

VOCs in Vapor by 8260B AZ Method

04/14/2009

Revision - 0.0

Analyte: Volatile Organic Compounds

Method No: 8260B AZ Vapor Method

Matrix: Vapor

Minimum Reporting Limits:

See Appendix A

Procedure: Vapor Samples are collected in appropriate containers and analyzed by GC/MS. After the initial introduction of vapor sample to 5 ml of water in a sparging vessel, the subsequent steps performed are as per EPA SW846 methods 5030C and 8260B.

VOCS in Vapor by 8260B AZ Method

04/14/2009

1. METHOD REFERENCE

- 1.1 EPA Method 8260B, Nonhalogenated Organics Using GC/MS, SW-846 Update III, December 1996.
- 1.2 EPA Method 8000C, Gas Chromatography, SW846 Revision III, March 2003.
- 1.3 EPA Method 5030C, Purge-and-Trap for Aqueous Samples, SW846 Revision III May 2003.
- 1.4 Compendium Method TO-15 Determination of Volatile Organic Compounds in Air Selected in Specially Prepared Canisters and Analyzed by Gas Chromatography / Mass Spectrometry Second Edition January 1999
- 1.5 Method 0040 – Sampling of principal hazardous organic constituents from collection sources using Tedlar Bags, SW-846 Revision 0 December 1996

2. DEFINITIONS

- 2.1 Vapor – The gas given off by substances that are solids or liquids at ordinary atmospheric temperature and pressure.
- 2.2 Soil Vapor - is the air found in the pore spaces between soil particles.
- 2.3 Batch – A group of 20 samples or less processed in the same 24 hour period.
- 2.4 LCS - Laboratory Control Sample defined as laboratory grade water fortified with a known concentration of secondary source standard, and analyzed with each batch of samples.
- 2.5 CCV - Continuing Calibration Verification defined as a purged aqueous mid-level primary source standard.
- 2.6 Tertiary Standard – is a mid-level third source vapor standard different from calibration and secondary standard analyzed immediately following the calibration curve used to verify the recovery of the vapor standard compared to the purged

aqueous standard. This standard containing partial or complete list of analytes is highly recommended.

3.0 SYNOPSIS

- 3.1 This method is an Arizona consensus method and was developed with collaboration among the Arizona Department of Health Services (ADHS), Arizona Department of Environmental Quality (ADEQ) and Environmental Laboratory Advisory Committee (ELAC). The method was initially written to complement the Soil Gas Guidance from ADEQ and is intended for testing soil vapor/soil gas but can be used for other vapors as long as the data quality objectives can be met with program approval. Results should be reported in mg/m³.
- 3.2 The use of this method is restricted to appropriately trained and experienced laboratory analysts who are familiar with analyses of volatile organics analyses by GCMS. Each analyst must demonstrate the ability to generate acceptable results with this method.
- 3.3 This method will be revised as needed as more experience is gained in the use of this method.
- 3.3 Subcommittee members of the Environmental Laboratory Advisory Committee (ELAC) formed a volunteer group of members and interested parties to compose a Standard Operating Procedure for an 8260B AZ Vapor method. A preliminary draft of the method was presented to the members of ELAC and other stakeholders. The document will be revised as needed based on comments and suggestions received from its reviewers and users.

The principal authors of this procedure are:

Julie Hoskin, Arizona Department of Environmental Quality
Prabha Acharya, Arizona Department of Health Services
Elizabeth Wueschner, TestAmerica Laboratories, Inc
Mary Tyer, Columbia Analytical Services, Inc.
Michael Tuday, Columbia Analytical Services, Inc.
Val Mallari, AirTech Lab
Tracy Dutton, TestAmerica Laboratories, Inc.
J.D. Gentry, Environmental Science Corp.
Danny Ramsey, Environmental Science Corp.
Dave Veratti, Environmental Science Corp

4.0. RANGES, SENSITIVITY, REPORTING LIMIT

- 4.1 This method is used for the detection of VOCs in vapor samples.

4.2 Minimum Reporting Limits – See Appendix A

5.0 INTERFERENCES

- 5.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.
- 5.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.
- 5.3 For samples containing high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis highly recommended to prevent contamination of the system.
- 5.4 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

- 5.5 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

6. SAMPLE HANDLING AND PRESERVATION

- 6.1 Vapor Samples should be collected in one of the following containers dependent on the program requirement. Hold Times vary depending on the collection vessel.

Tedlar Bag – When choosing a tedlar bag for sampling the vapor, the bag should have well constructed seams, be leak and contaminate free, and must have a minimum of 2-mm tedlar film. Tedlars with septa are optimal for ease of aliquot removal by gas-tight syringe. Hold Time 72 hours

Air Tight Syringes – (Luer Lock) – Glass and non disposable syringes should be utilized. Hold Time 2 hours.

Stainless Steel Canisters – Cleaned and evacuated canisters with the manufacturer's serial number, or a unique permanent identification number should be utilized. Hold Time 30 days

- 6.2 Sample tedlar bag storage and transport procedures - To ensure sampling integrity, perform sample recovery in a manner that prevents contamination of the bag sample. Protect the bag from sharp objects, direct sunlight and low ambient temperatures (below 0°C [32°F]) that could cause condensation of any of the analytes. Store the bags in an area that has restricted access to prevent damage to or tampering with the sample before analysis. Analyze the bag samples within 72 hours of sample collection unless it can be shown that significant (> 20%) sample degradation does not occur over a longer period of sample storage. Upon completion of the testing and sample recovery, check all the data forms for completeness and the sample bags for proper identification. Store the bags in rigid, opaque containers during all sampling, storage and transport procedures. Ship the bags using ground transportation. Follow all hazardous materials shipping procedures.
- 6.3 Tedlar Bags should only be filled to 70-75% capacity to avoid compromising the integrity of the bag.

7. APPARATUS AND MATERIALS

- 7.1 Purge-and-trap device for aqueous samples - Described in Method 5030C.
- 7.2 Injection port liners (HP Catalog #18740-80200, or equivalent) - modified for direct injection analysis by placing a 1-cm plug of glass wool approximately 50-60 mm down the length of the injection port towards the oven. A 0.53-mm ID column is

mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

7.3 Gas chromatography/mass spectrometer/data system

7.3.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

7.3.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

7.3.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

7.3.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

7.3.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

7.3.2 Gas chromatographic columns

7.3.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5-µm film thickness, or equivalent.

7.3.2.2 Column 2 - 30 - 75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt -502.2 (RESTEK), or VOCOL (Supelco), 3-µm film thickness, or equivalent.

7.3.2.2 Column 3 - 30 m x 0.25 - 0.32 mm ID capillary column coated with 95% dimethyl - 5% diphenyl polysiloxane (DB-5, Rt -5, SPB-5, or equivalent), 1-µm film thickness

7.3.2.3 Column 4 - 60 m x 0.32 mm ID capillary column coated with DB-624(J&W Scientific), 1.8-µm film thickness, or equivalent.

7.3.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact

ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC. An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB which meets all of the criteria in Table 3 when 5 or 50 ng is introduced.

7.3.4 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

7.3.4.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25 - 0.32 mm ID columns.

7.3.4.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

7.3.4.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

7.3.5 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

7.4 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000- μ L.

- 7.5 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.
- 7.6 Syringes - 5-, 10-, or 25-mL, gas-tight with shut-off valve.
- 7.7 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.
- 7.8 1ml or 2 ml vials with mininert caps or equivalent.
- 7.9 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

8. REAGENTS AND STANDARDS

- 8.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One of SW-846 Section 5.0.
- 8.2 Methanol, CH₃OH - Pesticide quality or equivalent, demonstrated to be free of analytes, stored away from other solvents.
- 8.3 Primary Standards - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare primary standard solutions in methanol. For more information on preparing stock solutions see Method 8260B Section 8.7.

8.3.1 Frequency of Standard Preparation

Standards should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as styrene may need to be prepared more frequently.

- 8.4 Secondary standard is a methanol standard that contains the reference materials of interest and is a separate source from the primary standard.
- 8.5 Preparation of Tertiary Standard from a Gas Mixture: The procedure involves using a certified gaseous mixture after each calibration, utilizing a commercially-available or in-house gaseous analyte mixture. The shelf life is determined as per manufacturer specifications as per Standard Operating Procedure.

8.5.1 Before removing the cylinder shipping cap, be sure the valve is completely

closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

8.5.1.1 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

8.5.1.2 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect a Luer syringe of the appropriate size to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

8.5.1.3 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 8.5.1.2.(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

8.5.1.4 The pressure in the cylinder is ~30 psi.

8.5.1.5 Remove an appropriate volume of the gas mixture at a concentration of the mid level of the calibration curve and quickly transfer into the reagent water through the female Luer fitting located above the purging vessel. NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

8.6 Surrogate standards - The recommended surrogates are toluene-d8, 4-bromofluorobenzene, 1,2-dichloroethane-d , and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. Select or prepare a surrogate solution at the appropriate concentration level.

8.7 Internal standards - The recommended internal standards are fluorobenzene,

chlorobenzene-d₄, and 1,4-dichlorobenzene-d₄. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Select or prepare an internal standard solution at the appropriate concentration level. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

- 8.8 Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/μL of BFB in methanol should be prepared.
- 8.9 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

9. PROCEDURE

- 9.1. Introduction of Aqueous Standards – Please refer to SW 846 Method 5030C

- 9.2 Sample Analysis

- 9.2.1 Removing aliquot from sampling container

- a) To transfer the sample from a Tedlar Bag, remove a maximum aliquot of 10mL utilizing a 10mL Luer syringe fitted with a needle. Rinse the syringe with the sample several times before removing an aliquot.

- b) For canisters with 1/4 valves (nupro style), a 1/4 stainless steel nut, 7/16 GC inlet septa, and gas-tight syringes ranging in sizes from 10ul to 10ml are required. Place the septa in the 1/4 ss nut so that it sits flush on the inside of the nut. Once the septum is flush, gently attach the nut to the canister valve. Over tightening the nut will cause it to collapse and will result in a leak. Once the 1/4 nut with septa is attached to the canister and the septa has been visually inspected to insure it has not collapsed, the canister valve can be opened and a sample aliquot can be removed by gas tight syringe. Sample aliquot volumes can vary as needed for sample concentrations of target compounds present.

- c) For canisters with quick connect valves, a 1/4 stainless steel nut and septum can be connected directly to the quick connect body fitting

- 9.2.2 Introduction of samples to instrument - Remove an appropriate volume of the vapor sample and quickly transfer into the reagent water through the female Luer fitting located above the purging vessel. NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

9.3 Analytical Procedure

9.3.1 Analysis is performed by GC/MS as per 8260B method.

10. CALIBRATION

Calibration standards -There are three sources of calibration standards used for this method: primary source, secondary source and tertiary vapor standard. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The calibration standards must also contain the internal standards and surrogates chosen for the analysis.

10.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the dilution of stock standards or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to the Limit of Quantitation (LOQ) / Method Reporting Limit (MRL) or lower.

10.2 Verify the calibration curve with an aqueous second source standard. The acceptance limits for this check standard are 80-120%. Some of the difficult compounds may not fall within these acceptance limits (i.e. Ketones and Gases). In-house limits can be generated once enough data has been collected.

10.3 A tertiary vapor standard is used to verify the recovery of the vapor standard compared to the aqueous standard. The acceptance limits are initially set at 50-150% and can later generate in-house limits once enough data is collected. Following is a suggested procedure for analyzing this standard:

10.3.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

10.3.1.1 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

10.3.1.2 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect a Luer syringe of the appropriate size to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

10.3.1.3 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 10.2.1.2.(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

10.3.1.4 The pressure in the cylinder is ~30 psi.

10.3.1.5 Remove an appropriate volume of the gas mixture at a concentration of the mid concentration of the calibration curve and quickly transfer into the reagent water through the female Luer fitting located above the purging vessel. NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

10.4 Either a five point calibration curve or a constant amount of surrogate standards can be added to the calibration standards to quantitate the surrogate recoveries.

11. CALCULATION

11.1 For the calculations applicable to this method refer to Instrument Calibration Training CD presented by the Arizona Department of Health Services (January 2007).

11.2 $C_f = C_i \times PF \times DF$

C_f = Final concentration in ug/L

C_i = Concentration in ug/L from the instrument

PF = Preparation Factor

DF = Dilution Factor

Example:

If 10mL of vapor sample is introduced using a 5mL purged aqueous calibration curve then the preparation factor would be 0.5.

11.3 Report as mg/m³

$$1 \text{ ug/L} = 1 \text{ mg/m}^3$$

$$1 \text{ ml} = (1\text{cm})^3 = 10^{-3} \text{ L}$$

$$1 \text{ cm} = 10^{-2} \text{ m}$$

$$\text{Therefore, } 10^{-3} \text{ L} = (10^{-2} \text{ m})^3 = 10^{-6} \text{ m}^3$$

Dividing each side of the equation by 10^{-3} results in $1 \text{ L} = 10^{-3} \text{ m}^3$

Substituting for $1 \text{ ug} / 1 \text{ L}$ is then $10^{-3} \text{ mg} / 10^{-3} \text{ m}^3$, or $1 \text{ mg} / 1 \text{ m}^3$

12. QUALITY CONTROL

12.1 Hold Times vary depending on the collection vessel:

Tedlar Bag – Hold Time 72 hours

Air Tight Syringes – (Luer Lock) – Hold Time 2 hours

Stainless Steel Canisters – Hold Time 30 days

12.2 The GC/MS system must be hardware tuned to meet the criteria with 5-50ng injection of purging bromofluorobenzene. Analysis must not begin until this criteria is met. The BFB tune must be analyzed at the beginning of each day and every twelve hours. All standards and samples following the tune must be analyzed under identical mass spec instrument conditions.

12.3 Minimum of one aqueous method blank must be analyzed for every batch up to 20 samples or less.

12.4 Minimum of one LCS (prepared in water) must be analyzed for every batch up to 20 samples or less. The spiking solutions must be derived from a secondary source different from the calibration curve standard (See Section 8). Acceptance range for LCS recovery is between 70% and 130% of true value. Some of the difficult compounds may not fall within these acceptance limits (i.e. Ketones and Gases). In-house limits can be generated once enough data has been collected or if limits have already been established by the testing laboratory.

12.5 Analysis of a LCSD is recommended in order to be compliant with ADEQ Policy 154. Contact ADEQ QA for a copy of the policy.

12.6 A minimum of one Sample / Sample Duplicate pair must be analyzed for every

batch up to 20 samples or less. Calculate precision (RPD) as per section 8.5.3.2 of EPA method 8000C. Default ADHS limits of 20% or in-house limits can be used as acceptance limits.

- 12.7 Acceptance range for surrogate recovery is between 70% and 130% of true value.
- 12.8 The relative standard deviation (RSD) for each compound must be less than or equal to 15%. If the RSD of any target analyte is greater than 15%, additional calibration options given in Method 8000C can be used. All calibration models used must be documented in the laboratory's standard operating procedure.
- 12.9 Verify the calibration curve with an aqueous second source standard. The acceptance limits for this check standard are 80-120%. Some of the difficult compounds may not fall within these acceptance limits (i.e. Ketones and Gases). In-house limits can be generated once enough data has been generated.
- 12.10 Calibration verification standards (CCV) should be prepared at a concentration near the mid-point of the initial calibration range from a primary source standard. Prepare these solutions in organic-free reagent water. All target analytes must be included in the calibration verification standard(s). The calibration standards must also contain the internal standards and surrogates chosen for the analysis. The CCV must be analyzed at the beginning of each day and every twelve hours. The CCV and BFB tune may be combined into one standard. The acceptance limits for this check standard are 80-120%. Some of the difficult compounds may not fall within these acceptance limits (i.e. Ketones and Gases). In-house limits can be generated once enough data has been generated.
- 12.11 Method Detection Limits must be established and verified for every compound of interest. Method Detection Limits may be determined according to the procedure in 40 CFR Part 136 Appendix B. This criteria may be revised at a later date depending on the EPA's decisions on the determination of reporting limits / detection limits.
- 12.12 When developing this method an initial demonstration of capability must be performed. This consists of four analyses using a mid-level tertiary standard that is compared to the accuracy and precision data for the aqueous standard. Acceptance limits, at a minimum must meet requirements for the tertiary standard.

APPENDIX A

Standardized 8260B AZ Vapor Analyte List and Minimum Report Limits

Analyte	ug/L or mg/m³
Acetone	20
Benzene	5
Bromobenzene	5
Bromochloromethane	5
Bromodichloromethane	5
Bromoform	10
Bromomethane	10
2-Butanone (MEK)	20
n-Butylbenzene	10
sec-Butylbenzene	10
tert-Butylbenzene	10
Carbon tetrachloride	5
Chlorobenzene	5
Chlorodibromomethane	5
Chloroethane	10
Chloroform	5
Chloromethane	10
2-Chlorotoluene	10
4-Chlorotoluene	10
1,2- Dibromoethane (EDB)	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
Dichlorodifluoromethane	10
1,1-Dichloroethane	5
1,2-Dichloroethane (1,2-DCA)	5
1,1-Dichloroethene	5
cis-1,2-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
1,3-Dichloropropane	5
2,2-Dichloropropane	5
1,1-Dichloropropene	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5

Ethylbenzene	5
2-Hexanone	20
4-Isopropyl toluene	10
Methylene chloride	10
4-Methyl-2-pentanone (MIBK)	20
Methyl-tert-butyl ether (MTBE)	5
n-Propylbenzene	10
Styrene	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	10
1,2,3-Trichlorobenzene	10
1,2,3-Trichloropropane	10
1,2,4-Trimethylbenzene	10
1,3,5-Trimethylbenzene	10
Vinyl acetate	20
Vinyl chloride	5
Xylenes, Total	15