STANDARD OPERATING PROCEDURE APPROVAL AND CHANGE FORM

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STANDARD OPERATING PROCEDURE	
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Authors	
Name Deborah Killeen Title SERAS QA/QC Officer Signature	Date 6/16/14, 6/27/14
Name Title Signature	Date
Approvals	
Name Amy Dubois Title Air Environmental Scientist Signature	Date 6/16/14
Name Jay Patel Title Analytical Support Leader Signature Jay Patel	Date 06/16/14
Name Dennis Miller Title Acting Program Manager Signature Dennis Miller	Date 6/16/14

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History	Effective Date
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Fixed formatting issues and defined all acronyms at first use	
Added operating conditions for DFTPP Tune in can mode in Table 4, Appendix A	
Added wrap tubes in aluminum foil to section 3.1	
Added set-up of field blank to Section 7.1	
Corrected spike concentrations in Sections 7.2.6, 8.3 and 9.8	
Added field blank into EICP area internal standard check requirement	
Added qualification of samples based on method blank to Section 9.6	
Added qualification of samples based on field blank to Section 9.9	

STANDARD OPERATING PROCEDURES

AIR SAMPLING AND ANALYSIS FOR 4-METHYLCYCLOHEXANE METHANOL BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

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AIR SAMPLING AND ANALYSIS FOR 4-METHYLCYCLOHEXANE METHANOL BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

1.0 SCOPE AND APPLICATION

The objective of this Standard Operating Procedure (SOP) is to outline the steps for the collection, extraction and analysis of 4-methylcyclohexane methanol (4-MCHM) in air samples collected on XAD-2 sorbent tubes using gas chromatography/mass spectrometry (GC/MS) in the selective ion monitoring (SIM) mode. This method can be used to quantitate 4-MCHM that is soluble in methylene chloride and capable of being separated into cis and trans isomers on a fused-silica capillary column. 4-MCHM exists as two diastereomers with similar properties. Table 1, Appendix A lists the method detection limits (MDLs) and reporting limits (RLs) for this compound based on the volume collected.

This method may not be changed without the expressed approval of the Analytical Support Leader and the Quality Assurance/Quality Control (QA/QC) Officer. Only those versions issued through the Scientific, Engineering, Response and Analytical Services (SERAS) document control system may be used. Modifications made to the procedure due to interferences in the samples or for any other reason must be documented in the case narrative and on a nonconformance memo.

2.0 METHOD SUMMARY

Air samples are collected on two-stage XAD-2 resin tubes and extracted with 2 milliliters (mL) methylene chloride. Prior to GC/MS analysis in the SIM mode, a 1-mL aliquot of the extract is spiked with an internal standard. 4-MCHM is identified by comparing the measured mass spectra and retention times with those obtained from calibration standards acquired under the same operating conditions used for the samples. Quantitation of this target analyte is calculated based on the internal standard method. Table 2, Appendix A lists the characteristic ions of the target analyte and the internal standard.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

The XAD-2 resin used for sampling is housed in an 8-millimeter (mm) outer diameter by 110 mm length glass tube that has been flame sealed and contains 600 milligrams (mg) of XAD resin. The XAD in the tubes is packed in two sections: 400 mg and 200 mg in the front and back sections, respectively. The sections are separated by a plug of glass wool. The maximum loading for the tube should not exceed 200 micrograms (µg) of 4-MCHM to avoid breakthrough.

3.1 Sample Storage

To preserve and store air samples collected using XAD-2 tubes, plastic caps are placed on the XAD-2 tube ends and the tube wrapped in aluminum foil. The samples are then placed into a Whirl-Pak bag. Samples must be protected from light and refrigerated at less than or equal to (≤) 6 degrees Celsius (°C) from the time of collection until extraction and analysis.

3.2 Holding Times

The recommended maximum holding time is 14 days from date of collection; however, there is no available information regarding the stability of this compound on sorbent tubes.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

High humidity and temperature, and high sampling flow rates may decrease the adsorption capacity of the resin. Contaminants may migrate from the front portion to the back portion of the tube.

Media (lot) blanks should be submitted and analyzed for each lot of XAD-2 tubes used. A field blank must be submitted for each day of sampling. Each lot of methylene chloride used for sample extraction should be screened by GC/MS prior to use.

5.0 EQUIPMENT/APPARATUS

- Syringes, various µL volumes, for spiking and preparation of standards
- Micro syringes, 10 µL and larger, 0.006 inch inner diameter (ID) needle
- XAD-2 resin sorbent tubes: 400/200 mg two-stage (SKC, Inc. Catalog No. 226-30-06, or equivalent)
- Air sampling pumps, pump/tube-holder combination capable of collecting 0.1 to 0.25 liters per minute (L/min) through the sorbent tube up to eight hours
- Rotameter/electronic flow meter, measure sampling flow rate
- Aluminum foil, protect sorbent tubes from ultraviolet (UV) radiation during sampling and shipment
- Vials, 2-mL for GC autosampler
- Desorption vials, 4- mL, screw cap with Teflon® cap liner (Supelco Cat #2-3178 or equivalent)
- Vortex (Thermolyne Maxi-Mix II, or equivalent) or ultrasonic bath.
- Agilent Technologies 7890 GC and 5975 mass selective detector (MSD), or equivalent, equipped with an autosampler and controlled by HP Enviroquant (or equivalent) software.
- Zebron, ZB-5 fused silica capillary column, 30 meter (m) x 0.25 mm ID, 0.5 micron (µm) film thickness (or equivalent)

6.0 REAGENTS

- Methylene Chloride (DCM), pesticide residue analysis grade or equivalent, each lot analyzed prior to use.
- Stock Internal Standard 2-Fluorobiphenyl, 96% neat standard
- Intermediate Internal Standard Solution, 10,000 micrograms per milliliter (µg/mL) – Add 100 µL of the neat 2-fluorobiphenyl internal standard to 10 mL of DCM.

- Intermediate Internal Standard Solution, 2000 µg/mL – Add 200 µL of the 10,000 µg/mL intermediate internal standard solution to 1.0 mL of DCM.

- Working Internal Standard Solution, 25 µg/mL – Add 125 µL of the 2000 µg/mL intermediate internal standard solution to 10 mL of DCM. Twenty µL of the internal standard (IS) solution is added to each 1 mL of the sample extract before analysis, resulting in a 500 micrograms per liter (µg/L) injection.

- Primary Stock 4-MCHM Calibration Standard, neat, accompanied by a certificate of analysis

- Primary Intermediate 4-MCHM Calibration Standard, 10,000 µg/mL – Add 0.1 grams (g) of the neat 4-MCHM stock calibration standard to 10 mL of DCM.

- Primary Intermediate 4-MCHM Calibration Standard, 10 µg/mL – Add 10 µL of the 10,000 µg/mL 4-MCHM calibration standard to 10 mL of DCM.

- Primary 4-MCHM Calibration Working Standards, 50, 100, 200, 500 and 1000 µg/L – Prepare as follows in DCM:

Concentration, µg/L	µL of 10 µg/mL Intermediate Std	µL of 25 µg/mL Internal Std (added to 1 mL of extract)	Final Volume (µL)
50	50	20	10000
100	100	20	10000
200	200	20	10000
500	500	20	10000
1000	1000	20	10000

- Stock Decafluorotriphenylphosphine (DFTPP) Mix, 1000 µg/mL, consisting of pentachlorophenol, DFTPP, benzidine and 4,4'-dichlorodiphenyltrichloroethane (DDT) each at 1000 µg/mL

- DFTPP Tune Solution, 50 µg/mL – Dilute 500 µL of the Stock 1000 µg/mL DFTPP to 10 mL of 65:35 v/v DCM/hexane. Add 200 µL of the stock 2,000 µg/mL internal standard mix. The amount of DFTPP in a 1-µL injection is 50 nanograms (ng).

- Secondary 4-MCHM Stock Standard, neat, accompanied by a certificate of analysis

- Secondary 4-MCHM Intermediate Standard, 10,000 µg/mL – Add 0.1 g of the neat 4-MCHM standard to 10 mL of DCM.

- Secondary 4-MCHM Working Standard, 10 µg/mL – Add 10 µL of the secondary 4-MCHM 10,000 µg/mL intermediate standard to 10 mL of DCM.

- Initial Calibration Verification Standard, 200 µg/L – Add 200 µL of the Secondary 10 µg/mL 4-MCHM intermediate standard to 10 mL of DCM.

- Helium, ultra high purity grade for GC/MS system

NOTE: All of the above mentioned standard solutions must be stored at ≤6°C in a dedicated refrigerator/freezer in tightly capped amber vials with Teflon liners. Commercially prepared standard solutions that are received in sealed ampules may be stored in the shelf section of the standards refrigerator.

NOTE: Premixed certified standards will be stored according to the manufacturer's documented storage requirements. These standards may be kept in storage up to the manufacturer's stated expiration date. Once the standard vials are opened, the standards will be stored with minimal headspace in the freezer (-10°C) for a period not to exceed six months or the manufacturer's expiration date, whichever is less.

NOTE: The IS mixture should be stored in the refrigerator at ≤6°C.

NOTE: All calibration standards, surrogates, internal standards, and spiking solutions will be prepared and documented in accordance with SERAS SOP #1012, *Preparation of Standard Solutions and Reagents.*

7.0 PROCEDURES

7.1 Sample Collection

1. Calibrate each personal sampling pump with the sorbent tube in line, use of single manifold or critical orifice may be needed to achieve flow rates. This sorbent tube may be used as the field blank or the ends of a XAD-2 tube may be broken prior to the set-up of the first sample.

2. Collect samples at 0.1 to 0.25 L/min for up to eight hours, use an opaque tube holder or wrap with aluminum foil to prevent sunlight from causing sample degradation. Record initial flow rate.

3. Upon sample completion, measure and record final flow rate and elapsed time. Calculate and record average flow rate. Use average flow rate and elapsed sample time to determine sample volume.

4. Cap the ends of all sorbent tubes used with plastic caps immediately after sampling; never use rubber caps or tape. Wrap each tube in foil and deposit in sample-specific re-sealable plastic bag. (Whirl-Pak). Label bag with sample number, date and time, and sample volume information.

5. Ship all samples to laboratory under chain of custody using an insulated sample container and bagged refrigerant.

6. Refer to SERAS SOP #2008, *General Air Sampling Guidelines* and SOP #2066, *XAD-2 Sampling in Ambient Air.*

7.2 Preparation and Extraction

1. Allow the XAD tubes to warm up to ambient temperature prior to extraction.

2. The front and back sections of the XAD-2 resin are routinely analyzed separately. For each sorbent tube, remove the glass wool plug from the back portion of the tube and discard. Remove the XAD-2 resin packing from the back of the tube and place it in a 4-mL screw-top vial. Label this vial "back" along with the sample identifier. Remove the glass wool that supports the XAD resin in the front portion of the tube and discard. Place the front XAD packing in a second sample vial. Label this vial "front" along with the sample identifier.

3. Add 2 mL of methylene chloride to each vial and screw the caps on tightly.

4. Vortex the vials containing the XAD sorbent for 2 minutes. Let the vials settle for 30 minutes.

5. Transfer 1.0 mL of the extracts from the vial containing the XAD resin to 2-mL autosampler vials.

6. Using two sorbent tubes from the same lot as the tubes used during sample collection, prepare the blank spike/blank spike duplicate (BS/BSD). Break the glass in the front portion of both tubes and transfer the resin from the front section into two separate vials. Add 40 µL of the 10 µg/mL secondary working standard directly onto the resin in the vial (equivalent to 200 ng of each isomer). Allow to stand. Add 1.9 mL of methylene chloride, cap the vials and vortex the vials for 2 minutes. Allow to stand for 30 minutes.

7. Prepare media blanks using three sorbent tubes from the same lot and adding 2 mL of methylene chloride to each. Cap the vial, vortex for 2 minutes and allow the vials to stand for 30 minutes.

8. Extract other QC samples (i.e., field blank) using steps 1 to 5 above.

7.3 GC/MS Operating Conditions

The following GC/MS operating conditions are recommended for the Agilent Technologies 6890/7890 GC and 5973/5975 MS detector system (refer to Table 3, Appendix A for complete listing):

Column	Zebron ZB-5 (5% phenyl - 95% dimethyl-polysiloxane) 30 meter x 0.25 mm ID, 0.50 µm film
Flow rate	1.5 mL/min., EPC enabled
Injection Temperature	270°C
Transfer Line/Aux Temp.	325°C
Source Temperature	280°C
Quad Temperature	190°C
GC Oven Temp. Program	50°C, hold 1.0 minute, ramp at 17.5 °C/min to 200 °C, hold 0.5 min, ramp at 35 °C/min to 315 °C, hold for 2 minutes
Pulsed Split Injection	Split ratio = 12:1, split flow = 18 mL/min, pulse = 22 psi for 0.5 min

	Gas Saver 35 mL/min. at 0.75 min
Injection Volume	2µL
	Use 4mm ID, single gooseneck liner packed with silanized glass wool

SIM Parameters Group 1
Resolution: Low
Plot 1 Ion: 54

Ions/Dwell in Group	(Mass, Dwell)	(Mass, Dwell)	(Mass, Dwell)
	(54.00 50)	(55.00 50)	(82.00 50)
	(95.00 50)	(97.00 50)	(128.00 50)
	(171.00 50)	(172.00 50)	

7.4 Decafluorotriphenylphosphine Tune

The recommended GC/MS tune parameters are listed in Table 4, Appendix A. The MS must be tuned with perfluorotributylamine (PFTBA) in the scan mode so that a 50 ng injection of DFTPP produces spectra that will meet the ion abundance criteria listed in Table 5, Appendix A. The tune is acquired by averaging the apex and ± one scan. Background subtraction should be done using a single scan no more than 20 scans prior to the elution of the DFTPP and should be used to eliminate column bleed or instrument background noise. The DFTPP tune criteria must be met every 12 hours during sample analysis. If the software does not indicate what scan was subtracted, the analyst will document the scan number directly on the tune report. The tuning standard should also contain 50ng/µL of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Degradation of DDT to dichlorodiphenyldichloroethylene (DDE) and dichlorodiphenyldichloroethane (DDD) is measured and should not exceed 20%. Benzidine and Pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

$$\text{Tailing Factor} = BC/AB$$

Where:
BC = the right side half of the peak width, measured from 10% up the baseline of the peak
AB = the left side half of the peak width, measured from 10% up the baseline of the peak

Figure 1, Appendix B illustrates how to determine the tailing factor.

Note: The Enviroquant software is capable of automatically calculating the degradation and tailing factors and should be used whenever possible.

7.5 Initial Calibration

1. Add 20 µL of the internal standard mix to each 1-mL aliquot of the six initial calibration (ICAL) standards. Do not add the internal standard if using commercially prepared calibration standards that already contain the internal standards.

2. After DFTPP passes the criteria, set up the run using the six calibration standards.
NOTE: A minimum of 5 standards must be used to generate the initial calibration curve.

3. Calculate and tabulate the relative response factor (RRF) against the concentration for each compound, including the surrogates, by using the equation below. The primary ion from the specific internal standard must be used for quantitation. The average RRF and percent relative standard deviation (%RSD) must also be calculated and tabulated.

4. The %RSD must be less than 20% for all compounds or a minimum correlation coefficient of 0.99 using linear regression. Since the method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, the recalculated concentration of the low calibration point should be within plus or minus (±) 30% of the standard's true concentration.

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where:

A_X	=	Area of the characteristic ion of each target analyte
A_{IS}	=	Area of the characteristic ion of each internal standard assigned to target analytes
C_{IS}	=	Concentration of each internal standard, nanograms per microliter (ng/µL)
C_X	=	Concentration of each target analyte (ng/µL)

5. Use the following equations to calculate and tabulate average RRF and %RSD for 4-MCHM.

$$\overline{RRF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

where:

$\overline{RRF}$ = relative response factor for each initial calibration level
N = total number of initial calibration levels

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RRF_i - RRF_{average}\right)^2}{n-1}}$$

$$\%RSD = \frac{SD}{RRF_{average}} x\ 100$$

where:

RRFi = individual RRF
RRF_{avg} = average RRF
n = number of calibration standards

7.6 Initial Calibration Verification

The initial calibration curve must be verified immediately after performing the ICAL using a 2nd source standard. The suggested acceptance limits for the initial calibration verification (ICV) analysis are 70-130%.

7.7 Continuing Calibration

1. A check of the initial calibration curve must be performed every 12 hours after an acceptable DFTPP analysis. Sample analysis may begin only after a successful DFTPP tune and a continuing calibration check have been acquired.

2. Inject 2 µL of a 200 µg/L calibration standard that contains the target analyte and internal standard. The internal standard concentration is 500 µg/L.

3. Calculate and tabulate the continuing calibration RRF for each compound.

4. Calculate the percent difference (%D) for the continuing calibration RRF compared to the average RRF from the initial calibration curve.

$$\%D = \frac{\left|(RRF_{Daily} - RRF_{Average})\right|}{RRF_{Average}} \times 100$$

The criteria for the continuing RRF and %D are found in Section 9.4.

5. The extracted ion current profile (EICP) area for each internal standard in the continuing calibration must be compared to the internal standard area in the mid-point standard of the current initial calibration. The criterion for comparison is found in section 9.4.

6. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift), then corrective action must be taken prior to the analysis of samples.

7.8 Sample Analysis

Prior to the analysis of calibration standards, media blanks, and/or samples, it is necessary to verify that the GC/MS:

- Met the DFTPP ion abundance criteria listed in Table 5, Appendix A and in Section 9.1. The DFTPP tune criteria must be demonstrated every 12 hours by analyzing 50 ng of DFTPP.

- Successfully passed an initial five-point calibration and/or continuing calibration check. The continuing calibration check must be demonstrated every 12 hours during sample analysis by analyzing a 200 µg/L 4-MCHM standard.

The media (lot) blanks, BS/BSD, field blanks, method blanks and samples must be analyzed with the same instrument conditions used for the calibration standards.

1. Add 20 µL of the internal standard mix into the media (lot) blank, BS/BSD, field blank, method blank and all sample extracts.

2. Inject 2 µL of the extract for each media blank, BS/BSD, field blank, method blank or sample.

3. If the response exceeds that of the highest calibration standard, the extract must be diluted so that the analyte response falls within the linear range established in the initial calibration. Ideally, the concentration of the analyte should fall midrange of the curve after dilution.

4. After a dilution is prepared, the internal standard mix is added accordingly, to maintain the required concentration of 500 µg/L in the diluted extract.

7.9 Identification of Target Analytes

The target analytes are identified by comparison of the sample mass spectra with the mass spectra of a calibration standard. Two criteria must be satisfied to verify the identifications:

- Elution of the sample component at the same GC relative retention time (RRT) as the standard component

- Correspondence of the sample component and standard component mass spectra

1. For establishing correspondence of the RRT, the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

2. For comparison of standard and sample components, reference mass spectra must be obtained from the 200 µg/L calibration standard. The standard mass spectra may be obtained from the run used to obtain the reference RRTs. In the case of co-elution of

standard components, the reference mass spectra from the National Institute of Standard and Technology (NIST) Mass Spectral Library should be used or the analyst can use professional judgment to establish the presence of target analytes. When technical judgment is used, the analyst must document the circumstances and their observations in the case narrative.

3. The requirements for qualitative verification of mass spectra are as follows:

   a. The three ions of greatest relative intensity must be present or any ions over 30% relative intensity if less than three such ions exist.

   b. The relative intensities of ions specified in (a) must agree within ±30% between the standard and sample spectra. For example, if an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 20-80%.

   c. Ions greater than 10% present in the *sample* spectrum but not in the *standard* spectrum must be considered and accounted for by the analyst making the comparison.

4. If the compound cannot be verified by all of the criteria in Step 3 but is identified by the technical judgment of the mass spectral interpretation specialist, the analyst shall report that identification and proceed with the calculation described in Section 8.0. The analyst should report in the case narrative the reasons why the compound is identified.

7.10 Desorption Efficiencies

The desorption efficiency (DE) is determined for each compound at 100, 400 and 1000 total ng for SIM analysis. Three replicate XAD tubes are spiked with a standard at each level, extracted with methylene chloride and analyzed by GC/MS. A blank is also extracted with methylene chloride and analyzed to determine any contamination in the methylene chloride. Typical desorption efficiencies are listed in Table 6, Appendix A for the 600 mg XAD tubes. Desorption efficiencies will be determined yearly. Supporting documentation will be kept on the SERAS local area network (LAN). The DEs are calculated using the equation in Section 8.2.

The concentration of the target compound found in the extract determines the application of the DE. For SIM analysis, use the DE determined from the 100 ng spike for samples with tentative concentrations from 0 to 200 ng, the DE from the 400 ng spike should be used for samples from 200 to <1000 ng, and the DE from the 1000 ng spike should be used for samples ≥ 1000 ng. The DE is applied separately to the front and back portions of each sorbent tube depending on the concentration calculated for each portion.

8.0 CALCULATIONS

8.1 Target Analytes

4-MCHM identified by the GC/MS method will be quantitated by the internal standard method. The internal standard (IS) used will be the one listed in Table 2. The area of the characteristic ions

of 4-MCHM and the IS are used. The target analyte concentrations and concentration conversions are calculated as follows:

1. Amount of analyte in each sorbent section (front or back) in µg/section:

$$\mu g/\text{section} = \frac{(A_s)(C_{is})}{(A_{is})(RRF_{avg})} x\ V_e\ x\ DE$$

where:

A_S	= Area of characteristic ion for the analyte to be measured
A_{IS}	= Area of characteristic ion for the internal standard
C_{IS}	= Concentration of internal standard (µg/mL)
RRF_{avg}	= Relative response factor of analyte
V_e	= Extraction Volume (mL)
DE	= Desorption efficiency

For the total mass of analyte per sample (µg/sample), add the mass calculated for both the front (W_f) and back (W_b) sections of the sample. If any target analyte is present in the media (lot) blanks, the average mass in µg for front (B_f) and back (B_b) sections will be subtracted from the total sample mass.

$$\mu g/\text{sample} = (W_f + W_b) - (B_{f\,avg} - B_{b\,avg})$$

NOTE: For the method blank, field blank and media (lot) blank, the results will be reported as total µg since no air is drawn through these samples.

2. Calculate the total sample concentration of each analyte in mg/m^3 ($C_{mg/m3}$) as follows:

$$C_{mg/m^3} = \frac{\text{Total } \mu g/\text{sample}}{\text{Liters sampled}}$$

All target concentrations are reported to three significant figures

3. Calculate the concentration of each analyte in parts per billion by volume (C_{ppbv}) as follows:

$$C_{ppbv} = C_{mg/m^3}\ x\ 24.45 x 1000/MW$$

where:

MW = Molecular Weight of the analyte.

4. Reporting Limit

The RL in µg/sample is equal to the mass injected for each analyte in the lowest concentration standard.

The RL in mg/m^3 is calculated using the following equation:

$$RL_{mg/m^3} = \frac{C_s}{RRF_{avg} \times V_s} x\ V_e\ x\ DE$$

where:

C_S = Concentration of lowest standard in the linear range of the six-point calibration curve
RRF_{avg} = Relative response factor of analyte
V_e = Extraction Volume (mL)
V_S = Sample volume (m^3)
DE = Desorption efficiency

The RL in ppbv is calculated using the following equation:

$$RL_{ppbv} = 24.45\,x\,\frac{(C_s)(V_e)(DE)}{(V_s)(MW)}$$

where:

C_S = Concentration of lowest standard in the linear range of the six- point calibration curve
V_E = Extract volume (mL)
V_S = Sample volume (m^3)
DE = Desorption efficiency
MW= Molecular weight of analyte

8.2 Desorption Efficiencies

The desorption efficiency (DE) is determined for each compound at 100, 400 and 1000 ng levels for the SIM analysis. A desorption study must be performed yearly. The DE for each concentration level is calculated as the inverse of the percent recovery (%R) times one hundred and expressed as a decimal.

$$\%R = \frac{\text{Average Amount Recovered}\,(\mu g)}{\text{Spiked Amount}\,(\mu g)}$$

$$DE = \frac{1}{\%R} x\ 100$$

The amount recovered is calculated by:

$$\mu g/\text{sample} = \frac{(A_s\ (C_{is})}{(A_{is})\ (RRF_{avg})}\ x\ V_e$$

where:

A_S	=	Area of the characteristic ion for the compound to be measured
A_{IS}	=	Area of the characteristic ion for the specific internal standard (IS)
C_{IS}	=	Amount of internal standard added in µg/mL
RRF_{avg}	=	Average relative response factor
V_e	=	Extraction volume (mL)

8.3 Blank Spike/Blank Spike Duplicate Recoveries

For SIM analysis, the BS/BSD spike is 200 ng of each isomer. No air is drawn through the XAD tubes. The amount recovered (ng/sample) is calculated using the equation in Section 8.1.

The %R for the BS/BSD and relative percent difference (RPD) can be calculated using the equations below:

$$\text{Blank Spike Recovery}(\%R) = \frac{SBR - BR}{SA}\ x\ 100$$

where:

SBR	=	spike blank result
BR	=	blank result

$$RPD = \frac{(|BSR - BSDR|)}{(BSR + BSDR)/2}\ x\ 100$$

where:

RPD	=	relative percent difference
BSR	=	blank spike recovery
BSDR	=	blank spike duplicate recovery

Note: RPD is always expressed as a positive value.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

9.1 GC/MS Tuning and Performance Criteria

The GC/MS tune must be verified using DFTPP. The ion abundance criteria listed in Table 5, Appendix A must be met prior to any standard, blank or sample analysis. In addition, the criteria must be achieved during every 12-hour period during which standards, blanks, and samples are analyzed. The 12-hour time period for GC/MS tuning begins at the time of DFTPP injection that the laboratory submits as documentation of a compliant tune. The degradation of DDT to DDE and DDD should not exceed 20%. The tailing factor for benzidine and pentachlorophenol must not exceed a tailing factor of 2.

Figure 1, Appendix B shows an example calculation of the tailing factor. Figure 2, Appendix B illustrates the tailing factor for pentachlorophenol and benzidine, and the % breakdown of DDT calculated using the HP Enviroquant software. This feature is found in the *ChromEval* options of *Qedit* in the data analysis mode.

9.2 GC/MS Initial Calibration

4-MCHM must meet the minimum acceptable response factor (RF) of 0.05. The %RSD should be less than or equal to 20% or the minimum correlation coefficient (r) criterion of 0.99 for 4-MCHM using linear regression. If the compound criteria is greater than (>) 20% but less than 40%, the compound will be qualified estimated and documented in the case narrative of the analytical data package. The analyst is responsible for determining if maintenance should be conducted prior to the analysis of any additional samples. If 4-MCHM included with the initial calibration exceeds the 20% RSD or the minimum 0.99 correlation coefficient, then the chromatographic system is considered too reactive for analysis to begin and corrective action must be taken. If linear regression is used, then the low point concentration is recalculated using the curve and should be ±30% of its true concentration.

NOTE: All initial calibration standards must be analyzed prior to the analysis of any media blanks, method blank, QC samples or environmental samples.

9.3 GC/MS Initial Calibration Verification

The initial calibration curve must be verified immediately after performing the ICAL using a 2nd source standard defined as the ICV. The suggested acceptance limits for the ICV are 70-130%.

9.4 GC/MS Continuing Calibration

After 12 hours of sample acquisition have passed, the GC/MS tune must be re-evaluated using DFTPP, and the initial calibration curve verified by analyzing a mid-level calibration standard.

1. The DFTPP tune must pass the criteria in Table 5, Appendix A.

2. The 200 µg/L calibration standard must be used for the continuing calibration.

3. The %D should be less than or equal to 20% for 4-MCHM and must meet the RRF limit of 0.05. The EICP area for the internal standard in the continuing calibration must be between 50% and 200% of the respective internal standard EICP area in the mid-point standard of the current initial calibration. If this criterion is not met, re-analysis is required.

4. A maximum of two continuing calibrations may be run to meet the requirements in item 3 above. If two continuing calibrations fail to meet acceptable criteria, corrective action must be taken prior to a new calibration curve being run.

   If the instrument is set up on an overnight run with two continuing calibrations back to back and the first continuing calibration passes but the second one fails, then the data is not usable and corrective action must be taken. It is not acceptable to use the first continuing calibration if the second continuing calibration is out of acceptance criteria.

5. If any of the requirements listed in Step 3 are not met, the Analytical Support Leader should be notified so appropriate corrective actions can be taken.

9.5 Internal Standard Area Evaluation

1. The amount of each internal standard in a 2-µL injection of sample extract must be 500 µg/L.

2. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, media blank, field blank, method blank and BS/BSD.

3. If samples, media blanks, field blanks, method blanks or BS/BSDs are analyzed immediately following an initial calibration but before another DFTPP tune and a continuing calibration, the evaluation will be conducted on the basis of the internal standard areas of the 200 µg/L initial calibration standard.

4. The EICP area for each internal standard in all samples, media blanks, field blanks, method blank and BS/BSDs must be between 50% and 200% of the respective internal standard EICP area in the appropriate calibration standard. In addition, the retention time of each internal standard must be within ±0.50 minutes (30 seconds) of its retention time in the continuing calibration standard.

5. If one or more internal standard EICP areas do not meet criteria, the GC/MS system must be inspected for malfunctions and corrections made as appropriate. When corrections are made, re-analysis of all affected samples is required.

6. If after re-analysis, the EICP areas for all internal standards meet criteria (between 50% and 200%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, <u>only</u> data from the analysis with EICPs within the limits are required to be submitted. If re-analysis confirms matrix effects, submit both sets of data but report the initial run.

9.6 Method Blank Analysis

A method blank is an aliquot of methylene chloride that is carried through the entire analytical procedure. The purpose of a method blank is to determine the level of contamination associated with preparation and analysis of samples.

1. One method blank must be prepared for each batch of 20 samples.

2. A method blank should contain less than (<) the RL of 4-MCHM.

3. If a method blank exceeds the contamination limits as described above, the analytical system is considered as unacceptable. The source of contamination must be investigated so that appropriate corrective actions can be taken and documented, before proceeding with any further sample analysis. Due to the nature of the samples, re-extraction is not possible. The method blank is used to qualify hits in the samples if the concentrations found in the samples are within five times the level found in the method blank. Data are qualified during the validation process according to standard validation procedures.

9.7 Media (Lot) Blank Analysis

A media (lot) blank is an unopened XAD-2 resin tube from the same lot as the sample tubes. The purpose of the media (lot) blank is to determine the levels of contamination associated with the manufacture, extraction, and analysis of the samples. The average concentration for the front section and the back section is subtracted from the concentrations found in the samples and QC samples.

1. At least three media (lot) blanks must be extracted and analyzed for every XAD tube lot represented in the sampling event for each project.

2. The media (lot) blank must contain less than or equal to the RL of any single target compound.

The average media (lot) blank concentration is subtracted from the sample results.

9.8 Blank Spike/Blank Spike Duplicate

The XAD tubes for the BS/BSD are spiked with 200 ng of each isomer of 4-MCHM. No air is drawn through the XAD tubes. The purpose of the BS/BSD is to evaluate the accuracy and precision of the extraction and analysis, including possible sample matrix effects.

1. One BS/BSD must be prepared every 20 samples or per project. The BS/BSD must be associated with a method blank that meets the criteria in Section 9.6 and must be extracted and analyzed within holding time.

2. The spike solution specified in Section 7.2.6 must be used.

3. Spike recoveries and relative percent difference (RPD) of each spike compound in the BS/BSD are calculated according to the equations specified in Section 8.3.

4. Quality control limits for the BS/BSD is 70-130% for %R and ±20% for the RPD. The QC limits are advisory at this time and no further action is required if the recovery fall outside the limits.

If the laboratory fails to meet the recovery QC limits and the RPD limits on a routine basis, the Analytical Support Leader must investigate the cause and take corrective action.

9.9 Field Blank Analysis

A field blank is collected each day of sampling. A field blank is an opened XAD-2 resin tube from the same lot as the sample tubes. The purpose of the field blank is to determine the levels of contamination associated with the collection, storage, and analysis of the samples. Just as for environmental samples, the average media (lot) blank concentration is subtracted from the sample results. The field blank may be used to qualify hits in the samples if the concentrations found in the samples are within five times the level found in the field blank. Data are qualified during the validation process according to standard validation procedures.

9.10 Breakthrough

Any target analytes observed in the back portions above the blank concentrations may indicate breakthrough of that analyte either due to high target concentrations exceeding the adsorption capacity of the sorbent tube or due to excessively high sample volumes. Any breakthrough observed in the back portion above 5% of the concentration in the front portion should be noted in the laboratory narrative. For excessive breakthrough (>30%) the laboratory should note that the final concentrations should be used with caution by the user. If the concentration of the analyte in the back portion is greater than in the front portion the laboratory should note that the results indicate gross breakthrough and that the data is not usable.

9.11 Dilution Analysis

If the concentration of 4-MCHM in a sample extract exceeds the initial calibration range, the sample extract must be diluted and reanalyzed as described in Section 7.8.

1. Use the results from the initial analysis to estimate the approximate dilution factor needed to bring the highest concentration within the linear calibration range.

2. The dilution factor chosen should bring the highest target analyte within the upper half of the calibration range.

3. Submit the data from the original sample and the dilution in which analytes fall within the calibration range. If the screening procedure determines that the extract cannot be analyzed undiluted, submit the data from the first dilution and a subsequent dilution in which analytes fall within the calibration range. NOTE: Except in extreme cases, all extracts should be run undiluted to achieve the lowest detection limit.

9.12 Manual Integrations

Manual integration of 4-MCHM and the internal standard for standards, samples and QC samples will be submitted for review. The manual integration results will be flagged with a "M" and will be initialed and dated by the analyst indicating that the integration was performed properly. A reason for the manual integration should be documented if not readily obvious to the data reviewer. Documentation of the manual integration (before and after total ion chromatograms) of quantitation ion peaks must be included in the data package. Refer to SERAS SOP #1001, *Chromatographic Peak Integration Procedures* for more details.

9.13 Demonstration of Capability

Prior to running a method or any time there is a change in instrument type, personnel or test method or any time a method or analyst has not performed a method in a twelve-month period, an analyst must demonstrate acceptable performance for that method. This is known as the initial demonstration of capability (DOC). Four aliquots of a QC standard (second source) also known as a laboratory control sample (LCS) must be prepared and analyzed according to the method over one or a period of days. If the method does not specify a concentration to be used for the DOC, then the concentration must be prepared at 1-4 times the RL. Using Excel, the analyst must provide a table containing the following: Results of the 4 replicates, Mean Concentration, Mean Recovery, Standard Deviation and %RSD with the LCS acceptance limits.

On-going demonstration of capability on an annual basis may be satisfied by the analyst successfully passing a performance testing (PT) sample or a minimum of 4 consecutive LCS samples with acceptable precision and accuracy levels or another initial DOC. Results of tabulated QC samples (i.e., control chart data) that are analyst-specific accompanied by precision and accuracy criteria agreed upon by the Analytical Support Leader and the QA/QC Officer may also be used.

9.14 Limit of Detection/Limit of Quantitation Studies

The LOD must be determined for each target analyte on every instrument that will be used for the analysis and reporting of samples. The LOD must be determined each time there is a change in the method that affects how the test is performed or when there is a change in instrumentation. The LOD must be verified annually for each matrix, method and analyte. The LOD will be run using a minimum of seven replicates of a sample prepared from the calibration source at 4 times the LOD for multiple analyte tests. Each of these 7 aliquots must be subjected to the entire analytical procedure. Calculate the mean, mean recovery, variance and standard deviation of the replicate measurements. The LOD is calculated by multiplying the standard deviation times the Students t-Value of 3.143. If more than 7 replicates are used, the Students t-Value must be adjusted accordingly.

The LOQ must be confirmed by the successful analysis of a secondary source QC sample containing each target analyte in each matrix at 1-2 times the claimed LOQ (typically the low standard). The recoveries must fall within the recoveries established for the LCS. Alternately, a control chart with recoveries established from previously analyzed LOQ samples may be used.

The LOQ must be subjected to the entire analytical procedure. The LOQ must be verified annually for each matrix, method and analyte. The LOD/LOQ must be determined at the same time.

9.15 Nonconformance Memo

A nonconformance memo will be generated any time an employee notices a deficiency suspected of being a nonconformance. This nonconformance memo will be forwarded to the QA/QC Officer for verification of corrective action.

10.0 DATA VALIDATION

Data will be assessed in accordance with the guidelines set forth in the most current version of SERAS SOP #1016, *Data Validation Procedures for Routine Semivolatile Organic Analysis*. However, data are considered satisfactory for submission when all the following requirements are met.

1. All samples must be analyzed under an acceptable tune, initial calibration, and continuing calibration check at the required frequency.

2. The QC requirements described in Section 9.0 should be met at all times. Any deviation or anomalous conditions should be discussed with the Analytical Support Leader and documented on a nonconformance memo.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, refer to EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to SERAS SOP #3013, *Chemical Hygiene Plan* and SERAS SOP #1501, *Hazardous Waste Management*.

12.0 REFERENCES

U.S. EPA. 1984. Federal Register, 40 Code of Federal Regulations (CFR) Part 136, Appendix B, *Definition and Procedure of the Determination of the Method Detection Limit - Revision 1.11*, October 26, 1984.

National Institute for Occupational Safety and Health. 1994. *NIOSH Manual of Analytical Methods*, Fourth Edition, Method 5515, Polynuclear Aromatic Hydrocarbons by GC, Issue 2, August 15, 1994, modified for 4-MCHM.

National Environmental Laboratory Accreditation Conference (NELAC), *2003 NELAC Standard*, EPA/600/R-04/003.

The NELAC Institute (TNI), 2009. Management and Technical Requirements for Laboratories Performing Environmental Analysis, Volume 1.

13.0 APPENDICES

A – Tables
B – Figures

APPENDIX A
Tables
SOP #1858
June 2014

TABLE 1. 4-MCHM Method Detection and Reporting Limits Based on Volume Collected[1]

Flow Rate, L/min	Sampling Time, minutes	Total Volume, Liters	Detection Limit[2], µg/m^3	Reporting Limit[3], µg/m^3
0.1	60	6	2.25	16.7
0.1	120	12	1.13	8.33
0.1	240	24	0.563	4.17
0.1	480	48	0.281	2.08
0.25	60	15	0.900	6.67
0.25	120	30	0.450	3.33
0.25	240	60	0.225	1.67
0.25	480	120	0.113	0.833

[1]Detection and Reporting Limits are not adjusted based on desorption efficiencies
[2]Based on method detection limit study dated 2/12/2014 on instrument Smyrna II
[3]Based on lowest calibration standard

TABLE 2. Characteristic Ions for Target Analyte and Internal Standard

Analyte	Primary Ion	Secondary Ion(s)
2-Fluorobiphenyl (ISTD)	172	171
4-Methylcyclohexane methanol	55	97, 95

TABLE 3. Recommended GC/MS Instrument Parameters

Sample Inlet:	GC
Injection Source:	GC ALS
Mass Spectrometer:	Enabled
Oven	
Equilibration Time	0.5 min
Oven Program	On
	50°C for 1 min
	then 17.5°C/min to 200 °C for 0.5 min
	then 35°C/min to 315 °C for 2 min
Run Time	15.357 min
Front Injector	
Syringe Size	10 µL
Injection Volume	2 µL
Solvent A Washes (PreInj)	2
Solvent A Washes (PostInj)	5
Solvent A Volume	8 µL
Solvent B Washes (PreInj)	2
Solvent B Washes (PostInj)	5
Solvent B Volume	8 µL
Sample Washes	2
Sample Wash Volume	5 µL
Sample Pumps	2
Dwell Time (PreInj)	0 min
Dwell Time (PostInj)	0 min
Solvent Wash Draw Speed	300 µL/min
Solvent Wash Dispense Speed	6000 µL/min
Sample Wash Draw Speed	300 µL/min
Sample Wash Dispense Speed	6000 µL/min
Injection Dispense Speed	6000 µL/min
Viscosity Delay	0 sec
Sample Depth	Disabled
Front SS Inlet He	
Mode	Pulsed Split
Heater	On 270°C
Pressure	On 13.329 psi

TABLE 3. Recommended GC/MS Instrument Parameters (cont.)

Total Flow	On	22.5 mL/min
Septum Purge Flow	On	3 mL/min
Gas Saver	On	35 mL/min After 0.75 min
Split Ratio	12:1	
Split Flow	18 mL/min	
Injection Pulse Pressure	20 psi Until 0.5 min	

Thermal Aux 2 {MSD Transfer Line}	
Heater	On
Temperature Program	On
	325 °C for 0 min
Run Time	15.357 min

Column #1	
ZB-5	Serial # 140533
	30 m x 250 µm x 0.5 µm
	In: Front SS Inlet He
	Out: Vacuum
(Initial)	50°C
Pressure	13.239 psi
Flow	1.5 mL/min
Average Velocity	43.91 cm/sec
Holdup Time	1.1387 min
Flow Program	Off
Run Time	15.357 min

MS ACQUISITION PARAMETERS

General Information
--- ---

Tune File:	Xtune.U
Acquisition Mode:	SIM

TABLE 3. Recommended GC/MS Instrument Parameters (cont.)

MS Information
-- ----------

Solvent Delay:	2.20 min		
EMV Mode:	Relative		
Relative Voltage:	47		
Resulting EM Voltage:	1635		
[SIM Parameters]			
GROUP 1			
Resolution: Low			
Plot 1 Ion: 54			
Ions/Dwell in Group	(Mass, Dwell)	(Mass, Dwell)	(Mass, Dwell)
	(54.00 50)	(55.00 50)	(82.00 50)
	(95.00 50)	(97.00 50)	(128.00 50)
	(171.00 50)	(172.00 50)	
[MSZones]			
MS Source:	280 C maximum 300ºC		
MS Quad:	180 C maximum 200ºC		

TABLE 4. Recommended DFTPP Tune GC/MS Instrument Parameters

Sample Inlet:	GC
Injection Source:	GC ALS
Mass Spectrometer:	Enabled
Oven	
Equilibration Time	0.5 min
Oven Program	On
	100°C for 0.5 min
	then 30°C/min to 225 °C for 4 min
	then 30°C/min to 315 °C for 3 min
Run Time	14.667 min
Front Injector	
Syringe Size	10 µL
Injection Volume	2 µL
Solvent A Washes (PreInj)	2
Solvent A Washes (PostInj)	2
Solvent A Volume	8 µL
Solvent B Washes (PreInj)	2
Solvent B Washes (PostInj)	2
Solvent B Volume	8 µL
Sample Washes	1
Sample Wash Volume	8 µL
Sample Pumps	2
Dwell Time (PreInj)	0 min
Dwell Time (PostInj)	0 min
Solvent Wash Draw Speed	300 µL/min
Solvent Wash Dispense Speed	6000 µL/min
Sample Wash Draw Speed	300 µL/min
Sample Wash Dispense Speed	6000 µL/min
Injection Dispense Speed	6000 µL/min
Viscosity Delay	0 sec
Sample Depth	Disabled
Front SS Inlet He	
Mode	Split
Heater	On 270°C
Pressure	On 15.583 psi

TABLE 4. Recommended DFTPP Tune GC/MS Instrument Parameters (cont.)

Total Flow	On	23.8 mL/min
Septum Purge Flow	On	3 mL/min
Gas Saver	On	29 mL/min After 0.5 min
Split Ratio	15:1	
Split Flow	18 mL/min	
Thermal Aux 2 {MSD Transfer Line}		
Heater	On	
Temperature Program	On	
	325 °C for 0 min	
Run Time	14.667 min	
Column #1		
ZB-5	Serial # 140533	
	30 m x 250 µm x 0.5 µm	
	In: Front SS Inlet He	
	Out: Vacuum	
(Initial)	100°C	
Pressure	15.583 psi	
Flow	1.3 mL/min	
Average Velocity	40.542 cm/sec	
Holdup Time	1.2333 min	
Flow Program	On	
	1.3 mL/min for 8 min,	
	then 2 mL/min to 2.5 mL/min for 4 min	
Run Time	15.357 min	

MS ACQUISITION PARAMETERS

General Information
--- ---

Tune File:	Xtune.U
Acquisition Mode:	Scan

TABLE 4. Recommended DFTPP Tune GC/MS Instrument Parameters (cont.)

MS Information
-- ----------

Solvent Delay:	3.00 min	
EMV Mode:	Relative	
Relative Voltage:	357	
Resulting EM Voltage:	1624	
[Scan Parameters]		
Low Mass:	15.0	
High Mass:	490.0	
Threshold:	75	
Sample #:	1	A/D Samples 2
[MSZones]		
MS Source:	280 C	maximum 300ºC
MS Quad:	180 C	maximum 200ºC

TABLE 5. Ion Abundance Criteria for Tune (DFTPP) [1]

Mass	Ion Abundance Criteria
51	10.0 - 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
70	Less than 2.0 percent of mass 69
127	10 – 80 percent of mass 198
197	Less than 2.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see note)
199	5.0 - 9.0 percent of mass 198
275	10.0 – 60.0 percent of mass 198
365	Greater than 1.0 percent of mass 198
441	Present but < 24% of mass 442
442	Base Peak or > 50% of mass 198
443	15.0 - 24.0 percent of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

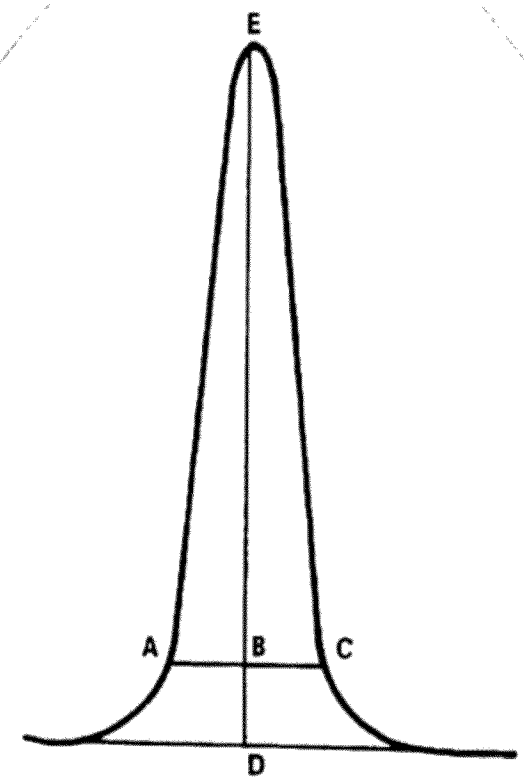
[1] Criteria taken from U.S. EPA/SW846, Method 8270D, Revision 4.0, February 2007

TABLE 6. Typical Desorption Efficiencies for a 600-mg XAD Tube

	Desorption Efficiency		
Compound	100 ng	400 ng	1000 ng
4-MCHM (cis and trans)	1.0885	1.1204	1.0926

APPENDIX B
Figures
SOP #1858
June 2014

FIGURE 1. Tailing Factor Calculation



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

FIGURE 2. Tailing factor for pentachlorophenol and benzidine, and the % breakdown of DDT calculated using the HP Enviroquant software

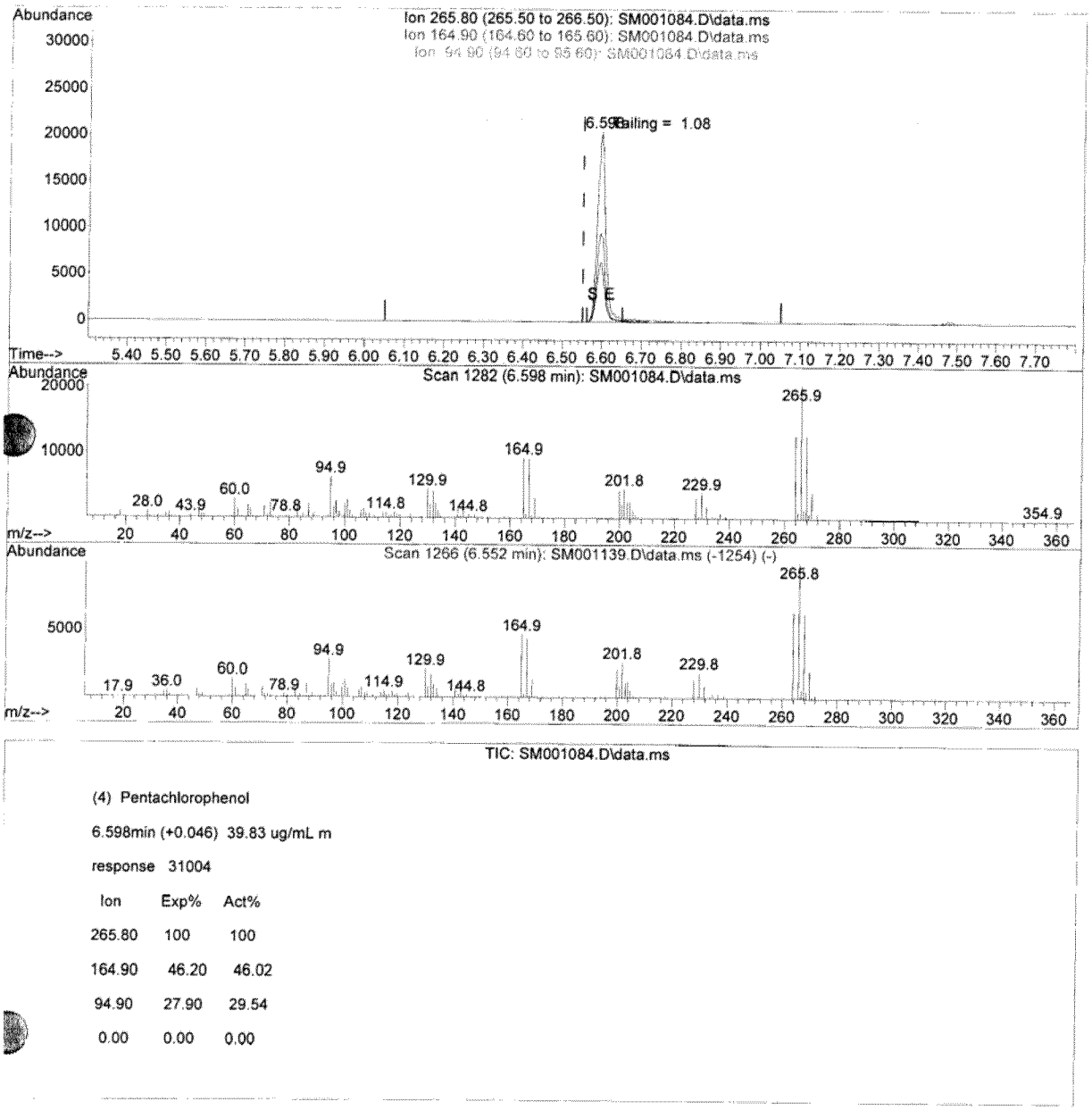
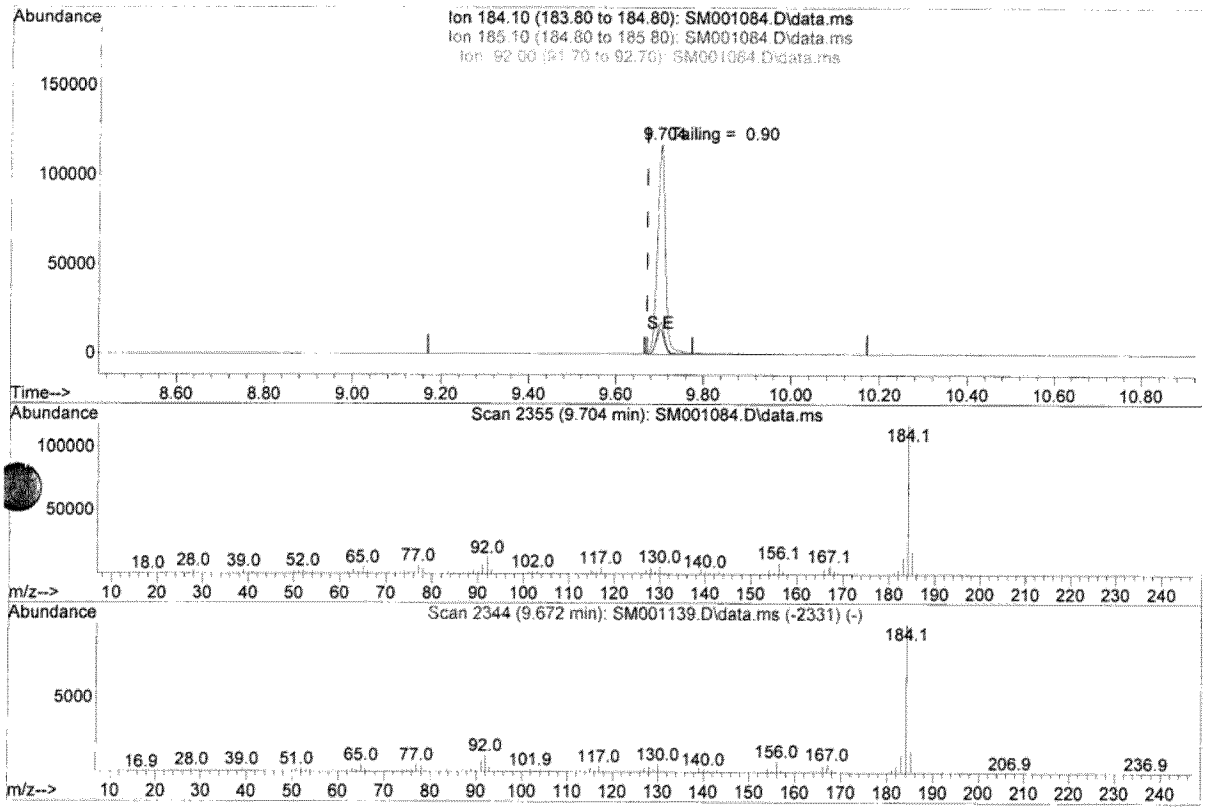


FIGURE 2. Tailing factor for pentachlorophenol and benzidine, and the % breakdown of DDT calculated using the HP Enviroquant software (cont'd)



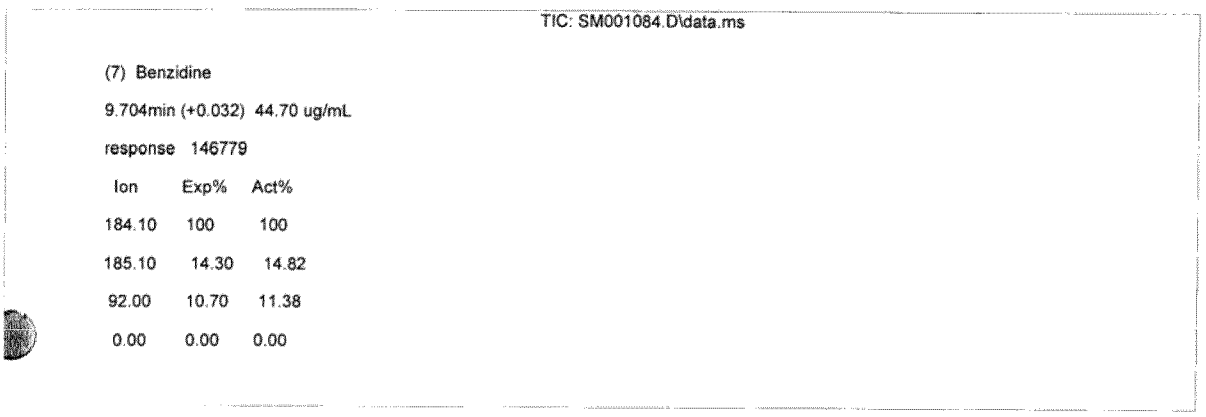
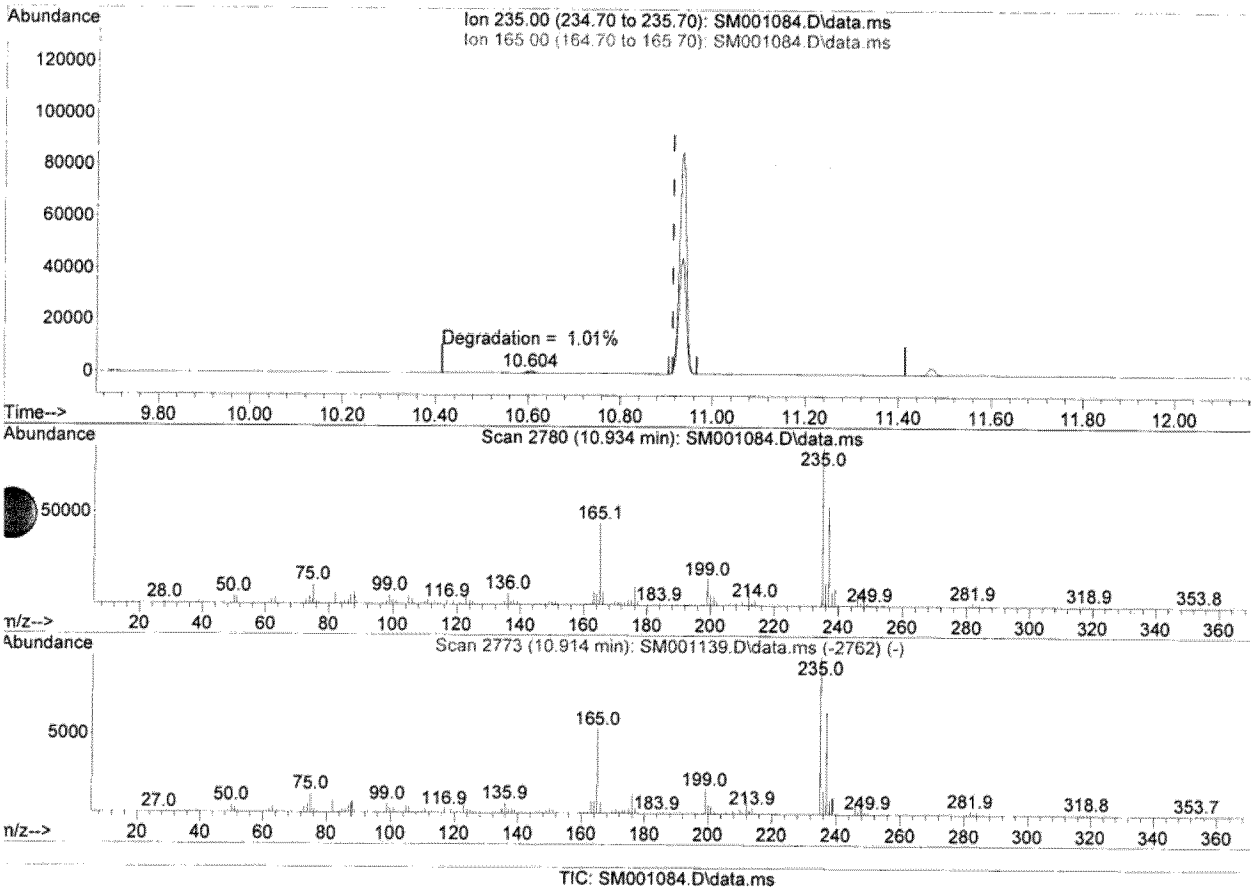


FIGURE 2. Tailing factor for pentachlorophenol and benzidine, and the % breakdown of DDT calculated using the HP Enviroquant software (cont'd)



(8) DDT

10.934min (+0.020) 52.85 ug/mL

response 90296

Ion	Exp%	Act%
235.00	100	100
165.00	52.10	53.15
0.00	0.00	0.00
0.00	0.00	0.00