State of Connecticut

Department of Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Volatile Organics by Method T0-15

Version 2.0

December 2006

Written by the Connecticut DEP QA/QC Workgroup

Revision	Comments	Date
1.0	Posted on EPOC Website	February 2006
2.0	Final version based upon public comments	December 2006

TABLE OF CONTENTS

LIST OF TABLES AND APPENDICES

1.0 Overview for Method TO-15

Method TO-15 gas chromatography/mass spectrometry procedure used to determine volatile organic compounds (VOC's) in air. This procedure requires an experienced GC/MS analyst familiar with air analysis using Summa canisters and the QA/QC requirements of the method. 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 atmosphere is sampled by introduction of air into a specially-prepared stainless steel canister. Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister. A pump ventilated sampling line is used during sample collection with most commercially available samplers. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into the pre-evacuated and passivated canister.

2.2 After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to the laboratory for analysis.

2.3 Upon receipt at the laboratory, the canister tag data is recorded and the canister is stored until analysis. Storage times of up to thirty days have been demonstrated for many of the VOCs (5).

2.4 To analyze the sample, a known volume of sample is directed from the canister through a solid multisorbent concentrator. A portion of the water vapor in the sample breaks through the concentrator during sampling, to a degree depending on the multisorbent composition, duration of sampling, and other factors. Water content of the sample can be further reduced by dry purging the concentrator with helium while retaining target compounds. After the concentration and drying steps are completed, the VOCs are thermally desorbed, entrained in a carrier gas stream, and then focused in a small volume by trapping on a reduced temperature trap or small volume multisorbent trap. The sample is then released by thermal desorption and carried onto a gas chromatographic column for separation. As a simple alternative to the multisorbent/dry purge water management technique, the amount of water vapor in the sample can be reduced below any threshold for affecting the proper operation of the analytical system by reducing the sample size. For example, a small sample can be concentrated on a cold trap and released directly to the gas chromatographic column. The reduction in sample volume may require an enhancement of detector sensitivity. Other water management approaches are also acceptable as long as their use does not compromise the attainment of the performance criteria listed in Section 11. A listing of some commercial water management systems is provided in Appendix A. One of the alternative ways to dry the sample is to separate VOCs from condensate on a low temperature trap by heating and purging the trap.

2.5 The analytical strategy for Compendium Method TO-15 involves using a high resolution gas chromatograph (GC) coupled to a mass spectrometer. If the mass spectrometer is a linear quadrupole system, it is operated either by continuously scanning a wide range of mass to charge ratios (SCAN mode) or by monitoring select ion monitoring mode (SIM) of compounds on the

target list. If the mass spectrometer is based on a standard ion trap design, only a scanning mode is used (note however, that the Selected Ion Storage (SIS) mode for the ion trap has features of the SIM mode). Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This establishes the compound concentration that exists in the sample. Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification. If the technique is supported by a comprehensive mass spectral database and a knowledgeable

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 Contamination

4.1 Very volatile compounds, such as chloromethane and vinyl chloride can display peak broadening and co-elution with other species if the compounds are not delivered to the GC column in a small volume of carrier gas. Refocusing of the sample after collection on the primary trap, either on a separate focusing trap or at the head of the gas chromatographic column, mitigates this problem.

4.2 Interferences in canister samples may result from improper use or from contamination of: (1) the canisters due to poor manufacturing practices, (2) the canister cleaning apparatus, and (3) the sampling or analytical system. Attention to the following details will help to minimize the possibility of contamination of canisters.

4.2.1 Canisters should be manufactured using high quality welding and cleaning techniques, and new canisters should be filled with humidified zero air and then analyzed, after “aging” for 24 hours, to determine cleanliness. The cleaning apparatus, sampling system, and analytical system should be assembled of clean, high quality components and each system should be shown to be free of contamination.

4.2.2 Canisters should be stored in a contaminant-free location and should be capped tightly during shipment to prevent leakage and minimize any compromise of the sample.

4.2.3 Impurities in the calibration dilution gas (if applicable) and carrier gas, organic compounds out-gassing from the system components ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with Buna-N rubber components must be avoided.

4.2.4 Significant contamination of the analytical equipment can occur whenever samples containing high VOC concentrations are analyzed. This in turn can result in carryover contamination in subsequent analyses. Whenever a high concentration (>25 ppbv of a trace species) sample is encountered, it should be followed by an analysis of humid zero air to check for carry-over contamination.

4.2.5 In cases when solid sorbents are used to concentrate the sample prior to analysis, the sorbents should be tested to identify artifact formation (see Compendium Method TO-17 for more information on artifacts).

5.0 Specific Quality Control Requirements for Method TO-15.

5.1 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 TO-15 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 and Licensed Environmental Profession (LEP) with "Reasonable Confidence" regarding the usability of analytical data to support DEP decisions

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 Tentatively Identified Compounds

6.1 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.

6.2 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.

7.0 Analyte List for Method TO-15

The Connecticut DEP (DEP) analyte list for Method TO-15 is presented in Table 1B. The compounds listed are readily determined by Method TO-15

7.1 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.

7.2 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.

7.3 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).

8.0 Routine Reporting Deliverables for Method TO-15

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 Reporting and Flagging of Results

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 ppbv and take into account the exact sample mass, 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 results for any library search compounds as estimated using a “J” suffix (e.g. 25J).

Results should be reported both in ppbv and ug/m^3.

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method TO-15*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Canister Cleaning and Certification	Assure canister are free from contamination	Per Section 8.4.1 of the method.	NO - Data kept on file in lab.	Do not use contaminated canisters.	Reclean as necessary.
Sampling System Components	Assure sampling system free form contamination	Per section 8.4.3 of the method.	NO – Records kept on file in lab	Do not collect samples with a contaminated system.	Reclean as necessary.
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. 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. 4) Must contain all target analytes	NO	Recalibrate as required by method.	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.
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	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 Section 10.7 of the method.	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.

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method TO-15*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
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 11.4 of the method.	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 analyze one sample, preferably one with detected analytes, 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, reanalyze duplicate.	Report non-conformances in case narrative
Method Detection Limit Study	System sensitivity	1) Performed annually per Section 11.2 of the Method. 2) MDL's for all compounds should be ≤ 0.5 ppbv	NO – Data kept on file in lab	Reanalyze MDL study	
Sample Analysis	Valid Results	1) Analyze per Section 10.8 of the method. 2) Must meet technical acceptance criteria of Section 10.8.	YES	If technical criteria not met: 1) Evaluate the analytical system for malfunctions and correct 2) Reanalyze the sample	Note any analytical problems in the narrative.
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. Use of IS in Section 9.2.2.3 of method is strongly recommended. 2) Must meet criteria of Sections 10.8.4 and 10.8.5 of the method.	NO	1) Evaluate the analytical system for malfunctions and correct 2) Reanalyze the sample	1) Note exceedances in narrative 2) If reanalysis confirms matrix interference, report initial analysis and note in narrative 3) If reanalysis in criteria, report only compliant analysis

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method TO-15*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Quantitation	N/A	1) Quantitation must be based on IS calibration 2) Quantitation based on Section 10.8.4 of the method. 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) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for both sets of data. Compounds which exceed the linear range should be flagged ("E" flag). 6) If a dilution is performed, the highest detected analyte must be in the upper half of the calibration curve, unless there are non-target analytes whose concentrations are so high as to cause damage to the instrumentation or saturate the mass spectrometer.	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 TO-15.

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 TO-15

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	

TABLE 1B ANALYTE LIST FOR METHOD TO-15 (con't)

Analyte	CAS Number	Notes
Toluene	108883	
1,1,1-Trichloroethane	71556	
1,1,2-Trichloroethane	79005	
Trichloroethene (TCE)	79016	
Trichlorofluoromethane	75694	
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-15

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.