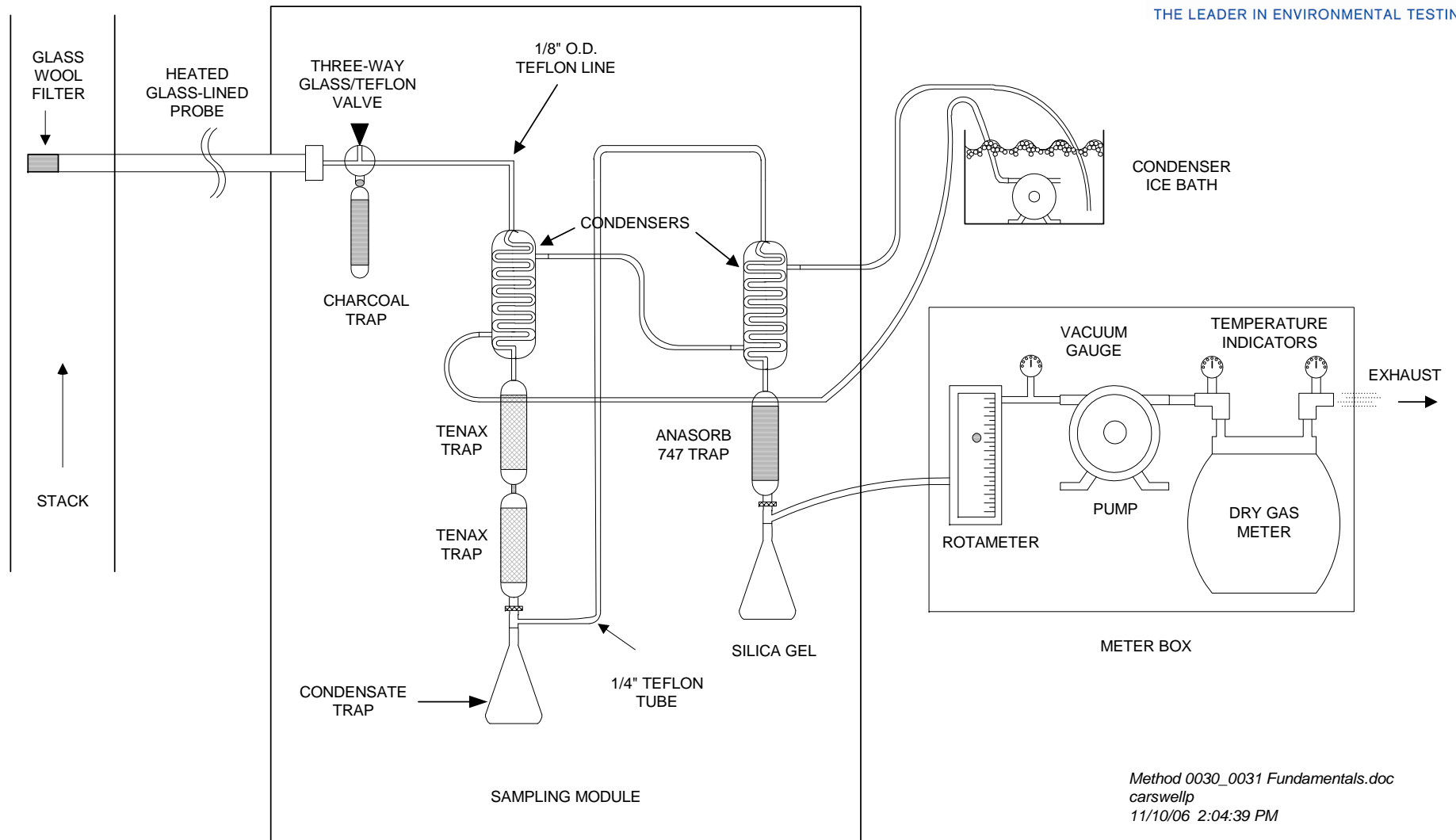


Figure 2. Schematic of Volatile Organic Sampling Train (VOST) Method 0031

2. DRE vs. Risk Evaluations

for CPT or trial burn testing, VOST data is typically used in destruction and removal efficiency (DRE) calculations, or calculations of stack emission rates used for risk assessment measurements. DRE measurements involve one or more compounds spiked into the incinerator and sampled in the stack gas to assess the proportion of compound destruction by the incinerator unit. Since these compounds are being used as performance evaluation compounds, care must be taken to demonstrate the 4-9's performance within the calibration range of the analytical method. The QC Handbook¹ requires that calculations be performed ahead of the test which derives the amount of POHC spike material required to be spiked into the incinerator so that POHC will be detected up in the calibration range of the analytical method an order of magnitude above the low calibration point. In this way, the achievement of 4-9's can be demonstrated by data that has been "hard quantitated" and not estimated. DRE calculations should never contain estimated values that are out of the calibration range, high or low, when proving compliance with performance standards. However, when DRE compliance has been easily displayed at the 4-9's level, additional performance demonstration can be acceptably displayed using calculations down to the RDL or MDL, if desired. Two orders of magnitude are often displayed over the required 4-9's required DRE when "clean" samples are evaluated. Six or 7-9's DRE have been estimated using data close to the MDL to calculate performance. While these DREs appear impressive in performance test reports, they have to be regarded as estimated performance measurements.

3. VOST vs. TO-14A Canister Analysis

On rare occasions, a test has to be run in which the compliance stack emissions level that has to be demonstrated is known to be above the high calibration point of the VOST analytical method. In this situation, dilutions of samples are required to be performed, or an alternate method of sampling has to be performed. Summa canisters can be used to take whole air samples of the stack gas for characterization purposes, provided regulatory approval has been obtained, and the limitations of the TO-14 approach have been evaluated. "In stack" detection limits are not as low using TO-14, and the integration of sampling over a 2 hour period requires equipping the canisters with calibrated flow controllers. Stacks that are saturated with moisture at elevated temperatures can cause moisture condensation in the Summa canisters on cooling, and cause possible low bias of polar organic compounds like alcohols and ketones. Table 1 compares the method detection limits (MDLs) and laboratory reporting limits (RLs) for VOST Methods 0030/0031 and Method TO-14A.

4. POHC Selections and Problem Analytes

Principal Organic Hazardous Constituents (POHCs) are compounds spiked into an incinerator for the purpose of evaluating DRE. To be universally characterized as a "good" selection as a POHC, a compound:

- √ must be ranked high on EPA's incineration list by incinerability ranking and in class
- √ must be able to be easily determined in low concentrations by available analytical methods,
- √ *and* must be a relatively inexpensive chemical to buy in bulk.

¹ "Handbook - Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration" (EPA-625/6-89-023).

Table 1
Comparison of VOST and TO-14A MDLs and RLs

Compound	(0031/5041A) MDL (µg)	TO14A/TO15 MDL (µg)	(0031/5041A) RL (ug)	TO14A/TO15 RL (µg)
Acetone	0.085	6.6	0.10	30
Benzene	0.0032	0.56	0.010	1.2
Bromobenzene	0.0036		0.010	
Bromochloromethane	0.012		0.025	
Bromoform	0.0066	0.60	0.025	1.2
2-Butanone (MEK)	0.035	1.9	0.10	3.0
n-Butylbenzene	0.0047		0.010	
sec-Butylbenzene	0.0036		0.010	
tert-Butylbenzene	0.0030		0.010	
Carbon disulfide	0.0011	0.47	0.010	1.2
Carbon tetrachloride	0.0011	0.66	0.010	1.2
Chlorobenzene	0.0013	0.52	0.010	1.2
Dibromochloromethane	0.010	0.60	0.025	1.2
Chloroethane	0.010	0.84	0.050	1.2
Chloroform	0.0019	0.49	0.010	1.2
2-Chlorotoluene	0.0023		0.010	
4-Chlorotoluene	0.0020		0.010	
1,2-Dibromo-3-chloropropane	0.020		0.050	
1,2-Dibromoethane	0.010		0.025	
1,2-Dichlorobenzene	0.0030	0.56	0.010	1.2
1,3-Dichlorobenzene	0.0031	0.59	0.010	1.2
1,4-Dichlorobenzene	0.0043	0.66	0.010	1.2
Dichlorodifluoromethane	0.0050	0.53	0.025	1.2
1,1-Dichloroethane	0.0019	0.37	0.010	1.2
1,2-Dichloroethane	0.0022	0.32	0.010	1.2
cis-1,2-Dichloroethene	0.0025	0.35	0.010	1.2
trans-1,2-Dichloroethene	0.0017	0.72	0.010	1.2
1,1-Dichloroethene	0.0023	0.60	0.010	1.2
1,2-Dichloropropane	0.0027	0.58	0.010	1.2
1,3-Dichloropropane	0.0018		0.010	
2,2-Dichloropropane	0.0015		0.010	
cis-1,3-Dichloropropene	0.0030	0.56	0.010	1.2
trans-1,3-Dichloropropene	0.0020	0.52	0.010	1.2
1,1-Dichloropropene	0.0010		0.010	

Compound	(0031/5041A) MDL (µg)	TO14A/TO15 MDL (µg)	(0031/5041A) RL (ug)	TO14A/TO15 RL (µg)
Ethylbenzene	0.0013	0.56	0.010	1.2
Hexachlorobutadiene	0.0048	0.58	0.025	1.2
2-Hexanone	0.0099	1.1	0.10	3.0
Methylene chloride	0.013	0.44	0.025	3.0
Naphthalene	0.010	0.78	0.025	1.2
n-Propylbenzene	0.0029	0.60	0.010	1.2
Styrene	0.0017	0.60	0.010	1.2
1,1,1,2-Tetrachloroethane	0.0010		0.010	
1,1,2,2-Tetrachloroethane	0.011	0.49	0.025	1.2
Tetrachloroethene	0.0021	0.66	0.010	1.2
Toluene	0.0022	0.78	0.010	1.2
1,2,3-Trichlorobenzene	0.014		0.025	
1,2,4-Trichlorobenzene	0.0030	0.60	0.025	1.2
1,1,1-Trichloroethane	0.0016	0.43	0.025	1.2
1,1,2-Trichloroethane	0.0050	0.78	0.025	1.2
Trichloroethene	0.0050	0.52	0.010	1.2
Trichlorofluoromethane	0.0049	0.54	0.050	1.2
1,2,3-Trichloropropane	0.0081	0.50	0.025	1.2
1,2,4-Trimethylbenzene	0.0048	0.58	0.010	1.2
1,3,5-Trimethylbenzene	0.0028	0.50	0.010	1.2
Vinyl chloride	0.0032	0.34	0.010	1.2
m-Xylene & p-Xylene	0.0034	0.51	0.020	1.2
o-Xylene	0.0017	0.56	0.010	1.2
1,1,2-Trichloro-1,2,2-trifluoroethane	0.0018	0.60	0.050	1.2
1,1,2-Trichlorotrifluoroethane		0.60		1.2
1,2-Dibromoethane (EDB)	0.010	0.47	0.025	1.2
1,2-Dichloro-1,1,2,2-tetrafluoroethane		0.59		1.2
1,2-Dichloroethene (total)	0.0018		0.020	
1,3-Butadiene	0.0016	0.96	0.010	1.2
1,4-Dichloro-2-butene (total)	0.050		0.10	
1-Butanol		2.2		3.0
2-Chloropropane	0.0020		0.010	
3-Chloropropene		0.84		1.2
4-Ethyltoluene		0.72		1.2
4-Methyl-2-pentanone (MIBK)	0.014	1.3	0.10	3.0
Acetonitrile	0.60	2.1	1.0	6.0
Acrolein		1.4		3.0

Compound	(0031/5041A) MDL (µg)	TO14A/TO15 MDL (µg)	(0031/5041A) RL (ug)	TO14A/TO15 RL (µg)
Acrylonitrile	0.076	1.1	0.50	3.0
alpha-Methylstyrene		0.60		1.2
Benzyl chloride		0.53		1.2
Bromodichloromethane	0.0016	0.49	0.010	1.2
Bromomethane	0.022	0.59	0.050	1.2
Chlorodifluoromethane		0.47		1.2
Chloromethane	0.0032	1.3	0.010	3.0
cis-1,4-Dichloro-2-butene	0.025		0.050	
Cumene		0.53		1.2
Cyclohexane		0.72		3.0
Dibromomethane	0.010	0.51	0.025	1.2
Ethyl ether		0.96		3.0
Hexane	0.0084		0.025	
Iodomethane	0.0016		0.050	
Isopropylbenzene (Cumene)	0.0023		0.010	
Methanol		5.5		60
Methyl tert-butyl ether	0.0040	1.3	0.025	3.0
n-Butane		0.78		1.2
n-Decane		0.58		1.2
n-Dodecane		0.72		1.2
n-Heptane		0.60		1.2
n-Hexane		0.40		1.2
n-Octane		0.60		1.2
Nonane		0.66		1.2
n-Undecane		0.66		1.2
Pentane		1.6		3.0
p-Isopropyltoluene (Cymene)	0.0038		0.010	
trans-1,4-Dichloro-2-butene	0.025		0.050	
Vinyl acetate		1.2		3.0
Vinyl bromide	0.0020		0.050	
Xylenes (total)	0.0048		0.030	

The most popular volatile POHC is chlorobenzene (CAS No. 108-90-7). Some criticisms have been levied against it because on its boiling point (131°C) is a borderline VOST application issue. VOST audits have routinely confirmed chlorobenzene's performance as a POHC using the standard train application. Other popular selections that have no analytical technical problems are toluene, tetrachloroethene and trichloroethane. These three are Class 2 compounds.

Benzene has been used under special conditions because of its excellent incineration ranking. However, the Tenax resin (polymer) can break down into benzene fragments under certain acidic conditions (Figure 3) including the presence of HCl or NO₂, both of which are prevalent in stack gas samples. Users of this POHC need to place excellent QC components into the analytical program to fully assess the background levels. Oftentimes the backgrounds can be tolerated without detrimental effects on the DRE demonstration. Acetonitrile is another compound that is often inquired about as a possible POHC selection. It is totally miscible with water and carries a high reporting limit. A potential user of acetonitrile should contact the lab before planning a trial burn demonstration around this chemical. Good planning and pre-testing can often circumvent the traditional POHC problems. A good technical approach can usually be prepared that allows an adequate demonstration to be performed, but the technical experts at the lab need to be involved in the planning process.

5. Target Concentration Levels for POHC Quantitations

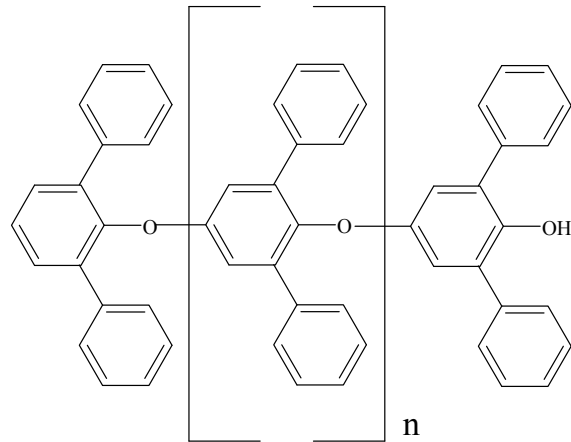
POHC spike levels into incinerator units should be set so that VOST samples analyzed by the lab target POHC concentrations that are approximately 10X the concentration of the low calibration standard when 4-9's are achieved. This approach insures that POHCs will be quantitated defensibly by having the required 4-9's DRE target performance level bracketed within the calibration levels of the method. Additionally, a 5- or 6-9's performance may be proven using this target level. Adequate lower range in the calibration curve is available to present and enhanced DRE performance by analyzing POHC levels that are less than the target performance level.

B. Defining and Implementing the Sampling Objectives

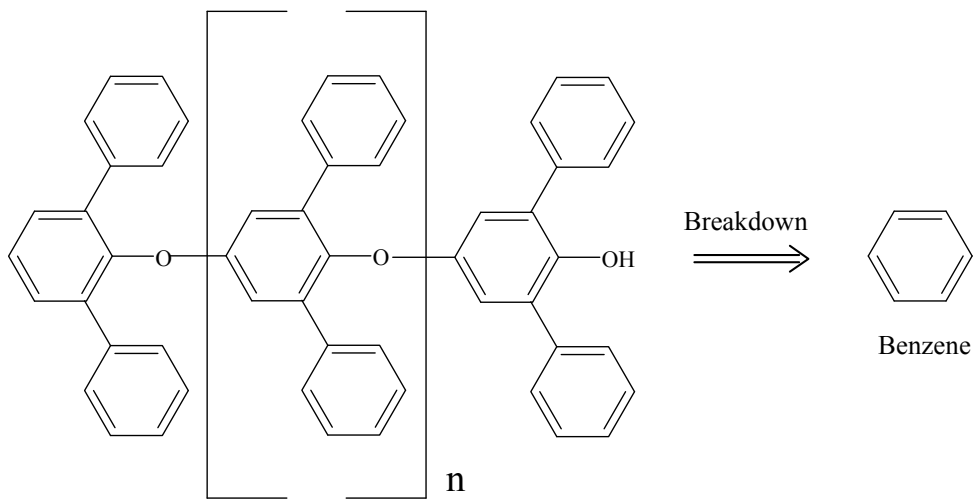
The sampling objectives are typically defined to acquire a representative set of stack gas samples by using VOST, and to handle them by procedures that will render valid analytical data. EPA has defined "representative" as being samples collected over a minimum of two (2) hours time of stack gas sampling. Sampling should coincide (operate simultaneously) with the other sampling trains being operated to characterize the stack gas for a given test condition.

The essential sampling procedure for VOST stack gas samples using Method 0031 is attached as Appendix A. This procedure is carried out by the incinerator stack sampling specialists.

Figure 3
2,6-Diphenylene Oxide Polymer



Tenax-GC



Tenax-GC

1. Sample Media Ordering, Shipping, Storage, and Preservation

The laboratory typically requests that a two-week lead time be given for the processing of VOST tube orders. Tubes are prepared before the test by purging them with nitrogen gas while they are heated in an oven to 200°C. Even though the tubes are new and have not been on some other sampling site, contamination or artifacts may accumulate on the resins during storage. All tubes should be “refreshed” by the laboratory before being used on a trial burn test. Trial burn tests should never be conducted with used VOST tubes. Tenax that has been exposed to acid gases is prone to decompose, even if it has been cleaned up or purged. Tenax[®] resin is composed of phenylene oxide that can break down to form benzene, quinones, and some other chemical artifacts. Trial burn tests are high profile projects that don’t need the risks of contamination from other sites coming to play in the data.

The lab should be instructed to ship VOST tubes to a location that is known to be reliable and clean. Shipments should not be sent blindly into a plant facility or location where the storage environment of the tubes has been exposed to volatile products. If you don’t know if the area is clean, don’t send the tubes. A person should be designated from either the sampling team or the test facility to receive the tubes. Subsequently, the VOST tubes should always be stored in an area and a container in which no contamination can get to them. Chilling of the tubes during shipment and before the test is optional, but some companies prefer refrigerated VOST tubes.

The SW-846 methods indicate that storage times should be limited to fourteen (14) days for the prepared VOST tubes. The length of time that tubes are stored should be a matter of judgment by the test supervisor. Highly competent testers that meticulously take care of details regarding media should be allowed to extend the storage life of VOST tubes where tests are delayed or extended. However, tubes which have apparently exceeded their storage shelf life, or have been exposed to contaminated environments should be returned to the laboratory for cleaning (refreshing).

Method 0031 requires that VOST tube samples and condensates be stored at <10°C during transport and storage. This specification is less stringent than the Method 0030 and the general volatiles storage and transport temperature that historically calls for $4 \pm 2^\circ\text{C}$. Note that the <10°C does not apply to volatile samples other than VOST.

The preservation of VOST condensate samples should include 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ since residual chlorine (Cl_2) is typically known to be present and a target analyte. Acidifying the condensates with HCl to $\text{pH} < 2$ is generally not required since these samples are not derived from surface or ground water, and therefore should not contain microbial action. The holding time for all of these VOST samples is 14 days from the time of sampling. Table 2 is a holding time and preservation summary for volatile organic compounds.

Table 2. Preservation and Holding Time Requirements for VOCs (Including Unspeciated Mass)

Measurement	Matrix	Preservation ^a	Holding Time ^b
Volatile Analytes on VOST	Tenax [®]	Chill on ice to about 10°C ±2°C	14 days to analysis
	Tenax [®] /Anasorb 747 [®]	Chill on ice to about 10°C ±2°C	14 days to analysis
	Tenax [®] /Charcoal	Chill on ice to about 10°C ±2°C	14 days to analysis
	Aqueous Condensate	Chill with ice 4°C ±2°C, 0.008% Na ₂ S ₂ O ₃	14 days to analysis

2. Recycled VOST Tubes

The use of recycled or used VOST tubes is not advised. As previously explained, Tenax[®] degrades over time, and the process of degradation appears to accelerate after resin exposure to the acid gas conditions of stack gas sampling. The risk of having major problems far outweighs the gain in costs of new tubes. See Figure 3 for the structure of Tenax resin and its major degradation product.

3. Tracking, Documentation and Communication Systems

The tracking system for trial burn samples commences in the field when the samples are collected. However, the function of a tracking and documentation system should be viewed as the “what makes the big ball bounce” since nothing related to the communications of the trial burn program facilitate the exchanges of data, sample information, and backup information more than this system. It is quite short-sited to conclude that the tracking and documentation system is required simply to provide labeling so that the report writer can identify which sample goes with which analysis. In essence, this would be claiming that the tracking and documentation system is simply needed so that the report writer can communicate with himself. Yet many of the designs of these systems of tracking and documentation make no regard for the other users of the system put into place. Some other applications of the tracking and documentation system should be:

- a. To provide a completeness check to see that all samples intended to be collected on site have been collected. This should be a real time expectation. A field crew leader should know the exact number of samples that are expected to be collected at a trial burn site. A typical trial burn may have as many as 225 samples and portions of samples to collect. Yet many planners never regard the total number of samples as a data quality objective that needs to be observed and monitored.
- b. To provide a system of organization to the laboratory so that sample entry into their coding system is organized and reviewable for expectations assigned in a QAPP. Most of the “systems” used have little or no regard for the process that takes place at the time samples are moved into the laboratory. In fact most engineers and stack samplers don’t think that it is important for the lab to be able to comprehend the objectives of the analytical process, but simply run the methods. Literally thousands of problems and errors would be prevented if

tracking and documentation systems were implemented after the planners comprehended the laboratory processes.

- c. To provide a system of organization by which data compilers, and data package compilers could check order, completeness, and reasonableness.
- d. To provide data validators the same facility as data compilers.

The tracking, documentation, and communication system is not a system that allows the planner to communicate with himself. It must take into regard the processes that contribute to the trial burn project at large. The laboratory is foremost on that list because all samples have to be processed for some objective at the laboratory. Nothing puts up bigger stumbling blocks than for the laboratory to process samples that are simply treated as commodities. Everyone benefits from a well designed tracking system. The system should have the following basic characteristics:

- Sequentially numbered so that independent verification can be made regarding completeness and order
- Pattern recognition is always a big contributor to spotting problems, or oversights
- A complete sample identity should always accompany a sample number (i.e. Test #1, Run #1 VOST Tenax[®] Tube Set #1)
- Sample numbers and identities allow automated setups of checklists, RFAs, COCs and laboratory data reporting

Figure 2 shows an example Master Sample List that is useful in the sample tracking process.

4. Cleaning VOST Equipment with Methanol

Probes, condensers, impingers, and connecting tubing can be cleaned with methanol before and after each project to prevent cross contamination between test sites. Methanol is a volatile solvent that has a molecular weight of 32 AMU. Therefore, its mass fragments are below the scan range of the typical VOST mass spectral measurement procedures. VOST components should be dried thoroughly, preferably in a drying oven prior to use to prevent masking of the Tenax capacity with methanol.

5. Quality Control Samples

- a. Field Quality Control Samples (Trip Blanks, Field Blanks, Temperature Blanks, and D.I. Water)

An ultra-clean source of water should be available on site during the trial burn testing. It can be obtained from the host laboratory “polished water” that is used as their source of method blanks. Therefore, routine background checks will be available for the analyte list being evaluated.

A field blank set of VOST samples should be collected during each run. A trip blank should accompany each shipment of samples to the laboratory, and should be made up of a pair of 40 ml VOA vials filled with the D.I. water. The purpose of the D.I. water trip blanks is to assess contamination that may have entered the samples in their storage, and shipping environments.

Trip blanks can be made up on site, or pre-prepared at the laboratory. They should be placed with the samples in storage at the test site. A second type of trip blank should be collected for the VOST resin tubes. A set of VOST tubes should be placed with the samples at storage that makes the trip to the laboratory without being opened in the field environment. A set of trip blanks should accompany each shipment of VOST tubes to the laboratory.

Table 3 summarizes the field QC sample requirements for a typical trial burn consisting of three (3) runs.

**Table 3. Summary of Field Quality Control Sample Requirements
 Trial Burn - Test Condition (3 Runs)**

Sample	QC Sample Type	Frequency	QC Sample Total
VOST Tube Sets	Field Blanks	One complete set per run	3 complete sets
	Trip Blanks	One set per trial burn test condition sample shipment	1 to 3 sets
VOST Condensate	Deionized Water Trip Blanks	One per trial burn sample shipment	1 to 3

Figure 2. Example Master Sample List

Field Sample No.	Client Sample ID	Test Condition No.	Run No.	RFA/ COC No.	Sampling Train or Process Source	Sample Description	Analytical Parameter	Laboratory Destination	QC Samples
A-3379	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #1 (Set #1)	Volatile Organic Compounds	Knoxville, TN	
A-3380	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #2 (Set #1)	Volatile Organic Compounds	Knoxville, TN	
A-3381	0031-02-1	2	1	001	VOST (Method 0031)	Anasorb 747 Tube (Set #1)	Volatile Organic Compounds	Knoxville, TN	
A-3382	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #1 (Set #2)	Volatile Organic Compounds	Knoxville, TN	
A-3383	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #2 (Set #2)	Volatile Organic Compounds	Knoxville, TN	
A-3384	0031-02-1	2	1	001	VOST (Method 0031)	Anasorb 747 Tube (Set #2)	Volatile Organic Compounds	Knoxville, TN	
A-3385	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #1 (Set #3)	Volatile Organic Compounds	Knoxville, TN	
A-3386	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #2 (Set #3)	Volatile Organic Compounds	Knoxville, TN	
A-3387	0031-02-1	2	1	001	VOST (Method 0031)	Anasorb 747 Tube (Set #3)	Volatile Organic Compounds	Knoxville, TN	
A-3388	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #1 (Set #4)	Volatile Organic Compounds	Knoxville, TN	
A-3389	0031-02-1	2	1	001	VOST (Method 0031)	Tenax Tube #2 (Set #4)	Volatile Organic Compounds	Knoxville, TN	
A-3390	0031-02-1	2	1	001	VOST (Method 0031)	Anasorb 747 Tube (Set #4)	Volatile Organic Compounds	Knoxville, TN	
A-3391	0031-02-1	2	1	001	VOST (Method 0031)	VOST Condensate	Volatile Organic Compounds	Knoxville, TN	
A-3392	0031-02-1	2	1	001	VOST (Method 0031)	VOST Tenax Tube Pair Field Blank	Volatile Organic Compounds	Knoxville, TN	FB
A-3393	0031-02-1	2	1	001	VOST (Method 0031)	VOST Anasorb 747 Tube Field Blank	Volatile Organic Compounds	Knoxville, TN	FB
A-3394	0031-02-1	2	1	001	VOST (Method 0031)	VOST Tenax Tube Pair Trip Blank	Volatile Organic Compounds	Knoxville, TN	TB
A-3395	0031-02-1	2	1	001	VOST (Method 0031)	VOST Anasorb 747 Tube Trip Blank	Volatile Organic Compounds	Knoxville, TN	TB
A-3396	0031-02-1	2	1	001	VOST (Method 0031)	VOST D.I. Water Trip Blank	Volatile Organic Compounds	Knoxville, TN	DI TB

Abbreviations:

FB = Field Blank
 TB = Trip Blank
 DI = Deionized Water

Two sets of spiked resin blanks are typically analyzed to display the resin cleanliness that is used for the test. TestAmerica Knoxville sets aside two (2) sets of tubes for each batch prepared. A separate batch is prepared for each project. This approach provides the background data on the resins at no additional costs to the customer. However, analysis of these resin background samples with delivery of the results before the project commences are considered billable pay items since they can't be batched with the sample.

b. Laboratory Quality Control Samples

Table 4 summarizes the laboratory QC sample requirements for volatile POHCs and PICs by Methods 5041A and 8260B.

C. Quality Issues During the Sampling Event

1. Use of Media, Pre-labeled, Sequenced and Documented

The Tenax and Anasorb 747 media should be ordered from the laboratory at least 2 weeks ahead of the first test day. VOST sets should be pre-labeled using a systematic designation that unambiguously names each tube. Figure 4 shows an example label. The list of information that should appear on the label is as follows:

- Test Site/Project Name
- Test Condition Number and Sample Number
- Tube Number (Tenax[®] Tube #1, Tenax[®] Tube #2, Anasorb 747[®] Tube)
- Date
- Time
- Sampled By

Table 4. Summary of Analytical Quality Control Checks, Frequencies, Target Acceptance Criteria, and Corrective Actions

Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
Volatile POHCs and PICs by GC/MS (Method 5041A, Method 8260B)	Mass scale calibration (tuning) using Bromofluorobenzene (BFB)	Daily or every 12-hour shift	Ion abundance within method specified ranges	Repeat tuning procedure
	Initial Calibration (ICAL) (minimum five (5) point calibration)	Prior to sample analysis	RRFs of CCC compounds: <ul style="list-style-type: none"> • %RSD \pm 30% Minimum response factor for SPCCs <ul style="list-style-type: none"> • Chloromethane: RRF \geq 0.100 • 1,1-Dichloroethane: RRF \geq 0.100 • Bromoform: RRF $>$ 0.100 • 1,1,2,2-Tetrachloroethane: RRF \geq 0.300 • Chlorobenzene: RRF \geq 0.300 	(1) Repeat ICAL (2) If still unacceptable make necessary adjustments (3) Repeat ICAL
	Continuing Calibration Verification	Daily (beginning of each 12-hour shift)	%Difference (%D) of RRFs from ICAL <ul style="list-style-type: none"> • %D \leq 20% for CCC compounds Minimum Response Factors <ul style="list-style-type: none"> • Meet criteria for ICAL 	(1) Repeat single-point check (2) If still unacceptable, perform multi-point calibration
	Laboratory Method Blanks	Once per sample batch (maximum 20 samples)	Target compound concentrations Concentration $<$ Reporting Limit (RL), 5X allowance for lab solvents	(1) Flag data associated with method blanks (2) Discuss in data package narrative
	Laboratory Control Sample/Laboratory Control Samples Duplicate (LCS/LCSD)	Once per sample batch (maximum 20 samples)	Accuracy, as %Recovery of spiked compounds 1. % Recovery within established control limits Precision, as RPD 2. RPD within established control limits	Discuss in data package narrative
	Internal Standards	All samples	Area counts relative to daily standard <ul style="list-style-type: none"> • 50 to 200% of standard area Retention times (RT) relative to daily standard <ul style="list-style-type: none"> • Within 30 seconds of standard RT 	Flag data
	Surrogate spike analysis	Every sample	Within established control limits	Flag data

Notes:

BFB	Bromofluorobenzene
CCC	Calibration check compound
ICAL	Initial calibration
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
PIC	Products of incomplete combustion
POHC	Principal organic hazardous constituents
RL	Reporting limit
RPD	Relative percent difference
RRF	Relative response factor
RSD	Relative standard deviation
RT	Retention time
SPCC	System performance check compounds

Figure 4. Example Sample Label

ABC Chemical Company CPT Run #1	
TestAmerica Knoxville Project No.: 142500	
Sample Type:	VOST Tenax Tube #2 (Pair #3)
Analysis Required:	Volatile Organic Compounds Sample No: N-1182
Destination:	TestAmerica Laboratories, Inc., Knoxville, Tennessee
Date: _____	Sampled By (Initials): _____
Time: ____:____ pm / am	Preservative: Cool, 10°C ± 2°C

Note that the information regarding Tenax[®] Tube #1 and Tenax[®] Tube #2 is very important to the laboratory for proper analytical sequencing. The two (2) Tenax[®] tubes are typically analyzed together, but the front tube during the sampling needs to be the front tube during analysis. Most of the volatile analyte loading occurs on the front tube, therefore thermal desorption should be aligned to reverse that process. Informing the laboratory of the sequencing identities is crucial for analyte recovery to be efficient. Other documentation and labeling issues have been discussed in another section.

2. Sampling Rates, Sampling Temperature and Sample Volumes

Methods 0030 and 0031 do not require isokinetic sampling, but simply collect stack gas over a minimum sampling time of two (2) hours. Time integration over a two-hour period is required to collect representative sample from the stack gas, and to evaluate emission fluctuations. While a sampling rate of 1.0 liters per minute is allowed by the method, a rate of 0.5 liters is always preferred. If the typical sample volume of stack gas is collected (20 liters), the duration of sampling for each tube set is 40 minutes. Three (3) tube sets acquire the needed two (2) hours of sampling. Typically, a 4th set is collected to provide a backup/contingency set in case some tubes are broken, or sample is lost during an analysis run. If the 1.0 liters per minute sampling rate is chosen, six (6) sets of VOST samples need to be sampled and analyzed in order to get in the 2 hours of stack sampling.

3. Sample Recovery and Transport

VOST samples are recovered on the stack during set change outs. Approximately every hour, a set has to be changed out (40 minutes for sampling per set plus leak checks). Tube sets should be capped, placed back in culture tubes, brought off the stack area, and stored on ice until shipping. During sampling, the VOST tube end caps should be stored in the sealed culture tube, and the tube stored in a clean area. Personal handling should be carried while donning powder free latex gloves.

Samples should be shipped on ice, and plenty of it, such that the samples arrive at the laboratory CONVINCINGLY at the required <10°C. These are compliance samples, and formal temperature measurements will be conducted on them upon arrival at the laboratory. Samples should be thoroughly packed to prevent breakage, and packed with an abundance of ice. There is no excuse for samples arriving at the laboratory out of temperature specifications.

4. Breakthrough Evaluation

For method 0030, the Tenax[®] trap is analyzed separately from the Tenax[®]/Charcoal trap, and POHC breakthrough is determined by calculating the percent analyte on the back tube. That is, breakthrough is not indicated when the analysis of the back trap measures <30% of the compound that is measured on the front trap. This criterion does not apply when less than 75 nanograms are detected on the back trap.

For Method 0031, the same evaluation is carried out, but the two (2) Tenax[®] traps are analyzed together.

5. Condensate Sample Volume and Collection

The volume of condensate sample volume can vary widely, depending on stack gas moisture content and temperature. The condensate is collected for a typical test, over a 160 minute period (4 sets × 40 minutes per set). A huge degree of confusion often ensues when collection of this sample is conducted with no real understanding regarding the VOST data assembly into train total calculations.

The analytical data objective is to be able to calculate the total amount of the target compounds in the condensate sample. The total is acquired by calculation of the product of analyte concentration multiplied by the condensate volume. The equation is as follows:

$$\text{Concentration of analyte } (\mu\text{g} / \text{L}) \times \text{Volume (L)} = \mu\text{g}$$

Therefore, if the volume of the condensate is known, the laboratory reported concentration is multiplied by this number to get the total analyte in the condensate sample. The guidelines to follow when conducting VOST sample collections are as follows:

- Measure the VOST Condensate volumes, including rinses, in the field. The sample and its rinses can be accumulated in a 100 ml graduated cylinder, followed by a reading and recording of the sample volume.
- Place the sample in a 40 ml VOA vial. Most of the time, there will be insufficient sample volume to fill two (2) vials. For simplicity sake, simply fill one vial, and record the volume of the sample on the laboratory communications documents (RFA/COC).
- If less than 40 mls of VOST condensate is collected in the graduated cylinder, pour the sample in a 40 ml VOA vial and top off the vial with polished DI water. Cap the vial with no headspace. Record this collection detail for the laboratory. If D.I. water is added to the condensate, let the lab know.
- If more than 40 mls are collected in the graduated cylinder, the best approach is to pour up a single VOA vial of condensate sample, and discard the rest. No top off with DI water is necessary. Cap the vial with no headspace. Note that a partially filled second vial gives a second concentration that is different than the first. The second vial can be eliminated all together if the final volume is recorded and used as the sample volume calculations. The single vial approach is also much simpler to track.

6. Solvent Control as Fugitive Contamination

Solvents provide contamination sources, and bottles of some common solvents are brought on site to conduct recovery of samples from other trains. The following solvents should be isolated from any contact with VOST equipment, opened only when ventilation to the outside environment is assured, and never opened in the same room that VOST tubes reside in, or will reside in:

- Methylene Chloride (CAS No. 75-09-2)
- Acetone (CAS No. 67-64-1)
- Toluene (CAS No. 108-88-3)

VOST tubes should be stored in isolated storage before and after their use on stack sampling. They should always remain in their sealed glass culture tubes, the end caps tightly in place on the tubes, and the whole assembly sealed in freezer storage bags.

7. VOST Audits

When a VOST audit is required to be performed on a performance test, a minimum of three (3) sets of VOST tubes should be collected. The instructions typically prescribe that a 10-liter sample be collected on each set. However, loading on these samples have been enormous over the past several years. Loading has been well over the high calibration point into the area where quantitation of the concentrations were estimated values, at best. TestAmerica Knoxville recommends cutting the sample size from the EPA audits to five (5) liters to lower the analyte loading on the tubes. Even at five liters, some levels will be up in the estimated region (flagged with “E”) of the quantitation.

D. Defining and Implementing the Analytical Objectives

The lab processes and deliverables from the analytical laboratory provide a major portion of the documentation of the performance of the incinerator being tested. Decisions and supervision regarding these processes should be handled by someone familiar with the internal workings of the laboratory, and the objectives of trial burn testing. Trial burn projects are highly technical projects with a convergence of many testing objectives taking place at one time in the laboratory.

1. Analytical Method Selection

The methods that have relevance during VOST testing are SW-846 Methods 5041A, and 8260B. (See Section A.1.).

2. The Problem of VOST Dilutions, Samples in Tedlar® Bags, and Desorbing VOST Tubes to Canisters and Methanol Extractions

The dilution of VOST samples is a very difficult task to pull off. So difficult that no EPA approved method exists, and when dilution of VOST samples is required, each laboratory performs its own design of a procedure. The common approaches include extractions and dilutions using methanol, dumping the VOST sample to a Tedlar® bag, or dumping it to a summa canister. None of these methods are standard methods, and the Tedlar® bag use is not advised due to significant sample losses to the bag in a relatively short amount of time. The article cited below from Environmental Science and Technology describes the losses of volatiles in Tedlar® Bags.²

The use of methanol as a solvent extraction method is capable of meeting recovery standards for surrogates, but large dilution factors are typically built into the process, and most data is delivered as “non-detect” since most analytes are diluted out of quantitation. Desorption to canisters preserves the VOST sample as a “whole air” sample, and in this form dilutions can be performed if needed. The transfer to the canister can be tricky and QC recoveries are difficult to track.

² Yan Wang, Tyler S. Raihala, Alan P. Jackman, and Richard St. John, “Use of Tedlar Bags in VOC Testing and Storage: Evidence of Significant VOC Losses,” *Environmental Science & Technology*, 30, No. 10 (1996), 3115-3117.

3. Volatile Analyte List

The laboratory has a standard analyte list (see Table 5) that is essentially derived from the EPA risk assessment guidance document. Non-standard analytes can be added, but the analytical objectives allow some variances on strategy. The three (3) tiers of analysis for non-standard volatile analytes are:

- Run all specialty analytes as TICs against a NIST library search,
- Run a one (1) point calibration for the acquisition of actual analyte retention times and response factors,
- Run a formal MDL determination, a five (5) point calibration, and set up each analyte to be reported from the standard reference data.

The second option gives completely reliable data when the absence of a compound is being proven at a given level of compliance, or reliable estimates are acceptable.

4. Tentatively Identified Compounds (TICs)

The TICs are identified by matching unknown peaks against the National Institute of Standards and Technology library of spectral information. Peaks that are lower than 10% of the nearest internal standard are not reported, and identities require an 85% matching of the mass spectral information. When spectral matching is below 85%, the peak is regarded as an unknown compound. Reporting of TICs as “Unknown” when no match is completed is common.

5. Column Bleed and Artifacts

Often, TICs appear that have silane or siloxane groups in their names. These compounds are generally attributed to column bleed, since most GC column support materials are siloxyl coatings. The lab typically excludes these compounds from the TIC reports. Other compounds that often show up on VOST samples that are difficult to justify being derived from the stack gas are high levels of benzene, chloromethane and bromomethane. While these compounds may be components of the stack gas, the distribution of these compounds across the tube sets may indicate a different origin. The chloromethane and bromomethane have been observed exclusively on the back tubes with non-detects on the Tenax[®] portions. Even if breakthrough accounted for distribution, some amount should be deleted on the Tenax[®].

6. MDLs³, RDLs⁴, and RLs⁵

³ The laboratory Method Detection Limit (MDL) is derived according to requirements outlined in 40 CFR Part 136, Appendix B.

⁴ The RDL is the Reliable Detection Limit. The RDL is the detection level recommended by EPA's National Research Laboratory in Cincinnati, Ohio, Environmental Monitoring Systems Laboratory (EMSL) in Cincinnati, Ohio, American Chemical Society (ACS) Committee on Environmental Improvement and the Drinking Water Standards Division (DWSD). It is defined as 2.623 times the MDL (2.623 X MDL).

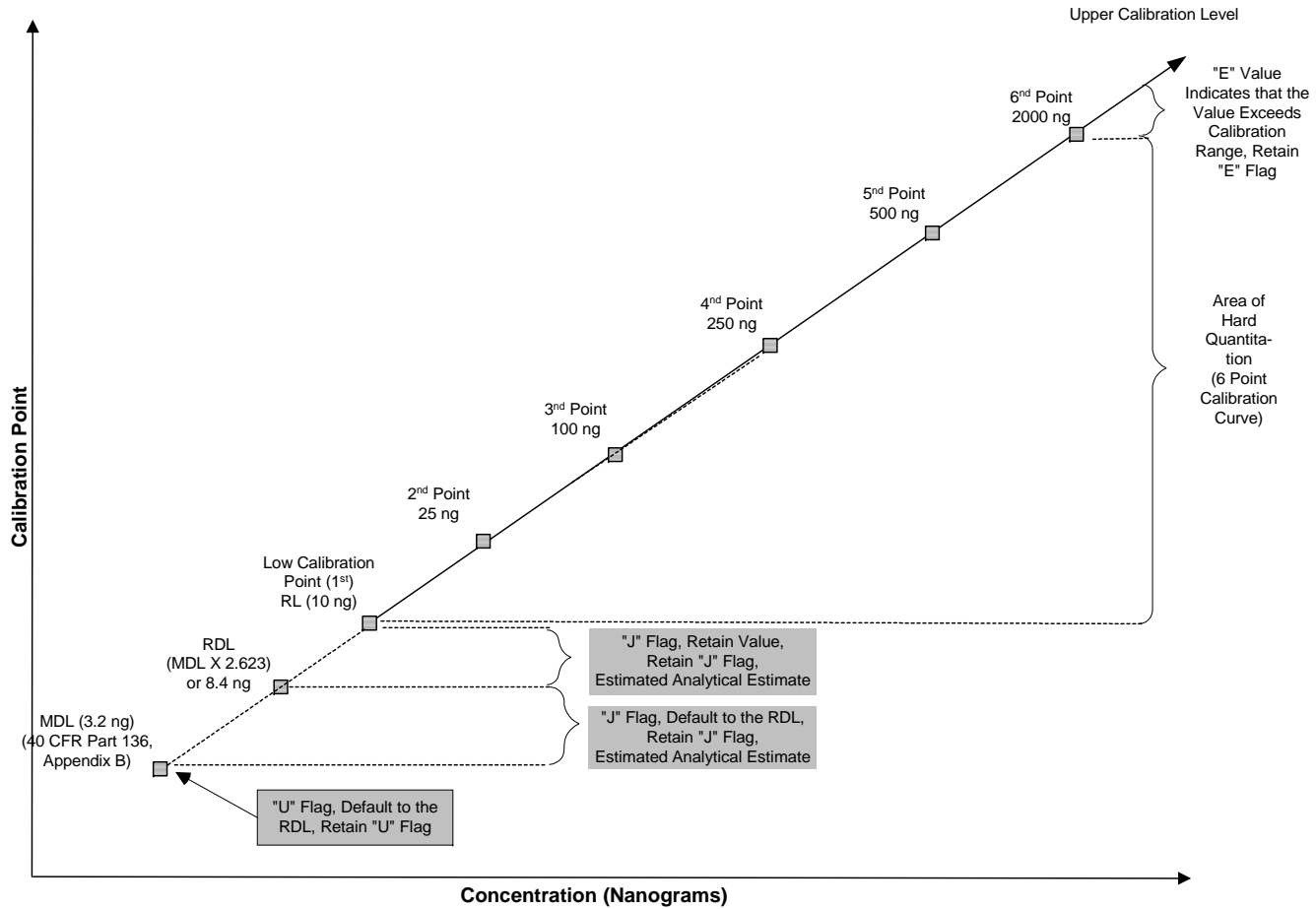
⁵ The RL is the laboratory Reporting Limit (RL).

The limits of detection and quantitation are essential analytical levels requiring basic understanding if data from the analytical laboratory is to be processed appropriately to meet DQOs, and the optimum level of performance demonstration. The method detection limit (MDL) and the reliable detection limit (RDL) are, as their names indicate, detection limits. The MDL is defined as the lowest level at which an analyte can be detected with 99% confidence that the analyte concentration is greater than zero. The reporting limit (RL) is a quantitation limit, or lower level of quantitation and is generally defined as the low calibration point of the method. Principal organic hazardous constituents (POHCs) should be targeted at an order of magnitude above the RL. Risk burn volatile analytes that are not being quantitated as POHCs should be evaluated all the way down to the MDLs. RDLs are used to remove statistical uncertainty from the MDL in calculating risks, and are calculated by multiplying the MDL by 2.623 ($MDL \times 2.623$). Figures 4 and 5 show actual calibration curves for chlorobenzene with quantitation areas for VOST tubes and condensates, respectively.

7. Data Quality Objectives (DQOs) for Volatile Surrogate Recoveries

Table 6 shows the SW-846 Method 8260 surrogate compounds along with their target recoveries.

Figure 4. Actual Calibration Curve for Showing Regions of Estimated Analytical Results and Hard Quantitation Areas of Chlorobenzene in VOST Tubes



Method 0030_0031 Fundamentals.doc
Figure 4_Implementation of the VOST Sampling & Analytical Procedures
Friday, November 10, 2006 3:25:19 PM

Figure 5. Actual Calibration Curve for Showing Regions of Estimated Analytical Results and Hard Quantitation Areas of Chlorobenzene in VOST Condensate Samples

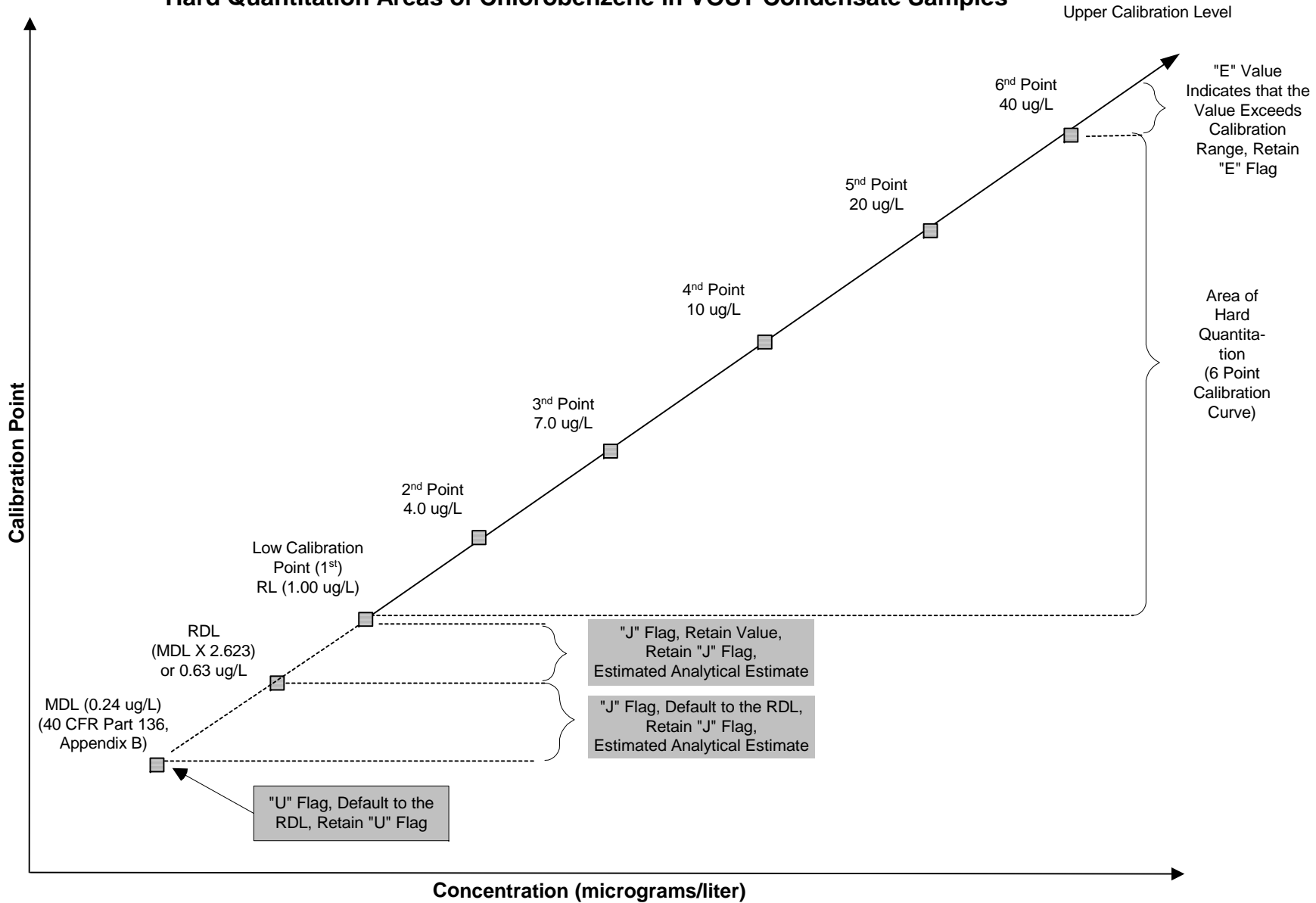
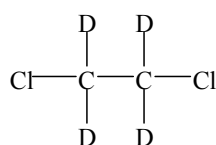


Table 6. Method 8260B Surrogates with Target Percent Recoveries

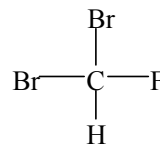
Volatile Surrogate Compound	CAS Number	Target Percent Recoveries for Aqueous Condensate Samples	Target Percent Recoveries for VOST Tube Samples
Toluene-d ₈	2037-26-5	80-120%	57 to 127%
4-Bromofluorobenzene	460-00-4	69-126%	50 to 125%
1,2-Dichloroethane-d ₄	107-06-2	71-127%	50 to 134%
Dibromofluoromethane	1868-53-7	79-120%	50 to 134%

Figure 6 shows the molecular structures of these surrogate compounds:

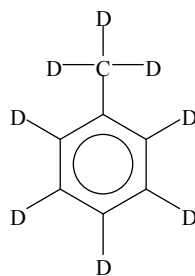
**Figure 6
Method 8260B Surrogate Compound Molecular Structures**



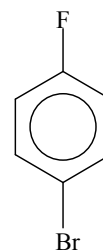
1,2-Dichloroethane-d₄



Dibromofluoromethane



Toluene-d₈



4-Bromofluorobenzene

8. Data Quality Objectives (DQOs) for Volatile Internal Standards

Percent recoveries are not typically calculated for VOC internal standards. The preferred quality assessment is to calculate the percent difference of peak areas and retention times. Table 7 gives the TestAmerica volatile internal standard compounds and their target percent differences.

Table 7
Method 8260B Internal Standard Compounds with Target Percent Differences

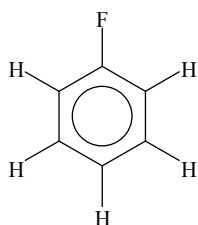
Volatile Internal Standard Compound	CAS Number	Area Count Target % Difference	Retention Time Target % Difference
Bromochloromethane	74-97-5	± 50% *	± 0.5 Minutes**
1,4-Difluorobenzene	540-36-3	± 50% *	± 0.5 Minutes**
Chlorobenzene-d ₅	3114-55-4	± 50% *	± 0.5 Minutes**

*Target percent difference from the corresponding Continuing Calibration Internal Standard Area.

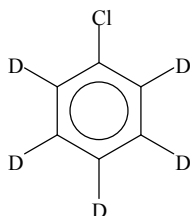
**Target percent difference from the corresponding Continuing Calibration Internal Standard Retention Time.

Figure 7 shows the molecular structures of these internal standard compounds.

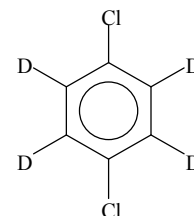
Figure 7
Method 8260B Internal Standard Compound Molecular Structures



Fluorobenzene



Chlorobenzene-d₅



1,4-Dichlorobenzene-d₄

9. Typical Laboratory Contamination

There are a few compounds that seem ubiquitous to environmental laboratory facilities because they are used as solvents in some parts of the lab areas. Methylene chloride, acetone, and toluene are common laboratory solvents. Low level hits on VOST samples are often due to the presence of these solvents in the lab areas. Benzene, chloromethane, and bromomethane are common artifacts that appear on VOST stack gas samples, but are not always derived from the emissions source. Distribution of the analytes within the VOST set can often help determine if “hits” are real.

E. Quality Issues at the Laboratory

Data acquisition processes at the analytical laboratory need to be clearly delineated. Some of them are options as preferences, and some recommended approaches are discussed below.

1. Collect four sets to analyze three sets (archiving a VOST set)

The on-site sample collections should provide the laboratory with four (4) sets of VOST tubes with the intention of analyzing three (3) of the sets. One (1) set should be used as a backup set to be analyzed in the case of tube breakage or data loss. The lab should be given the authority to make the decision when and if the backup tubes are to be analyzed.

2. Analyzing blanks as pairs (or sets)

Field and trip blanks can be analyzed with tubes in tandem, if conditions warrant. For Method 0030, the Tenax and Tenax/Charcoal tubes may be run together, and for the Method 0031 blanks, the two (2) Tenax tubes are run together. The laboratory does not have the capability of analyzing three or more tubes at one time.

3. Analytical measurements down to the MDL

The laboratory should always report data down to the MDL for risk assessment measurements. The MDLs should be formally derived according to 40 CFR Part 136, Appendix B, which defines the MDL as the minimum concentration of an analyte with 99% confidence that the value is greater than zero. For an MDL of 2 ppb, and at a 95% confidence level, the MDL would actually fall in the range of 1.28 to 4.4 ppb. This range represents statistically the same number. Therefore, it may be stated that at a concentration of 2 ppb, we are 99% confident that the analyte will be detected. Our ability to find 2 ppb with 95% confidence limits is represented by a range of results from 1.28 to 4.4 ppb which is statistically the same number.

Formal MDL data should be on file at the laboratory, and each fraction of the VOST should have a matrix specific data set to provide backup for the values to be reported.

4. Decisions using “E” values

An “E” value in the data set indicates that analyte(s) are present above the top calibration point of the method. This situation is not desirable because the data point flagged with an “E” is not bracketed by a calibration point on the upper end and provides a potential situation of low bias for the flagged values. For risk assessment data, the values are completely usable unless gross saturation is indicated in areas on the extended (extrapolated) calibration curve where definite low bias is occurring. VOST data for POHCs that are flagged with an “E”, are generally regarded as invalid, and should be avoided.

5. Data, Data Reduction, and Data Presentation

An example VOST data report is included as an attachment. VOST sets are combined to give total analyte in a given set, followed by the combination of sets to give the VOST run total. Note that the VOST run total includes contribution from the aqueous condensate sample. Significant figures for combining results follow the ASTM guidelines found in E29-93a(Reapproved 1999), "Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications", *American Society for Testing and Materials; Annual Book of ASTM Standards*; ASTM: Philadelphia, PA.

Example VOST Volatile Organic Compounds Analytical Results Summary VOST Totals

Field Sample Name: Volatile Organic Sampling Train (VOST) Totals
 Sample Description: Tenax[®] and Anasorb 747 Tubes (Set #1, Set #2, Set #3, and the VOST Condensate) for Volatile Organic Compounds Analysis

Analyte	CAS Registry Number	VOST Tube Set #1 ¹ (µg/Set) ⁴		VOST Tube Set #2 ¹ (µg/Set) ⁴		VOST Tube Set #3 ¹ (µg/Set) ⁴		VOST Condensate ² (µg) ⁴		VOST Run Total ³ (Total µg) ⁴	
Acrylonitrile	107-13-1	1.5	U	1.5	U	1.5	U	4.2	U	8.7	U
Benzene	71-43-2	0.074	U,J	0.077	U	0.073	U,J	0.21	U	0.43	U,J
Bromoform	75-25-2	3.8	E	4.1	E	3.9	E	0.21	U	12	E
Bromomethane	74-83-9	0.084	U,J	0.081	U,J	0.035	J	0.42	U	0.62	U,J
Carbon disulfide	75-15-0	0.075	U	0.043	U,J	0.075	U	0.21	U	0.4	U,J
Carbon tetrachloride	56-23-5	0.13	U	0.19	U,J	0.14	U	0.21	U	0.67	U,J
Chlorobenzene	108-90-7	0.063	U,J	0.06	U,J	0.059	U,J	0.21	U	0.39	U,J
Chloroethane	75-00-3	0.15	U	0.15	U	0.15	U	0.42	U	0.87	U
Chloroform	67-66-3	1.5	U	1.4	U	1.4	U,J	0.21	U	4.5	U,J
Chloromethane	74-87-3	0.25	U,B	0.28	U,B	0.16	U,J,B	0.42	U	1.1	U,J,B
Dichlorodifluoromethane	75-71-8	0.074	U,J	0.12	U	0.065	U,J	0.42	U	0.68	U,J
1,1-Dichloroethane	75-34-3	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
1,2-Dichloroethane	107-06-2	0.075	U	0.058	U,J	0.075	U	0.21	U	0.42	U,J
1,1-Dichloroethene	75-35-4	0.075	U	0.062	U,J	0.075	U	0.21	U	0.42	U,J
1,2-Dichloropropane	78-87-5	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
cis-1,3-Dichloropropene	10061-01-5	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
trans-1,3-Dichloropropene	10061-02-6	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
Ethylbenzene	100-41-4	0.055	U,J	0.075	U	0.075	U	0.21	U	0.42	U,J
Methylene chloride	75-09-2	0.6	U	14	E	0.35	U	0.21	U	15	E
Styrene	100-42-5	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U

Analyte	CAS Registry Number	VOST Tube Set #1 ¹ (µg/Set) ⁴		VOST Tube Set #2 ¹ (µg/Set) ⁴		VOST Tube Set #3 ¹ (µg/Set) ⁴		VOST Condensate ² (µg) ⁴		VOST Run Total ³ (Total µg) ⁴	
Tetrachloroethene	127-18-4	0.059	U,J	0.078	U	0.067	U,J	0.21	U	0.41	U,J
Toluene	108-88-3	0.55	U,J	0.53	J	0.42	J	0.21	U	1.7	U,J
1,1,1-Trichloroethane	71-55-6	0.075	U	0.063	U,J	0.075	U	0.21	U	0.42	U,J
1,1,2-Trichloroethane	79-00-5	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
Trichloroethene	79-01-6	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
Trichlorofluoromethane	75-69-4	0.064	U,J	0.055	J	0.11	U,J	0.42	U	0.65	U,J
Vinyl chloride	75-01-4	0.033	U,J	0.033	U,J	0.054	U,J	0.42	U	0.54	U,J
1,1,2,2-Tetrachloroethane	79-34-5	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
1,2,3-Trichloropropane	96-18-4	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
2-Butanone	78-93-3	0.2	U,J	0.25	U,J	0.25	U,J	0.84	U	1.5	U,J
Acetone	67-64-1	0.42	J	0.44	J	0.33	J	0.34	J	1.5	J
Bromodichloromethane	75-27-4	0.95	U	0.85	U,J	1	U,J	0.21	U	3	U,J
Chlorodibromomethane	124-48-1	1.8	U	1.7	U,J	2	U,J	0.21	U	5.7	U,J
cis-1,2-Dichloroethene	156-59-2	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
Dibromomethane	74-95-3	0.067	U,J	0.07	U,J	0.069	U,J	0.21	U	0.42	U,J
Iodomethane	74-88-4	0.15	U	0.12	U,J	0.15	U	0.42	U	0.84	U,J
trans-1,2-Dichloroethene	156-60-5	0.075	U	0.075	U	0.075	U	0.21	U	0.44	U
Xylenes (total)	1330-20-7	0.16	U,J	0.081	U,J	0.15	U,J	0.21	U	0.6	U,J

¹ The VOST Set (µg/Set) result consists of the sum of the two Tenax[®] Tube contents (analyzed separately), and the Anasorb 747[®] Tube contents.

The calculation is as follows:

$$\begin{aligned} &(\text{Total } \mu\text{g on the Tenax}^{\circledR} \text{ Resin Tube \#1 and the Tenax}^{\circledR} \text{ Resin Tube \#2}) + (\text{Total } \mu\text{g on the Anasorb 747}^{\circledR} \text{ Tube}) = \text{Total } \mu\text{g on the VOST Set.} \\ &\text{Therefore: } (\mu\text{g}) + (\mu\text{g}) + (\mu\text{g}) = \text{Total } \mu\text{g} \end{aligned}$$

- ² The VOST Condensate Total μg was obtained by multiplying the found concentration in the condensate sample by the VOST condensate volume.
- ³ The VOST Total μg consists of the sum of all sets of Tenax and Anasorb 747[®] Tube results that were analyzed from this run and the VOST condensate sample.
- ⁴ The data qualifiers (flags) for these samples are as follows:
- A "U" qualifier indicates this analyte was analyzed for, but not detected at or above the number indicated which is the reporting limit for that sample.
 - An "E" qualifier indicates the result exceeded the upper calibration range. The result is therefore an estimated value.
 - A "J" qualifier indicates this compound was detected but at a concentration below the quantitation limit. The result is therefore an estimated value.
 - A "B" qualifier indicates this compound was found in the associated method blank. Under these conditions this value is regarded as an estimated value.
 - A "Y" qualifier indicates this compound was an indistinguishable isomer in a tentatively identified compound (TIC).
 - An "N" qualifier indicates there is presumptive evidence that this compound is present.
 - A "D" qualifier indicates this result was obtained through dilution of the sample. This original analysis yielded a result that exceeded the calibration range.
 - An "M" qualifier indicates that its result was measured against the nearest internal standard and assumed a response factor of one (1).