3M Environmental Laboratory

Method

Analysis of Volatile Organic Compounds Using Purge and Trap Gas Chromatography/Mass Spectrometry Using EPA Method 8260C

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Approved By:

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1 Scope and Application

This method is used to quantitate most volatile organic compounds contained in an aqueous phase, adsorbed onto a solid matrix, or in a gas-phase that have boiling points below 200ºC. It is applicable to all types of compounds that elute as peaks from a GC column and are amenable to quantitation using a mass spectrometer operated in either the scan mode or in a selected ion monitoring (SIM) mode of operation.

This method is in accordance with EPA method 8260C titled "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GCMS)" and the sample introduction technique described by EPA Method 5030 titled "Purge-and-Trap for Aqueous Samples". This method is applicable to the analysis of volatile compounds in water and the determination of physical and chemical properties, including, but not limited to, water solubility, octanol:water partition coefficients, and Henry's Law constants.

2 Method Summary

Volatile compounds are introduced into the gas chromatograph using the purge and trap method. An inert gas is bubbled through or above an aqueous solution at ambient or elevated temperature, transferring the volatile components from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column (trap) where the components are adsorbed. This trap is heated and back-flushed to transfer the gaseous analytes to a temperature-programmed chromatographic column for analyte separation and mass spectrometer detection and quantitation.

Identification of target analytes is accomplished by comparing their retention time and mass spectra to authentic standards. Tentative identification of an unknown compound may be made by comparing a full scan spectrum of the unknown to a NIST library mass spectral search. An estimated concentration of a non-target analyte identified in a sample may be made using the response factor of the nearest internal standard free of interferences.

3 Definitions

Water Mode of Operation. Each sample vial is a zero-headspace, screw-top septum sealed vial. The purge and trap autosampler transfers an aliquot to the sparge vessel where it is purged with helium. As sample is transferred to the purging vessel, the autosampler may fortify internal and surrogate standards.

Soil Mode of Operation. Each sample vial is non-zero headspace, screw-top septum sealed vial. The autosampler adds water that may be fortified with internal and surrogate standards to the partially filled vial. The autosampler purges the entire contents of the vial without a sample transfer step to a separate purging vessel. This soil mode of operation is suitable for analysis of vials containing water, soil, or gas-phase analytes. Purging typically takes place at an elevated temperature of 40ºC, but may be at ambient or other elevated temperatures. At the 3M Environmental Laboratory, soil mode is typically used to analyze samples for determination of physical/chemical properties.

4 Warnings and Cautions

The operator must be familiar with the purge and trap and GC/MS systems and their associated hazards, such as high temperature, effluent venting, solvent use, moving autosampler parts, and low-pressure vacuum system. Refer to 3M Environmental Laboratory Guidance Document ETS-17-012 titled "Laboratory Handling of Hazardous Compressed Gas Sample Materials Safety Procedure" and the appropriate equipment procedures, methods, SOPs, or operator manuals for additional information and cautions.

All exhaust vents, including the sample concentrator purge vent, split vent and mass spectrometer pump exhaust must be connected to a laboratory hood or vented instrument enclosure to keep potentially hazardous effluent from mixing with laboratory air.

Wear appropriate protective gloves, eyewear, and clothing when handling samples or solvents.

5 Interferences

Impurities present in solvents, purge gas, and organic compounds out-gassing from the components of the analytical system are potential sources of contamination. Sample integrity can be influenced by diffusion of volatile organic materials through the septum seal of the vial.

Contamination by carryover can occur whenever high-concentration and low concentration samples are analyzed sequentially. The analysis of blank samples is used to check for cross-over contamination. Carryover may be reduced by adjusting analytical parameters, such as line and valve temperatures, carrier gas split flows, and bake times and temperatures of the analytical and moisture control traps.

6 Instrumentation, Supplies, and Materials

A variety of vendors and models for purge and trap GC/MS systems may be used. Any combination of these or other suitable equipment may be used, provided all data quality objectives are met.

Purge and Trap Instrumentation

EST Analytical Centurion Purge and Trap Autosampler and EST Analytical ENCON Evolution Sample Concentrator

Varian Archon Purge and Trap Autosampler and Teledyne Tekmar Stratum Purge and Trap Sample Concentrator

GC/MS Instrumentation

Agilent Technologies 7890A GC System with 5975C inert XL EI/CI MSD with Triple-Axis Detector

Agilent Technologies 7890B GC System with 5977 Mass Spectrometer

Agilent Technologies 6890A GC System with 5973 Network Mass Selective Detector

Agilent Technologies 6890N GC System with 5973 Inert XL Mass Selective Detector

Hewlett Packard 5890N GC System with 5973 Mass Selective Detector

Supplies and Materials

The following list of supplies and materials is not exhaustive, but rather provides a guide of what the experienced purge and trap GC/MS operator may use during the course of a study.

Analytical Column. Agilent J & W DB-624, 0.25mm, 30m, 1.4µm df or other column type, diameter, length or stationery phase to provide suitable analyte retention and resolution.

Analytical Trap. K Trap (Vocarb 3000) provided by instrument manufacturer. Different analytical traps may be used depending on the adsorptive properties of the target analyte(s).

Autosampler Vials. Vials are referred to as VOA vials (Volatile Organic Analysis vials). VOA vials consist of a 40 mL (nominal) VOA vial assembled with 24mm bonded septa cap, pre-cleaned and certified from Greenwood Products, Inc. (Part No. 03-4033BTS1443, or equivalent). These vials have been experimentally determined by weight to contain 42.5 mL of water. I-CHEM™ Brand vials with two-piece caps may also be suitable if shown to meet data quality objectives.

GC Carrier Gas. Helium, ultra high purity or equivalent.

Miscellaneous. Gas-tight glass microsyringes, single-use plastic syringes with disposable needles, volumetric flasks, disposable pipettes, mininert re-closable valves, gloves, and solvents of suitable grade for the intended use are some of the required supplies generally needed to complete studies using this method. Analytical balances, orbital shakers, centrifuges, incubators, and other devices may also be required. Identification of these devices and the settings used will be included in the raw data as appropriate.

7 Reagents and Standards

Methanol: HPLC grade or equivalent. Purge and Trap grade (higher purity) may also be used.

ASTM Type I Water: Milli-Q™ water or equivalent provided by a Milli-Q TOC Plus system, or other suitable system.

Internal Standards (ISTD): The autosampler may be programmed to add ISTD solution to every sample, blank, and standard. The ratio of the response of the target analyte to internal standard is used for quantitation. With justification, an external standard calibration may be used.

Purchased EPA 8260C or internal standard solutions prepared in the lab can be added by the autosampler or by an analyst directly to each vial. Alternatively, a solvent or gas phase ISTD may be spiked by the analyst into each vial analyzed in the soil mode.

Surrogate Standards: A known amount of a standard mixture may be added separately from the ISTD to every sample, blank, and standard to evaluate accuracy and precision. Surrogates are not required for all studies, particularly for laboratory generated (i.e. non-environmental) samples.

Solvents: Additional solvents, including, but not limited to, acetone, n-octanol and ethyl acetate, may be used in limited studies. The source and purity of solvents used will be documented in the raw data.

8 Standard and Sample Handling

Stock solutions may be prepared in the lab from neat liquids, solids, or gaseous materials or may be purchased as certified solutions. Stock solutions are prepared in methanol or other appropriate solvent and then further diluted to appropriate working concentrations. Analyte concentrations are adjusted for purity as appropriate. Stock standards prepared from liquids or gases may be stored in a refrigerator or freezer to minimize possible losses due to volatility.

Liquid Stock Standard Preparation: Liquid reference materials should be added to a tared ground glass-stoppered volumetric flask partially filled with the dilution solvent so that the liquid falls onto the surface of the solvent. Reweigh, dilute to volume, and mix by inverting the flask three times. Additional mixing should be done only if needed to ensure homogeneity or to get the liquid into solution. Calculate the concentration by the net weight gain. Alternately, liquids may be injected directly into the solvent with a syringe to minimize possible evaporation and allow the analyst to visually inspect the mixing process.

Solid Stock Standard Preparation: Solid reference materials may be weighed into an empty tared volumetric flask. Dilute to volume with appropriate solvent. The flask may be mixed vigorously or sonicated as needed to ensure that the solution is homogenous. If sonication is required, ensure that the flask equilibrates to room temperature prior to use or for making secondary solutions due to possible volume changes associated with the increased temperatures due to sonication.

Gas Standard Preparation in Solvent: For neat gaseous materials, fill a gas tight syringe with reference material and slowly introduce the gas above a tared volumetric-flask containing solvent. The gas will dissolve into the solvent. Reweigh, dilute to volume, and mix by inverting the flask three times. Calculate the concentration by the net weight gain.

Gas Phase Standard Preparation: Gas standards may be prepared in a pressurized canister in accordance to 3M Environmental Laboratory Method ETS-8-196 titled “Gas Standard Preparation”. Gas standards injected through the septum of a sealed vial may be analyzed in the soil mode of operation with or without the presence of water because the entire contents of the vial are purged through the adsorbent trap. Intermediates are prepared in sealed, empty VOA vials. An example calculation of an intermediate prepared by adding 5.00 cubic centimeters (cc) of gas from a 100 ng/cc summa canister stock standard into a VOA vial previously determined to have a volume of 42.5 cc is as follows:

$$Concentration\ of\ Intermediate = \frac{5cc * 100\ ng/cc}{(42.5 + 5)cc} = 10.5\ ng/cc$$

This 10.5 ng/cc standard might then be used to prepare calibration standards that contain reagent water equal to the volume of the analytical sample analyzed. For example, if 0.5 cc of the 10.5 ng/cc were added to a VOA vial containing 10.0 mL of reagent water, the calibration standard will be at a level of 5.25 ng (0.525 ng/mL).

Intermediate gas standards prepared in VOA vials are spiked with up to 5.00 cc of gas phase standards and are used to prepare further dilutions or spikes. Samples analyzed are typically spiked with a volume ranging from 0.025 cc to 3.00 cc.

Intermediate standard solutions are typically prepared in methanol using volumetric flasks. Partially fill the flask with methanol and add the appropriate volume of stock solution. Fill to volume with methanol and immediately recap. Mix by inverting the flask a minimum of three times.

Analytical standards are prepared in reagent water. Use a micro-syringe to inject the solvent standard into the expanded area of the volumetric flask underneath the surface of the reagent water. Remove the needle as quickly as possible after injection and dilute to the volume mark with additional reagent water. Mix by inverting the flask three times.

For water mode analysis, a VOA vial is filled by pouring sample to near overflowing and is capped to contain zero headspace. The automated sampler transfers a specific volume of water and internal and surrogate standard as appropriate to the purging vessel.

For soil mode analysis, an aliquot of water between 5 ml to 20 mL is dispensed into an empty vial that is immediately capped. Standard aliquots may also be dispensed through the septum using a syringe. In this case, the cap is loosely fitted (or a vent needle inserted through the septum of a tightly fitted cap) to allow the displaced air to escape. The cap immediately tightened after the addition.

Alternatively, inject an aliquot of a solvent standard through the septum into a volume of water for the water or soil mode. Standards should contain no more than 1% methanol to avoid negative effects on chromatography. Standards are typically purged within 24 hours, but are given a two day expiration date to allow a lengthy analytical sequence to finish.

9 Quality Control

Data quality objective requirements vary for different studies. Unless specified in a protocol or general project outline, precision and accuracy requirements for Laboratory Matrix Spikes and Laboratory Control Spikes are 25% RSD (or RPD) and 100%±30%, respectively. Precision and accuracy assessments based on these spike results are described in each analytical report.

Method Blanks

A method blank is used to document possible contamination resulting from the analytical process. It is carried through the complete sample preparation and analytical procedure. It is recommended that a minimum of one method blank per ten study samples be prepared. For example, the method blank should be prepared and analyzed in triplicate for a study containing 21 to 30 study samples.

Instrument Blank

An instrument blank is a sample of analyte-free medium that is not taken through the entire sample preparation process, but used to evaluate instrument background levels. Instrument blanks are analyzed after calibration standards, continuing calibration verification (CCV) standards, after known highly concentrated samples, and periodically throughout an analytical sequence at the discretion of the analyst. If successive instrument blanks are analyzed, only the last injection need be evaluated. If an instrument blank within an analytical sequence does not meet criteria, technical justification may be made to accept the data. For example, if trace contaminants are present in a blank and the next sample does not contain analyte above the LOQ, the data may be accepted because the sample is unaffected by any contamination bias shown in the previous blank. Conversely, if trace contaminants are present in a blank and the next sample contains analyte at a significantly higher concentration, the data may also be accepted. In this instance, subsequent samples are evaluated for evidence of background contamination.

Replicate Analysis

A replicate analysis is defined as separate aliquot(s) from the same study sample. Results should agree within 20% RPD (duplicates) or 20% RSD (triplicates) to be used without technical justification. One example of technical justification would be that replicate sample values are near the limit of quantitation where the quantitative measurements are more susceptible to variability.

Internal Standard

Internal standard areas counts may drift throughout an analytical sequence. The areas should be monitored for anomalies that suggest that a problem exists with a specific sample. Specifically, if the area of the internal standard in a sample changes by a factor of two (-50% to + 100%) from the mid-level standard in the initial calibration or the most recent CCV as appropriate, technical justification must be made in order to use the data.

Surrogate

Surrogates standards may be required when analyzing environmental samples that are not fortified at the laboratory. Recoveries should be within 100%±25% to be used without technical justification. Surrogates may be fortified into each sample during the preparation to fully evaluate matrix effects or added to each sample vial by the autosampler. Surrogate standards are not required for most physical and chemical property studies.

Lab Control Spike

A laboratory control spike (LCS) is a sample of analyte-free medium fortified with the target compound and brought through the same analytical steps as other samples. LCS results are used to evaluate the method accuracy and precision in samples that are not influenced by sample matrix. Acceptance criteria are 25% RPD (or RSD) and 100%±30% accuracy to be used without technical justification.

A successful LCS analysis coupled with a failed matrix spike analysis suggests that instrument is performing correctly but the sample matrix may affect the accuracy of results and therefore must be discussed in the report. A minimum of one LCS must be analyzed with each set of 20 samples or fewer. If insufficient volume of sample exists to complete an MS/MSD set, an LCS Duplicate should also be analyzed.

Lab Matrix Spike

A lab matrix spike (LMS) is an aliquot of a sample fortified with the target compound(s). Results are used to evaluate the method accuracy and precision including possible matrix interference(s). LMS standard solutions may be from the same source as the initial calibration standards to restrict the influence of accuracy on the determination of recovery throughout the analysis. For environmental samples, the matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed. For laboratory prepared samples, the matrix spiking solution should contain the target analytes of interest. Acceptance criteria are 100%±30% accuracy and %RSDs or RPDs should be $\leq$ 25% to be used without technical justification.

A minimum of one Lab Matrix Spike and Matrix Spike Duplicate (MS/MSD) set should be analyzed with every 20 samples or fewer. The targeted spike levels should be two to ten times the concentration of the target analytes present in the sample. A concentration near the mid-point of the curve may be spiked if the expected concentrations are not known.

Method Detection Limit (MDL) Determination

The MDL is the minimum concentration of a substance that can be measured with 99% confidence that the analyte concentration is greater than zero. An MDL may be determined by multiplying the one-sided 99% t-statistic (3.14 for seven replicates) by the standard deviation obtained from the analysis of seven lab control spikes fortified at a concentration at or near the LOQ. The MDL should be less than the LOQ to meet data quality objectives. Any outliers, including but not limited to common lab solvents, may be accepted if the failing compound(s) are not critical to a specific project. An MDL study may be repeated for the outlier(s) if needed to meet project requirements.

An MDL study is not required for studies where samples are generated by the lab to determine physical and chemical properties. For these studies, the lowest level calibration standard is defined as the LOQ. Sample concentrations are typically measured above the LOQ and therefore an MDL study is not required.

Initial Demonstration of Capability (IDC)

A minimum of four replicates of reagent water fortified near the mid-point of the calibration curve should be analyzed. Each average recovery should be 70-130%. Each RSD should be less than 20%. The large number of analytes in Method 8260C presents a substantial probability that one or more analyte will fail at least one of the performance criteria. If any individual average recovery or RSD falls outside the range, system performance may be unacceptable for that analyte, meaning that the analyst must closely evaluate instrument performance prior to reporting that analyte. The IDC may be repeated for the failed analyte(s) or may be reported with an appropriate statement of accuracy.

An IDL is not required for studies where samples are generated by the lab to determine physical and chemical properties.

10 Calibration and Standardization

Instrument Calibration

Samples are quantitated against a standard curve containing varying amounts of test substance and a fixed amount of internal standard. The curve is calculated from the plot of individual calibration points using Target ™ software or equivalent chromatography and/or data reduction software. A set of six or more calibration standards is analyzed at the beginning of each analytical study and within a study if Continuing Calibration Verification standards do not meet the criteria (see next section). The acceptance criterion for the residual of each standard is 100±25%, but is 100±30% at the lower limit of quantitation (LLOQ). Low or high curve points may be deactivated, depending on instrument sensitivity, linearity of response, and levels required to bracket sample concentrations. Curve points not at either end of the curve may be deactivated or re-prepared if there is evidence of instrument malfunction or preparation error.

The average relative response factor may be used for quantitation if the relative standard deviation (RSD) of the relative response factors (RRF) is <20% for any analyte as it is assumed that the RRF is constant over the calibration range. If the RSD of any target analyte is greater than 20%, a linear or quadratic curve fit with or without weighting are options for quantitation. The regression fit may include, but not be forced through, the origin. A minimum correlation coefficient of 0.990 is required.

After completing an 8260 calibration curve from newly prepared stock standard solutions, analyze a minimum of one standard using a stock solvent solution from a separate preparation than what was used to make the calibration standards. This preparation should be from a different ampoule of a purchased standard or from a stock solution prepared from a separate weighing of neat material. The acceptance limits for this initial calibration verification (ICV) are 70–130%. If replicate analyses are made, the average results are evaluated. Analysis may continue for analytes that fail the criteria with an understanding results are considered estimated values or reported with an expanded uncertainty.

Alternately, the stability of analytes and the continued accuracy of the calibration curve may be verified concurrently or at the end of a study by the analysis of a separately prepared standard in compliance with the requirements of an appropriate regulatory program.

Continuing Calibration Verification (CCV)

After a successful initial calibration curve, the continued accuracy of the curve may be shown by the analysis of, one or more calibration check standards at the beginning of a new analytical sequence, after every ten study samples or fewer, after an elapsed time of 12 hours (environmental samples only), and at the end of the analytical sequence.

Only samples bracketed by a successful CCV may be reported without technical justification. If evidence exists that a vial leaked, the instrument malfunctioned, or that an anomaly existed during CCV analysis, the standard may be re-prepared. If successive CCVs were analyzed to demonstrate continued accuracy of the calibration, the successful CCV may be used to accept results.

The CCV concentration is typically near the mid range of the curve, but may be varied. For example, low level CCVs may be used for low level samples, whereas a low and a high CCV may be analyzed in succession to further document the continued accuracy of the entire curve range.

System Suitability

The purge and trap GC/MS system is deemed suitable for an intended use if the GC/MS generates acceptable auto tune reports and if an initial calibration or CCV meets the data quality objectives.

EPA Method 8260C requires that an aliquot of 50 ng, or less, of 4-Bromofluorobenzene (BFB) be analyzed every 12 hours or less to verify that the full scan mass spectrum produced meets certain criteria. Three scans across the apex of the peak should be acquired and averaged. Background subtraction is required to eliminate column bleed or instrument background ions.

Table 1. BFB Mass Spectrum Intensity Criteria

m/z	Required BFB Mass Intensity Criteria (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

The analysis of BFB is required prior to method 8260 scan mode analysis of environmental samples. However, the analysis of BFB is not appropriate and therefore not required when the SIM mode of operation is used to quantitate study samples to determine physical and chemical properties. This is true because the mass spectrometer may be tuned using algorithms to enhance particular mass ranges appropriate to maximize sensitivity of the target analyte and therefore the tuning criteria (Scan Mode) are not appropriate. The analysis of BFB is also not required if full scan analysis is utilized only to confirm the identity of a target analyte, contaminant, or degradation product during a study.

11 Procedures

Purge and trap conditions and GC/MS parameters are set to provide suitable chromatography and sensitivity for the intended purpose. Analytical conditions used for a study will be influenced by properties such as the volatility, molecular weight, presence of isomers, and impurities present. Settings used for a study should be documented in the raw data and the final report as appropriate.

All calibration standards, CCVs, blanks, study samples, and control samples must be analyzed using the same settings. If changes are made after analysis has been initiated, recalibration is necessary. The following analytical conditions and settings may be used as a guide.

Example Purge and Trap Settings

Set the Water Mode or the Soil Mode of operation in the software

Purge Flow Parameters: 40 mL/min of helium for 11 minutes

Dry Purge: 40 mL/min for 1 to 4 minutes

Line and Valve Temperatures: 105 ºC to 150 ºC

Analytical Trap Desorb Parameters: 0.5 to 1 minutes at 250 ºC to 260 ºC

Analytical Trap and Condensation Trap Bake: 8 to 10 minutes

Set sample loop fill times, transfer times, rinse times, equilibration times as appropriate

Example GC/MS Parameters

Analytical Column: J & W DB-624, 0.25mm, 30m, 1.4µm df; column flow 1.1 mL/min

Column split flow: set between 20:1 and 50:1

Example GC temperature program profile: 30 ºC for 3 minutes, ramp 20 ºC / min to 230 ºC, and hold for 5 minutes for an 18 minute run time. (Sub-ambient starting temperatures require the use of a cryogen, such as liquid nitrogen.)

MS Quadrupole Temp: 150 ºC

MS source Temperature: 230 ºC

Multiplier Voltage: Typically set by the software as the result of an auto tune. This may be increased to meet sensitivity requirements, especially in the SIM mode. The addition of 100 to 400 electron volts, EV, to the set point is common.

Full Scan Mode: 35 to 270 atomic mass units (amu). The high end of the range may be changed to bracket the molecular weight of the target compound(s). Typically, the scan range does not exceed 500 amu for volatile target analytes.

Selected Ion Mode (SIM): Dwell times for appropriate ions are 35 to 100 msec so that there are a minimum of 10 to 15 scans across each chromatographic peak of interest.

12 Data Analysis and Calculations

Each dataset should be processed using chromatography software (e.g. Target Falcon integrator, Agilent Chemstation, Agilent MassHunter, Applied Biosystems Analyst). Integration parameters should be set to minimize the number of manual integrations required yet still result in uniform integration of peaks at all concentration levels. It is acceptable to perform manual integrations for any or all standards, samples, or blanks if appropriate integration parameters cannot be found, especially when near baseline resolved isomers are present. All integrations must be done consistently for all standards, samples, and blanks and in compliance with ETS-12-010.

Means will be calculated by adding the individual entities and dividing the resultant sum by the number of individual entities.

Standard deviations will be calculated using Microsoft Excel ®. The Microsoft® built-in function STDEV is typically used.

Sample precision will be reported as % relative standard deviation (%RSD) or as % coefficient of variation (%CV) for triplicates or above, and relative percent difference (RPD) for duplicate data. Sample %RSD will be calculated using the following equation:

$$Sample\ \%RSD = \left(\frac{A}{B}\right) * 100$$

Where: A = standard deviation of averaged samples and B = average of samples

The RPD is defined as the absolute value of the difference of two values divided by the average of the two values and multiplied by 100. If data quality objectives for are not met, the Dixon's Q-Test described in the next section may be used for identification and rejection of outliers.

Dixon's Q-Test:

A data point may be excluded if "$Q_{observed}$" is greater than "$Q_{tabulated}$" with 95% confidence.

$$Q_{observed} = \frac{\text{gap}}{\text{range}}$$

where "gap" is the difference between the questionable data point and the closest value of the data set, and "range" is the difference between the highest and lowest value of the data set. The following table provides $Q_{tabulated}$ values to compare $Q_{observed}$. If Confidence Levels of greater than 95% are calculated, the "Q" Values from the same source will be used and documented in the raw data and the report.

Table 2. Dixon Q Test Values for $Q_{tabulated}$.

[(1)]$Q_{tabulated}$ Values:	
Number of Observations:	**Confidence Level= 95% Percentile**
3	0.970
4	0.829
5	0.710
6	0.625
7	0.568
8	0.526
9	0.493

(1) David B. Rorabacher, Statistical Treatment for Rejection of Deviant Values: Critical Values of Dixon's "Q" Parameter and Related Subrange Ratios at the 95% Confidence Level, Anal. Chem. 1991, 63, 139-146.

If an outlier value is rejected, exclude the sample from further data reporting and calculate the average and %RSD or RPD for the remaining values. If no data points can be excluded and the data does not meet criteria, analysis may be repeated or the data used, but outliers flagged and impact on data quality discussed in the report. Document the non-compliant data within the final results.

Calculate any matrix spike percent recoveries using the following equation:

$$\%\ Recovery = \frac{(observed\ concentration - unfortified\ sample\ concentration)}{spike\ concentration} * 100\%$$

13 Pollution Prevention and Waste Management

Dispose of sample vials in low BTU and flammable solvent in high BTU containers.

Dispose of glass pipette waste in broken glass containers located in the laboratory.

Follow 3M policies for all sample handling and disposal. Zero headspace aqueous samples that have an aliquot removed generally lose sample integrity for reanalysis after one hour. Sample vials with an aliquot removed may be disposed of at the discretion of the analyst. Alternatively, the remaining aqueous sample may be transferred to a smaller zero-head space vial for future analysis within one hour of opening the original vial.

14 Records

Analytical records needed to reconstruct the sample preparation, sample analysis, and to reconstruct the results calculations must be included in the final data package or kept as facility records. The required documents may include, but are not limited to, the following handwritten or electronic records:

Standard Preparation Log Book Records

Sample Preparation Worksheet(s)

Purge and Trap Equipment Settings

Gas Chromatograph and Mass Spectrometer Identification and Settings

Analytical Sequence(s)

Initial Calibration Results Summary

Chromatograms

Quantitation Reports for Analyses

Notes to File

15 Attachments

None

16 References

16.1 EPA Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, August 2006

16.2 EPA Method 5030C, Purge and Trap for Aqueous Samples, Revision 3, May 2003

16.3 ETS-8-196 titled “Gas Standard Preparation”

16.4 ETS-12-001 titled “Analytical Definitions, Abbreviations, Acronyms, and Symbols”

17 Affected Documents

None

18 Revisions

Revision Number	Summary of Changes
1	Extensive revisions made to allow the method to be used more universally throughout the lab
2	Extensive revisions made to reflect that this method is compliant with EPA Method 8260, update sampling equipment, sample handling procedures, and the expanded scope and use of this method for the analysis of physical and chemical properties of chemicals.