State of Connecticut

Department of Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Volatile Organics by Method T0-17

Version 2.0

December 2006

Written by the Connecticut DEP QA/QC Workgroup

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1.0	First version for publication	2/3/2006
2.0	Edited version based on comments, Final Guidance	December 2006

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1.0 Overview of Method T0-17

1.1 Method T0-17 is a sorbent tube/thermal desorption/ gas chromatography/mass spectrometry procedure used to determine volatile organic compounds (VOC's) in air. This procedure requires an experienced gas chromatography/mass spectrometry (GC/MS) analyst familiar with air analysis using sorbent tubes and the QA/QC requirements of the method. Since the original publication methods TO-1 and TO-2, the technology of the solid absorbents has dramatically improved. New absorbents are commercially available, either signally or in multisorbent packings. The Connecticut Reasonable Confidence Protocol for this method does not allow any detector except a mass spectrometer. All method references are to the latest published version of the method found in the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, published by the US EPA.

2.0 Summary of Method

2.1 The monitoring procedure involves pulling a volume of air through a sorbent packing to collect VOCs followed by a thermal desorption-capillary GC/MS analytical procedure.

2.2 Conventional detectors are not allowed under this modification to achieve "Reasonable Confidence".

2.3 Key steps of this method are listed below.

2.3.1 Selection of a sorbent or sorbent mix tailored for a target compound list, data quality objectives and sampling environment.

2.3.2 Screening the sampling location for VOCs by taking single tube samples to allow estimates of the nature and amount of sample gases.

2.3.3 Initial sampling sequences with two tubes at nominally 1 and 4 liter total sample volumes (or appropriate proportional scaling of these volumes to fit the target list and monitoring objectives).

2.3.4 Analysis of the samples and comparison to performance criteria.

2.3.5 Acceptance or rejection of the data.

2.3.6 If rejection, then review of the experimental arrangement including repeat analysis or repeat analysis with backup tubes and/or other QC features.

[Note: EPA requires the use of distributed volume pairs (see Section14.4) for monitoring to insure high quality data. However, in situations where acceptable data have been routinely obtained through use of distributed volume pairs and the ambient air is considered well characterized, cost considerations may warrant single tube sampling. Any attendant risk to data quality objectives is the responsibility of the project's decision maker.]

2.4 Key steps in sample analysis are listed below.

2.4.1 Dry purge of the sorbent tube with dry, inert gas before analysis to remove water vapor and air. The sorbent tube can be held at temperatures above ambient for the dry purge.

2.4.2 Thermal desorption of the sorbent tube (primary desorption).

2.4.3 Analyte refocusing on a secondary trap.

2.4.4 Rapid desorption of the trap and injection/transfer of target analytes into the gas chromatograph (secondary desorption).

2.4.5 Separation of compounds by high resolution capillary gas chromatography (GC).

2.4.6 Measurement by mass spectrometry (MS).

2.5 The target compound list (TCL) is the same as listed in Compendium Method TO-15 (i.e., subsets of the 97 VOCs listed as hazardous pollutants in Title III of the Clean Air Act Amendments of 1990). Only a portion of these compounds has been monitored by the use of solid adsorbents. This method provides performance criteria to demonstrate acceptable performance of the method (or modifications of the method) for monitoring a given compound or set of compounds.

2.6 The reporting limit (RL) for a compound is dependent on the concentration of the lowest standard in the initial calibration, the sample volume, the sample introduction method, and any dilution of the sample. Lower reporting limits may be achieved using select ion monitoring, an ion trap mass spectrometer, or newer instrumentation

3.0 General Quality Control Requirements

Each laboratory is required to operate a formal quality assurance program and be proficient in the analysis performed. The minimum requirements include initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples (LCS) to assess precision and accuracy.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Table 1A. The Initial Demonstration of Proficiency must include the elements listed in Table 1.1. Records of this must be kept on file by the laboratory and available for inspection.

Table 1.1 IDOC Requirements

QC Element	Performance Criteria
BFB Tuning	Table 1C
Initial Calibration	Table 1A
Continuing Calibration	Table 1A
Method Blanks	Table 1A
Average Recovery	Table 1A
% Relative Standard Deviation	Table 1A
Surrogate Recovery	Table 1A
Internal Standards	Table 1A

Note: Because of the extensive analyte list and number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more analytes may not meet the performance standards for one or more QC elements. The laboratory should make every effort to find and correct the problem, and repeat the analysis. All non-conforming analytes along with the laboratory acceptance criteria should be noted in the Initial Demonstration of Proficiency data.

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits and surrogate recovery limits. These limits must meet or exceed the limits specified in Table 1A.

4.0 Interferences and Limitations

4.1 Interference from Sorbent Artifacts

4.1.1 Minimizing Artifact Interference.

4.1.1.1 Stringent tube conditioning and careful tube capping and storage procedures (see Sections 10.2.1 & 10.2.2 of Method T0-17)) are essential for minimizing artifacts. System and sorbent tube conditioning must be carried out using more stringent conditions of temperature, gas flow and time than those required for sample analysis.

4.1.1.2 A reasonable objective is to **reduce artifacts to 10% or less of individual analyte** masses retained during sampling. A summary of VOC levels present in a range of different atmospheric environments and the masses of individual components collected from 1, 2 or 10 L samples of air in each case is presented in Table 4 Method T0-17.

4.1.1.3 Given that most ambient air monitoring is carried out in areas of poor air quality, for example in urban, indoor and factory fenceline environments where VOC concentrations are typically above 1 ppb, Table 4 demonstrates that the mass of each analyte retained will, therefore, range from ~5 ng to ~10 µg in most monitoring situations. Even when monitoring 'ultraclean' environments, analyte masses retained will usually exceed 0.1 ng (3).

4.1.1.4 Typical artifact levels for 1/4 inch O.D. tubes of 3.5" length range from 0.01 ng and 0.1 ng for carbonaceous sorbents and Tenax® respectively. These levels compare well with the masses of analytes collected - even from sub-ppb atmospheric concentrations (see Table 4). Artifact levels are around 10 ng for Chromosorb® Century series and other porous polymer sorbents. However, these types of sorbents can still be used for air monitoring at low ppb levels if selective or mass spectrometer detectors are used or if the blank profile of the tube demonstrates that none of the sorbent artifacts interfere analytically with the compounds of interest.

4.1.1.5 Some varieties of charcoal contain metals which will catalyze the degradation of some organic analytes during thermal desorption at elevated temperatures thus producing artifacts and resulting in low analyte recoveries.

4.1.2 Artifacts from Long-term Storage of Blank Tubes.

4.1.2.1 Literature reports of the levels of artifacts on (a) Carbotrap/pack™ C, Carbotrap/pack™ B and Carbosieve™ SIII multi-bed tubes and (b) Tenax® GR tubes, by workers sealing the tubes using metal Swagelok®-type caps and PTFE ferrules with multi-tube, glass storage jars are

reported to be between 0.01 ng [after 1-2 months (4)] and 0.1 ng [after 6 months (5)] for (a) and (b) respectively.

4.1.2.2 Artifact levels reported for other porous polymers are higher - for example 5 ng for Chromosorb 106 after 1 week (5). More information is given in the Technical Assistance Document (TAD) referred to in Section 4.3.

4.1.3 Artifacts Generated During Sampling and Sample Storage.

4.1.3.1 Benzaldehyde, phenol and acetophenone artifacts are reported to be formed via oxidation of the polymer Tenax® when sampling high concentration (100-500 ppb) ozone atmospheres (6).

4.1.3.2 Tenax® should thus be used with an ozone scrubber when sampling low levels (<10 ppb) of these analytes in areas with appreciable ozone concentrations. Carbotrap™/pack type sorbents have not been reported to produce this level of artifact formation. Once retained on a sorbent tube, chemically stable VOCs, loaded in laboratory conditions, have been shown to give good recoveries, even under high ozone concentrations for storage of a year or more (7-9).

4.2 Minimizing Interference from Water

4.2.1 Selection of Hydrophobic Sorbents

4.2.1.1 There are three preferred approaches to reducing water interference during air monitoring using sorbent tubes. The first is to minimize water collection by selecting, where possible, a hydrophobic sorbent for the sample tube.

4.2.1.2 This is possible for compounds ranging in volatility from n-C5 (see SSVs listed in Appendix 1 of Method T0-17). Tenax®, Carbotrap™ or one of the other hydrophobic sorbents listed in Table 2 should be used.
[Note: It is essential to ensure that the temperature of the sorbent tube is the same and certainly not lower than ambient temperature at the start of sampling or moisture will be retained via condensation, however hydrophobic the sorbent.]

4.2.2 Sample Splitting

4.2.2.1 If the sample loading is high, it is usually possible to eliminate sufficient water to prevent analytical interference by using sample splitting (10).
4.2.2.2 Samples may be split either (1) between the focusing trap and the capillary column (single splitting) during trap (secondary) desorption or (2) between both the tube and the focusing trap during primary (tube) desorption and between the focusing trap and the column during secondary (trap) desorption (see Section 8.2.3 of Method T0-17) (double splitting). It may, in fact, be necessary to split the sample in some cases to prevent overloading the analytical column or detector.

4.2.3 Dry Purge

4.2.3.1 The third water management method is to “dry purge” either the sorbent tube itself or the focusing trap or both (11-13). Dry purging the sample tube or focusing trap simply involves passing a volume of pure, dry, inert gas through the tube from the sampling end, prior to analysis.

4.2.3.2 The tube can be heated while dry purging at slightly elevated temperatures (11). A trap packing combination and a near ambient trapping temperature must be chosen such that target analytes are quantitatively retained while water is purged to vent from either the tube or trap.

4.3 Atmospheric Pollutants not Suitable for Analysis by this Method

4.3.1 Inorganic gases not suitable for analysis by this method are oxides of carbon, nitrogen and sulfur, O3 and other permanent gases. Exceptions include CS and N O_2.

4.3.2 Other pollutants not suitable are particulate pollutants, (i.e., fumes, aerosols and dusts) and compounds too labile (reactive) for conventional GC analysis.

4.4 Detection Limits and Maximum Quantifiable Concentrations of Air Pollutants

4.4.1 Detection limits for atmospheric monitoring vary depending on several key factors. They are:

- Minimum artifact levels.
- GC detector selection.
- Volume of air sampled. The volume of air sampled is in turn dependent upon a series of variables including SSVs (see Section 10.8, Table 1 and Appendix 1 of Method T0-17), pump flow rate limitations and timeweighted- average monitoring time constraints.

4.4.2 Generally speaking, detection limits range from sub-ppb for volatile hydrocarbons in 1 L air samples using the GC/MS operated in the full SCAN mode.

4.4.3 Detection limits are greatly dependent upon the proper management of water for GC capillary analysis of volatile organics in air using sorbent technology (14).

4.5 Suitable Atmospheric Conditions

4.5.1 Temperature range.

4.5.1.1 The normal working range for sorbent packing is 0-40º C (8).

4.5.1.2 In general, an increase in temperature of 10ºC will reduce the breakthrough volume for sorbent packings by a factor of 2.

4.5.2 Humidity.

4.5.2.1 The capacity of the analytical instrumentation to accommodate the amount of water vapor collected on tubes is usually the limitation in obtaining successful results, particularly for GC/MS applications. This limitation can be extreme, requiring the use of a combination of water management procedures (see Section 4.2).

4.5.2.2 The safe sampling volumes of VOCs on hydrophobic adsorbents such as Tenax®, other porous polymers, Carbotrap™ and Carbopack™ are relatively unaffected by atmospheric humidity. Spherocarb® or carbonized molecular sieve type sorbents such as Carbosieve™ SIII and the Carboxens® are affected by high humidity, however, and SSVs should typically be reduced by a factor of 10 at 90-95% RH (8). Hydrophilic zeolite molecular sieves cannot be used at all at high humidity.

4.5.3 Wind speeds.

4.5.3.1 Air movement is not a factor indoors or outdoors at wind speeds below 10 miles per hour (<20 km per hour).

4.5.3.2 Above this speed, tubes should be orientated perpendicular to the prevailing wind direction and should be sheltered from the direct draft if wind speeds exceed 20 miles per hour (30-40 km per hour) (see Section 10.5).

4.5.4 High concentrations of particulates.

4.5.4.1 It may be necessary to connect a particulate filter (e.g., a 2 micron Teflon® filter or short clean tube containing a loose plug of clean glass wool) to the sampling end of the tube in areas of extremely high particulate concentrations.

4.5.4.2 Some compounds of interest may, however, be trapped on the Teflon® or on the glass wool. Particulates trapped on the sorbent tube have the potential to act as a source or sink for volatiles, and may remain on the tube through several cycles of sampling and desorption. Frequent replacement of the particulate filter

5.0 Specific Quality Control Requirements for Method T0-17

5.1 For general quality control requirements for determinative chromatography methods refer to the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air* for general quality control requirements. These requirements insure that each laboratory maintain a formal quality assurance program and records to document the quality of all chromatographic data. Quality Control procedures necessary to evaluate the GC system operation may be found in the published method and include evaluation of calibrations and chromatographic performance of sample analyses, instrument quality control and method performance requirements for the GC/MS system.

5.2 Specific QA/QC requirements and performance standards for Method T0-17 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the environmental professional ("EP") with "Reasonable Confidence" regarding the usability of analytical data to support DEP decisions

5.3 While optional, parties electing to utilize these protocols will be assured that "Reasonable Confidence" data, will be generally accepted by agency reviewers. In order to achieve "Reasonable Confidence" parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;

2. Evaluate and narrate, as necessary, compliance with performance standards prescribed in Table 1A for this test method; and

3. Adopt the reporting formats and elements specified herein.

6.0 Analyte List for Method T0-17

The Connecticut DEP (DEP) analyte list for Method T0-17 is presented in Table 1B.

6.1 Additional Reporting Requirements for Method T0-17

While it is not necessary to request and report all the analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEP strongly recommends that full list of analytes be reported during the initial stages of a site investigation and/or at sites with an unknown or complicated history of chemical usage or storage.

In cases where a shortened list of analytes is selected, the laboratory must still meet the method specific quality control requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

The Reporting Limit (RL) is based upon the lowest standard in the initial calibration. In order to meet the reporting limit for some compounds, it may be necessary to use select ion monitoring (SIM).

7.0 Tentatively Identified Compounds

7.1 The evaluation of Tentatively Identified Compounds (TICs) in conjunction with GC/MS analyses is a powerful and cost-effective analytical tool that can be utilized by the EP to support RSR due diligence requirements. This analytical approach is particularly effective at locations with suspect disposal practices, complex or uncertain site history, and/or sites that require detailed evaluation of critical exposure pathways. When GC/MS analytical methods are utilized an analysis of TICs is not usually expected but should be considered, at the discretion of the EP, in support of site characterization activities for releases at locations with complex and/or uncertain history.

7.2 Reporting of Tentatively Identified Compounds (TICs)

If evaluated, all TICs that meet the chromatographic criteria presented in Appendix A of this method must be reported by the laboratory either in the Environmental Laboratory Report or in the Environmental Laboratory's case narrative. In turn, the EP must include a discussion regarding the disposition of all reported TICs as part of the RSR submission to DEP. Depending on specific site circumstances (e.g., a potentially toxic contaminant is found in adjacent to a building, etc.), re-sampling/re-analysis with analyte-specific calibration and quality control may be required to definitively assess the risk posed by the TIC to human health and the environment. No regulatory judgments or remedial decisions should be made without re-analysis of samples for the TICs using a five-point analyte specific calibration and appropriate quality control. This may require re-sampling in order to meet analytical holding times.

8.0 Routine Reporting Deliverables for Method T0-17

The following table (Table 1.2) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report these items in special circumstances.

8.1 The following rules apply to reporting results:

Non-Detects: Report all non-detects and results below the reporting limit as "ND" (Below Quantitation Limit). The reporting limit for each compound in each sample must be in ppbv and ug/m^3 taking into account the exact sample volume, any dilution factors, etc.

Compounds detected above the reporting limit in blanks and found in samples, also above the reporting limit, shall be flagged with a "B" suffix (e.g. 25B).

Report concentrations above the upper level of the calibration curve with an "E" suffix.

Table 1A Specific QA/QC Requirements and Performance Standards for Method T0-17*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Sorbent Tube Cleaning and Certification	Assure tubes are free from contamination	Per Sections 6.2 & 10.2 of Method T0-17. Tubes must be identified per Section 10.3. One tube per batch must be analyzed for contamination and results documented.	NO - Data kept on file in lab.	Do not use contaminated or tubes of unknown history without cleaning.	Reclean as necessary.
Sampling System Components	Assure sampling system free form contamination	Per section 8.1 of the method. Pump calibrations must be documented. Tubes must be stored to prevent contamination as per Section 10.1 of Method T0-17.	NO – Records kept on file in lab	N/A	N/A
GC/MS Tunes with BFB	Inter-laboratory consistency and comparability	1) Criteria listed in Table 3 of Method TO-15 (the same criteria must be used for all analyses) 2) Every 24 hours	NO	Perform instrument maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified.
Initial Calibration	Laboratory Analytical Accuracy	1) Minimum of 5 standards. Standards must be prepared per Section 9 of the Method T0-17. 2) Low standard must be ≤ reporting limit (RL) 3) Must meet technical acceptance criteria as per Section 10.5.5 and 10.5.6 of Method TO-15. 4) Must contain all target analytes	NO	Recalibrate as required.	Sample analysis cannot proceed without a valid initial calibration. Report no non-conforming compounds in case narrative.
ICAL Verification Standard	Laboratory Analytical Accuracy	1) Each ICAL must be verified against a second source standard. 2) Std should be at mid-point 3) All target analytes present	NO	1) Compounds must recover within 80-120% 2) Laboratories are allowed to have 20% of compounds out, as long as all compounds within recover 65-135%	1) Perform maintenance as needed, recalibrate. 2) Note outliers in narrative.

Table 1A Specific QA/QC Requirements and Performance Standards for Method T0-17*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Daily Calibration Std (CCAL)	Laboratory Analytical Accuracy	1) Every 24 hrs prior to analysis of samples 2) Must meet criteria stated in Section 10.6 of the Method TO-15.	NO	Recalibrate as required by method.	Report non-conforming compounds in case narrative.
Method Blanks	Laboratory Contamination Evaluation	1) Every day prior to running samples and after calibration standards. 2) Must meet criteria as stated in Sections 10.7 & 13.1 of Method T0-17.	YES	Locate source of contamination and correct problem. Reanalyze method blank.	1) Report non-conformances in case narrative. 2) All results for compounds present in method blank must be "B" flagged if detected in samples associated with the method blank.
Audit Accuracy or Laboratory Control Sample (LCS)	Laboratory Method Accuracy	1) Every 20 samples or weekly, whichever is more frequent. 2) Standard source different from initial calibration source. 3) Concentration level near or at the mid-point of the initial calibration. 4) Must contain all target analytes 5) Must meet the criteria of Section 14 of Method T0-17.	YES	Recalculate the percent recoveries Reanalyze the LCS Locate & correct problem, reanalyze associated samples	1) Report non-conformances in case narrative. 2) Individual laboratories must identify and document problems.
Sample Replicates	Method Precision	1) Every 20 samples collect and analyze one sample in duplicate. 2) RPD's should be ≤ 25% for compounds present above the concentration of the low std.	YES	If more than 10% of analytes present above the concentration of the low std fail the RPD criteria, investigate system. Reanalyze CCAL to verify system performance.	Report non-conformances in case narrative

Method Detection Limit Study	System sensitivity	1) Performed annually per Section 14.2 of Method T0-17. 2) MDL's for all compounds should be ≤ 0.5 ppbv	NO – Data kept on file in lab	Reanalyze MDL study	

Table 1A Specific QA/QC Requirements and Performance Standards for Method T0-17*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Sample Analysis	Valid Results	1) Analyze per Section 11 of Method T0-17.	YES	N/A	Note any analytical problems in the narrative. Flag all compounds over the highest calibration std with an "E" suffix.
Internal Standards (IS)	Laboratory Analytical Accuracy and Method Accuracy in Sample	1) Laboratory must use a minimum of 3 IS at retention times across the GC run. See Section 9.4 of Method T0-17. 2) Must meet criteria of Sections 10.8.4 and 10.8.5 of Method TO-15.	NO	1) Evaluate the analytical system for malfunctions and correct	1) Note exceedances in narrative
Quantitation	N/A	1) Quantitation must be based on IS calibration 2) Quantitation based on Section 10.8.4 of the Method TO-15 3) The IS used for quantitation must be the IS nearest to the retention time of the target analyte.	N/A	N/A	Note any problems in narrative.
General Reporting Issues	N/A	1) The laboratory should report only concentrations detected above the sample specific RL. 2) Concentrations below the reporting limit (RL) as "ND" with the reporting limit. 3) Compounds, which exceed the linear range, should be flagged ("E" flag).	N/A	N/A	1) Qualification of results reported below the RL is required. 2) Performance of dilutions must be documented in the case narrative

Footnotes for Table 1A:

* Refers to latest published version of Method T0-17.

GC/MS = Gas Chromatography/Mass Spectrometry
BFB = 4-Bromofluorobenzene
%RSD = Relative Percent Standard Deviation
RPD = Relative Percent Difference
CCC = Calibration Check Compound
N/A = Not Applicable

Table 1.2 Report Deliverables

Parameter	Deliverable	Comments
GC/MS Tunes	NO	Analysis cannot proceed without meeting tuning criteria.
Initial Calibration	NO	Note non-conformances in narrative
Continuing Calibration	NO	Note non-conformances in narrative
Method Blanks	YES	Note non-conformances in narrative. Flag all positive results above RL with "B" flag.
Lab Control Sample (LCS)	YES	Note non-conformances in narrative
Sample Replicate	YES	Note non-conformances in narrative
Internal Standard Areas	NO	Note non-conformances in narrative
General Reporting Issues	YES	Note non-conformances in narrative

Table 1.3 GC/MS Tune Criteria for BFB

m/z	Required Intensity (relative abundance)
50	8-40% of m/z 95
75	30-60% of m/z 95
95	Base peak, 100% relative abundance
96	5 – 9% of m/z 95
173	Less than 2% of m/z 174
174	50 – 120% of m/z 95
175	4 – 9% of m/z 174
176	93 - 101% of m/z 174
177	5 – 9% of m/z 176

The mass spectrum of BFB should be acquired in the following manner. Three scans (the peak apex and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and may be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not subtract part of the BFB peak. Alternative BFB criteria, such as the Method 524.2 criteria, is allowed provided all samples, standards, blanks, etc. are analyzed using the same GC/MS tuning criteria. If alternative approaches are utilized, the approach must be documented in the laboratory standard operating procedure. The laboratory is not allowed to vary its approach from day to day in order to in order to pass a tune on an instrument requiring maintenance.

Table 1B Analyte List For Method T0-17

Analyte	CAS Number	Notes
Acetone	67641	
Acrylonitrile	107131	
Benzene	71432	
n-Butylbenzene	104518	
Sec-Butylbenzene	135988	
Bromodichloromethane	75274	
Bromoform	75252	
2-Butanone (MEK)	78933	
Carbon Tetrachloride	56235	
Chlorobenzene	108907	
Chloroethane	75003	
Chloroform	67663	
Chloromethane	74873	
Dibromochloromethane	124481	
1,2-Dibromoethane (EDB)	106934	
1,2-Dichlorobenzene	95501	
1,3-Dichlorobenzene	541731	
1,4-Dichlorobenzene	106467	
Dichlorodifluoromethane	75718	
1,1-Dichloroethane	75343	
1,2-Dichloroethane	107062	
1,1-Dichloroethene	75354	
cis-1,2-Dichloroethene	156592	
trans-1,2-Dichloroethene	156605	
1,2-Dichloropropane	78875	
1,3-Dichloropropane	142289	
cis-1,3-Dichloropropene	10061015	
trans-1,3-Dichloropropene	10061026	
Ethylbenzene	100414	
Isopropylbenzene (Cumene)	98828	
4-Isopropyltoluene	99876	
Methylene Chloride	75092	
4-Methyl-2-pentanone (MIBK)	108101	
Methyl-tert-butylether (MTBE)	1634044	
Styrene	100425	
1,1,1,2-Tetrachloroethane	630206	
1,1,2,2-Tetrachloroethane	79345	
Tetrachloroethene (Perc)	127184	
Toluene	108883	
Toluene	108883	
1,1,1-Trichloroethane	71556	
1,1,2-Trichloroethane	79005	
Trichloroethene (TCE)	79016	
Trichlorofluoromethane	75694	

Table 1B Analyte List For Method T0-17 (cont)

Analyte	CAS Number	Notes
1,2,4-Trimethylbenzene	95636	
1,3,5-Trimethylbenzene	108678	
Vinyl Chloride	75014	
o-Xylene	95476	1
m-Xylene	108383	1
p-Xylene	106423	1

Footnotes
1. May be reported as total xylenes or any combination of the three isomers.

Appendix A

Laboratory Requirements for Evaluation of

Tentatively Identified Compounds

Method TO-17

A-1. Chromatographic Criteria

A-1.1 Initially include all of the non-target compounds that elute 30 seconds before the first target compound and 3 minutes after the elution of the last target compound. The peak area count of the unknown compound must also be ≥ 10% of the nearest internal standard. The EP may request evaluation of unknown peaks before the first internal standard based on site-specific information.

A-2. Mass Spectral Criteria

A-2.1 All spectra must be evaluated by a qualified mass spectrometrist and the Organic Supervisor/Laboratory Director.

A-2.2 The spectral library match must be ≥ 85% for a tentative identification to be made.

A-2.3 The major ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample spectrum.

A-2.4 The relative intensities of the major ions must agree within ± 20%.

A-2.5 Molecular ions present in the reference spectrum should be present in the sample spectrum.

A-2.6 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks.

A-2.7 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different chromatographic retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs (as a mixture of two isomers).

A-2.8 Spectra identified as “unknown” should be assigned to a general chemical class, if possible. Classification as a halogenated hydrocarbon, aldehydes/ketone, carboxylic acid, or cyano compound, etc. is acceptable. An explanation as to why more specific identification cannot be made (e.g., truncated spectra due to insufficient mass scanning range) must be provided in the analytical laboratory case narrative to support any “unknown” classification.

A-2.9 TICs which are identified as petroleum aliphatic hydrocarbons should not be reported as TICs. TICs identified as aromatics or other hydrocarbons should be reported. However, there must be a statement in the laboratory case narrative discussing the presence of these hydrocarbons in the sample(s).

A-2.10 After the above criteria are met, the top ten (10) compounds for VOCs, chosen by comparing the area of the TIC to the area of the nearest internal standard, must be tentatively identified, quantitated, and reported. All TIC concentrations should be flagged as estimated by using a “J” suffix.

A-3. Toxic Spectral Characteristics Criteria

A-3.1 Regardless of the number of peaks present, the laboratory must evaluate any peak where the mass spectrum exhibits a characteristic chlorine or bromine spectral pattern. This only applies to peaks having an area >10% of the nearest internal standard.

A-4. Semi-Quantitative Analysis

A-4.1 Once a TIC has been identified, the semi-quantitation of that compound will be based on the integrated abundance of the TIC and internal standard total ion chromatogram. The response factor for all TICs will be assumed to be 1.0. The internal standard used shall be the one with the nearest retention time to a given TIC and that is interference free.

A-4.2 The resulting semi-quantitative concentration must be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine the concentration.

A-5. Reporting Criteria

A-5.1 All TICs eluting after the first internal standard and 3 minutes after the last target compound meeting the requirements in A-2 must be reported by the laboratory with the clear indication that the reported concentration is an estimated value unless analyte-specific calibration and QA/QC were performed. This reporting requirement may be fulfilled by discussion in the laboratory case narrative or by using a “J” flag designation.

NOTE: In most circumstances the laboratory must order standards in order to be able to run a calibration curve and the appropriate QA/QC. The EP should be prepared to expect longer analytical turn-around-times in order to attain TIC results that are scientifically defensible.