



Determination of Volatile and Semi-Volatile Organic Compounds from Vehicle Automotive Materials using a Micro-Scale Chamber

1 Scope

Note: Nothing in this standard supercedes applicable laws and regulations.

Note: In the event of conflict between the English and domestic language, the English language shall take precedence.

1.1 Purpose. This standard is used for the qualitative and quantitative determination of Volatile Organic Compound (VOC) and Semi-Volatile Organic Compound (SVOC) emissions in automotive material (AM) utilizing the micro-scale chamber and a Thermal Desorber – Gas Chromatograph / Mass Selective Detector (TD-GC/MSD). Individual VOC and SVOC emissions in different vehicle materials can be identified, quantified, and compared. This method is an efficient and effective way to isolate emission sources and odors from automotive materials.

1.1.1 Individual VOC and SVOC emissions in different vehicle automotive materials can be identified, quantified, and compared.

1.1.2 VOC emissions in the boiling point range $\leq +345$ °C or chromatographic elution range from n-pentane (n-C5) to n-eicosane (n-C20) (VOC value, GM) can be detected by one of the sorbent tube types specified in this method. It is assumed that these substances can be detected from vehicle interior air and total vehicle emissions.

1.1.3 Emissions that condense on a vehicle surface (e.g., glass, plastic lens, etc.) at ambient temperature are classified as SVOC or FOG emissions, as defined in glossary. SVOC emissions are in the boiling point range $\geq +280$ °C or chromatographic elution range from n-hexadecane (n-C16) to n-dotriacontane (n-C32) (SVOC or FOG value, GM) and can be detected by this method.

1.1.4 Individual VOC and SVOC emissions identified as an odor can be quantified and compared.

1.2 Foreword. Determination of VOCs and SVOCs for Vehicle Interior Air Quality (VIAQ) is essential to meet customer and regulatory requirements in some global markets. VOCs and SVOCs found in interior air and total vehicle emissions originate from an automotive material source. Measurements are reported in parts per million (ppm) concentrations, which warrant accurate analytical results.

1.3 Applicability. All Automotive materials that can contribute to emissions in the vehicle interior air and total vehicle emissions can be tested. Examples include emissions from textiles, carpets, polymers, rubbers, elastomers, films, leather, and composite materials. Additional examples include cured adhesives, sealants, foams, primers, paints and coatings.

1.3.1 In relation to emissions, the relevant vehicle interior volume comprises all areas that are linked to the passenger compartment either directly or by air contact. Therefore, it includes the luggage compartment, the Heating, Ventilation, and Air Conditioning (HVAC) system, etc.

1.3.2 The results from this test procedure will provide additional information to interpret findings in GMW3059, GMW3205, GMW3235 and GMW15634.

1.3.3 Suppliers must submit the micro-scale chamber parameter conditions and Thermal Desorption - Gas Chromatography / Mass Spectrometry (TD-GC/MS) analysis results together with an automotive material sample.

1.3.4 This standard complements ISO 12219-3 and results may be compared to American Industrial Hygiene Association (AIHA) health standards.

2 References

Note: Only the latest approved standards are applicable unless otherwise specified.

2.1 External Standards/Specifications.

DIN 55531 ISO 16000-6
ISO 9001 ISO/IEC 17025
ISO 12219-3

2.2 GM Standards/Specifications.

GMW3059 GMW15634
GMW3205 GMW15654
GMW3235

2.3 Additional References.

Odor thresholds for chemicals with established occupational health standards may be purchased at www.aiha.org.

3 Resources

3.1 Facilities.

3.1.1 Calibration. The test facilities and equipment shall be in good working order and shall have a valid calibration label.

3.1.2 Alternatives. Alternative test facilities and equipment may also be used. However, all measuring variables as specified in this standard shall be determined correctly with respect to their physical definition.

3.1.3 All tests per this standard shall be performed by a laboratory that conforms to ISO/IEC 17025 or ISO 9001 or other 3rd party accreditation that is acceptable to the GM Materials Engineer. Supplier shall submit documentation showing proof of accreditation.

Note: Meets GM requirements but may not be formally recognized by third parties.

3.2 Equipment. This standard utilizes commonly available sampling equipment and analytical instrumentation. Specific test instrumentation is given in Appendix A. Other equipment can also be used when compatibility has been shown.

3.2.1 Balance. Balance with a sensitivity of ± 0.01 mg.

3.2.2 Micro-scale Chamber Apparatus. A 6-chamber (44 cm³ capacity, 45 mm ID x 28 mm deep), +120 °C ceiling maximum or a 4-chamber (114 cm³ capacity, 64 mm ID x 36 mm deep), +250 °C ceiling maximum may be used.

3.2.3 Directly coupled thermal desorption (TD) gas chromatography system with trapping and split system.

3.2.4 Gas Chromatograph (GC) with Mass Selective Detector (MSD), software, and mass spectra libraries. The method may use single-quadrupole or triple-quadrupole mass selective detectors.

3.2.5 GC Capillary Columns. A 2 m length of 0.32 mm Internal Diameter (ID) deactivated, uncoated fused silica GC capillary transfer column is used within the heated transfer line to connect the effluent from the TD focusing trap to the specified analytical capillary column within the GC oven. An inert, zero dead-volume capillary-capillary coupling is used to connect the two. The specified analytical column is a low-bleed Gas Chromatograph / Mass Selective Detector (GC/MSD) capillary column with a (5 %-phenyl) methylpolysiloxane stationary phase (e.g., Agilent Technologies 60 m x 0.32 mm x 0.50 μ m DB-5MS, referenced in Appendix A). Other columns can also be used when compatibility has been shown.

3.2.6 Sample Tubes. Sample tubes with deactivated glass surface.

3.2.6.1 Sample Tubes packed with Tenax TA. Compatible with the specific corresponding thermal desorption system (Figure G1). These tubes are suitable for VOC and FOG analysis, but retention of the lightest VOCs, such as n-pentane to n-heptane peak elutions will be limited. Sample tubes packed with Tenax TA backed up by Carbograph 5TD (Tenax/CG5TD). Compatible with the specific corresponding thermal desorption system (see Figure G1). As described in ISO 16000-6, Annex D. They are suitable for all VOC and FOG analyses including the lightest compounds.

3.2.7 Calibration Solution Loading Rig™ (CSLR). The CSLR is an unheated injection port with a controlled carrier gas supply that connects to the sampling end of the Solid Absorbent Media Glass Thermodesorption Tubes. The flow of Nitrogen (N₂) carrier gas through the tube is set between 80 ml/minute and 100 ml/minute. The liquid calibration standard is introduced through the injector septum using a (1 to 10) µl GC syringe volume. Liquid standard solutions vaporize in the flow of N₂ carrier gas allowing standards to reach the sorbent media in the gas phase. A sufficient volume of N₂ carrier gas is allowed to pass through the sorbent tube such that most of the solvent is purged from the tube to vent while target compounds are quantitatively retained (see Figure G2).

Note: Solvent purging is most readily achieved using Tenax tubes. Tenax/CG5TD tubes will retain more of the solvent so smaller liquid injection volumes ((0.5 to 1) µl) should be used in this case.

3.2.8 Carrier Gas. Grade ≥ 5.0 Helium (He) carrier gas and a corresponding in-line purifier trap are used for the GC and can be selected for the micro-scale chamber.

3.2.9 Conditioning Purge Gas. Grade ≥ 5.0 N₂ carrier gas and a corresponding inline purifier trap are used for conditioning Solid Absorbent Media Glass Thermodesorption Tubes, supplying the CSLR and can be selected for the micro-scale chamber. This same gas may also be used as the dry gas supply for the electrically-cooled focusing trap of the desorber and to manage the TD auto sampler pneumatics.

3.2.10 Sampling Gas. Grade ≥ 5 Ultra Pure Air is recommended for purging the micro-scale chamber to simulate the vehicle atmosphere conditions. Carrier gases (paragraphs 3.2.8 and 3.2.9) may also be used in certain circumstances (e.g. when testing oxidative, reactive or uncured materials.)

3.2.11 Conditioning the Solid Absorbent Media Glass Thermal Desorption Tubes. The Solid Absorbent Media Glass Thermodesorption Tubes are cleaned thermally with an apparatus designed for that purpose, such as a multi-tube conditioner and dry purge unit, or equivalent. Conditioning consists of heating new Solid Absorbent Media Glass Thermodesorption Tubes at +320 °C continuously for 2 h between 50 ml/minute and 100 ml/minute flow of N₂. Reconditioning consists of heating continuously for 30 minutes at +335°C using between 50 ml/minute and 100 ml/minute flow of N₂ Solid Absorbent Media Glass Thermodesorption Tubes must be conditioned before each use.

3.2.12 Conditioning of Tube Caps for Analysis and Field Use. The (metal) components of the screw-fitting tube storage caps used in the field may be conditioned in an oven at +300 °C for 20 minutes if/when required. Note that the polytetrafluoroethylene (PTFE) ferrules used in these caps are not compatible with temperatures > +210 °C and shall be removed before this treatment.

Alternatively, field storage caps and analytical caps (i.e. caps used on the tubes during thermal desorption) may be conditioned with their plastic fittings in place (e.g. PTFE seals and/or Viton o-rings) using a vacuum oven set at +200 °C.

Examples of tube caps used for analysis and field use are shown in Appendix G, Figure G3.

3.2.13 GC Syringes. The required GC syringe volumes for spiking tubes are 1 µl, 5 µl, and 10 µl.

3.2.14 Solvents for Standard Dilutions.

- Methanol (MeOH), HPLC grade.
- Dichloromethane (DCM), HPLC grade.

3.2.15 Standards and Dilutions. All standards and dilutions shall be kept in sealed containers and refrigerated ≤ -8 °C when not in use.

3.2.15.1 Standards of desired VOC and SVOC components at ≥ 98 % purity (or equivalent).

3.2.15.2 Check standard containing a mixture of 18 individual components, (110 ± 10) µg/ml each in MeOH (see Appendix B, Table B1).

3.2.15.3 Internal standard calibration mix (ISCM) consisting of deuterated toluene-D8 [CAS# 2037-26-5] and deuterated n-hexadecane-D34 [CAS# 15716-08-2], (500 ± 50) µg/ml each in DCM (Appendix B, paragraph B2).

3.2.15.4 n-Alkane standard containing n-C5 to n-C32 of 28 individual n-alkane components, 2000 µg/ml each in DCM.

3.2.15.5 Aromatics standard containing aromatic hydrocarbons, 14 individual components, 2000 µg/ml each in MeOH.

3.2.15.6 Grob test mix for testing the GC column performance.

3.2.15.7 Prior to analysis, the standard should be taken from the refrigerator and stored sealed at room temperature for 2 h to prevent condensation and solidification. Standards shall be mixed vigorously before being drawn upon.

3.2.15.8 All standards except Internal standard calibration mix shall be used within 60 days and shall be disposed of properly. Internal standard calibration mix standards shall be used within 30 days.

3.3 Test Vehicle/Test Piece. This standard is used to test vehicle automotive materials that are contributing emissions. Automotive materials should be obtained from suppliers as soon as possible after production. The sample should be taken within seven (7) days after production. Manufacturing date and sampling date should be documented. Care should be taken to prevent material out-gassing before analysis (e.g., by using clean, air-tight packaging during transportation and storage and by storing packed samples under refrigerated conditions – See details below).

3.3.1 For the analysis, a representative sample of the material is taken. For larger parts, smaller samples are cut into an approximate sample size of 10 cm x 15 cm. The sample should not be touched with bare hands, fingers and nails. The cutting shall be done with clean oil-free tools. The sample should be packed into aluminum composite films (e.g. Vacupack™, or any other material according to DIN 55531). In the exceptional case, the sample can be packed twice into thick silicone-free aluminum foil (cleaned with n-pentane), the edges should be folded multiple times to prevent contamination and to ensure a gastight packing. The aluminum foil packed sample should then be placed within a polyethylene bag. The bag should be labeled with a GM part number; final sample name (cannot be changed afterwards), manufacturing date, and date of sampling. The part itself will not be marked with any type of labeling.

Note: Samples without proper identification labels will not be analyzed until written clarification is received.

3.3.2 Cured Paints or Cured Coatings. Paints are sprayed on silicone-free aluminum foil of 30 µm maximum foil thickness, the paint process guidelines of the paint supplier including coating, thickness and drying requirements shall be followed. For multilayer paints, the thickness of the individual layers of the film shall be the same as the thickness on the painted part. If necessary each layer will be analyzed separately. The supplier process guidelines including film thickness and curing requirements must be documented. After curing, the samples are vented at room temperature for seven (7) days. After venting, packaging is prepared as described in paragraph 3.3.1.

3.3.3 After receipt in the laboratory, the packaged samples shall be refrigerated at $\leq 0^{\circ}\text{C}$ until analysis.

3.3.4 Prior to analysis, the sample should be taken from the refrigerator, unwrapped and stored open at $(+23 \pm 5)^{\circ}\text{C}$ room temperature for 2 h to prevent condensation. From manufacturing to analysis, it should be assured that the sample has been stored open at room temperature for no more than seven (7) days.

3.4 Test Time.

Calendar time: 1 days

Test hours: 8 h

Coordination hours: 4 h

3.5 Test Required Information.

3.5.1 Parts. Part number, drawing and circa dimensions of the automotive material are supplied.

3.5.2 For all samples, manufacturing date and sampling date must be provided.

3.6 Personnel/Skills. Proper training is required to operate the instrumentation within this procedure. Professional degrees in chemistry, chemical engineering, or material sciences are preferred.

4 Procedure

4.1 Preparation. Each sample test requires two (2) conditioned Solid Absorbent Media Glass Thermodesorption Tubes and one automotive material sample. The material sample shall have the same size in diameter than the respective micro-scale chamber vessel. The 1st Solid Absorbent Media Glass Thermodesorption Tubes is inserted and secured into the connector on the lid of a micro-scale chamber that has been previously equilibrated at $+90^{\circ}\text{C}$ under 50 ml/minute ($\pm 1\%$) flow of air. Samples are loaded within the equilibrated micro-scale chamber immediately after inserting the tube for 30 minutes.

4.1.1 The 1st Solid Absorbent Media Glass Thermodesorption Tubes is sampled for 30 minutes then removed and capped for further preparation. This 1st tube will be analyzed for the VOC value. Immediately thereafter, the second tube is inserted into the lid of the same micro-scale chamber and the temperature elevated and equilibrated to +120 °C under 50 ml/minute ($\pm 1\%$) flow of sampling gas, as described in paragraph 3.2.10.

4.1.2 The 2nd Solid Absorbent Media Glass Thermodesorption Tubes is sampled for 60 minutes then removed and capped for further preparation. The 2nd Solid Absorbent Media Glass Thermodesorption Tubes will be analyzed for SVOC/FOG value.

4.1.3 Sampled tubes shall be immediately sealed using clean long-term field storage caps and stored in a non-VOC emitting refrigerator at $\leq 0^\circ\text{C}$ for no longer than 7 days before analysis.

- **1st Tube:** Document tube number for VOC analysis run.
- **2nd Tube:** Document tube number for SVOC/FOG analysis run.

4.1.4 For Multi-Layer Sandwich Samples. With relatively thick individual layers, i.e., $> 0.5\text{ mm}$, the material in each layer should be analyzed separately. This helps to allocate the emitted substances to the respective individual materials.

4.1.5 For Paint Films. The paint sample sprayed on alumina foil shall be cut to the same diameter as the Micro-Scale Chamber. The weight of the aluminum foil shall be subtracted.

4.1.6 Samples with High Water Content. For example, leather. Unless care is taken to selectively eliminate water during the analytical thermal desorption process (see analytical conditions below), water released by large wet samples could lead to a freezing of the cold trap causing incorrect values or even causing the run to be aborted. This can be prevented by reducing the weight of these types of samples or by setting the analytical conditions to selectively eliminate water during the trapping/focusing step of TD operation.

4.1.7 Olfactory Sniff-Port for Odor Identification. Using an empty Glass Thermal Desorption Tube (GTDT) without a frit (see Appendix A, Table A1), the micro-scale chamber can also be used in synergy as an olfactory sniff-port for odor identification. Air or carrier gas can be selected so atmospheres are specific to a particular emission. The micro-scale chamber allows flow rate and temperature adjustments so conditions are specific to a particular odor emission. The micro-scale chamber is also capable of simulating real world environmental conditions and replicating field sample collection. Under these identical conditions, particular odor emissions can be captured and later identified.

4.2 Conditions.

4.2.1 Environmental Conditions. Not applicable.

4.2.2 Test Conditions. Deviations from the requirements of this standard shall have been agreed upon. Such requirements shall be specified on component drawings, test certificates, reports, etc.

4.2.2.1 Sample Preparation. The sampled Solid Absorbent Media Glass Thermodesorption Tubes (see paragraph 4.1.3) are allowed to equilibrate with ambient temperature. The long term (field) storage caps are then replaced with clean analytical caps (see paragraph 3.2.12).

4.2.2.2 Introduction of Internal Standard calibration mix to Sampled Tubes. Remove the analytical caps and inoculate the sampled tubes (paragraphs 4.1.1 and 4.1.2) with 4 μl of Internal Standard Calibration Mix (see paragraph 3.2.15.3) using a 10 μl syringe and the CSLR with the flow of pure N_2 gas set to (80 to 100) ml/minute. The injection rate should be a constant 2 μl /minute. In the case of Tenax/CG5TD tubes, inject 0.5 μl volume using a precision 1 μl syringe and use a higher concentration of Internal Standard Calibration Mix accordingly. The original analytical caps are then replaced for immediate analysis.

4.2.2.3 Spiked Standard(s). The standard solutions ($\mu\text{g}/\mu\text{l}$) of target compounds are spiked on separate, blank preconditioned Solid Absorbent Media Glass Thermodesorption Tubes at $(+25 \pm 1)^\circ\text{C}$. This is accomplished by using a precision syringe (1 to 10) μl and the CSLR with the flow of pure N_2 gas set at (80 to 100) ml/minute. The N_2 flow rate is maintained for ≈ 3 minutes (i.e. until a total N_2 volume of (240 to 300) ml has passed through the tube).

4.2.2.4 n-Alkane and Aromatics Standard(s). Use the analytical parameters specified in paragraphs 4.4 thru 4.6. Perform a TD-GC/MS analysis on a Solid Absorbent Media Glass Thermodesorption Tubes spiked with 1 µl of n-alkane standard (see paragraph 3.2.15.4) or 1 µl aromatics standard (see paragraph 3.2.15.5) in SCAN mode with VOC and SVOC conditions. The retention times (RTs) of the components in these mixtures are suitable as reference points to determine the RT indices of unknown substances (see Appendix G, Figures G4 and G5). This is used for an additional check of MSD identification during test runs. Keep a running record of the individual components' response factors and RTs as a method monitor.

4.2.2.5 Check Standard(s). Using the same analytical conditions, perform a TD-GC/MS run on an Solid Absorbent Media Glass Thermodesorption Tubes spiked with 4 µl of check standard (see paragraph 3.2.15.2), or (0.5 to 1) µl volume in the case of Tenax/CG5TD tubes, and with SCAN mode set to acquire data for both target VOC and SVOC compounds. The RTs of the components in this mixture are suitable as reference points to determine the RT indices of unknown substances. This is used as an additional check of the MSD identification during test runs. Keep a running record of the individual components' response factors and RTs as a method monitor.

4.2.2.6 Testing the TD-GC/MSD System. The performance of the analytical system is tested by analyzing a check standard during the analytical sequence. A calibration curve is prepared by spiking a number of Solid Absorbent Media Glass Thermodesorption Tubes with different concentrations of the check standard solution (range (0.01 to 6) µg per substance per injection). The calibration will be performed regularly. Analysis of the check standard, calibration standard and ISCM shall be implemented for each test series. Other standards listed in paragraph 3.2.15.1 may also be used to evaluate system performance and for RT locking confirmation.

4.2.2.6.1 The check standard specified in this method contains non-polar, polar, basic, and acid components that will display noticeable peak tailing even with low adsorption effects. Peaks occurring in close succession can be used to check the resolution (separation efficiency) of the GC capillary column.

4.2.2.6.2 All substances in the check standard must be clearly identified from the mass spectral libraries during the search routines.

4.2.2.7 The TD system shall also be checked for possible memory effects by performing a "blank" run with a clean conditioned Solid Absorbent Media Glass Thermodesorption Tubes. at least once before every sample series.

4.2.2.8 The performance of the MSD is verified by means of mass and sensitivity tuning and the specification required by the manufacturer must be achieved. An air/water check must be performed to verify the integrity of the entire TD-GC/MSD system.

4.3 Instructions. Prior to the analysis of automotive material emissions, a calibration shall be performed and the following limits of detection must be reached at a Signal-to-Noise (S/N) ratio of 3:1 or above.

- Toluene < 0.04 µg
- n-Hexadecane (n-C16) < 0.05 µg
- n-Eicosane (n-C20) < 0.06 µg (at VOC conditions)
- n-Dotriacontane (n-C32) < 0.2 µg (at SVOC conditions)

4.3.1 The recovery of the individual substances in the mandatory standards should be between (80 to 120) % under VOC conditions with the exception of toluene which should be between (90 to 110) %.

4.3.2 For a complete test, the following measurements shall be accomplished under the specified analytical conditions (paragraphs 4.3.4 thru 4.3.6):

4.3.2.1 System blank to check the cleanliness and background of the TD-GC/MSD system.

4.3.2.2 Check standard mix spiked on an Solid Absorbent Media Glass Thermodesorption Tubes for testing the performance of the TD-GC/MSD system.

4.3.2.3 As soon as the Solid Absorbent Media Glass Thermodesorption Tubes from the first (VOC) step (paragraph 4.1.1) and the second (SVOC/FOG) step (paragraph 4.1.2) have been spiked with ISCM (paragraph 4.2.2.2), they shall be analyzed.

4.3.3 After every TD-GC/MSD system maintenance procedure (e.g., cleaning, changing the column, etc.), the Grob test mix, n-alkane standard and aromatics standard shall be analyzed to determine the RT of the analytes for quality assurance.

4.3.4 TD Parameters. The thermal desorber and its integrated electrically-cooled focusing device (trap) must operate in one fully-automated two-stage desorption process.

4.3.4.1 Solid Absorbent Media Glass Thermodesorption Tube Pre-Desorption. The minimum carrier pressure shall be set at 34.5 kPa (5.0 psi). During pre-desorption, Solid Absorbent Media Glass Thermodesorption Tubes are dry purged for 1 minute with 20 ml/minute He flowing through the tube in the sampling direction. This is followed by prepurging the tube in the desorption direction for 1 minute with 20 ml/minute He flow passing through the tube and to the trap.

4.3.4.2 Solid Absorbent Media Glass Thermodesorption Tubes. During the primary (tube) desorption stage, the Solid Absorbent Media Glass Thermodesorption Tubes is heated to +320 °C for 20 minutes using 50 ml/minute He flow rate and with the focusing trap in line. The VOCs desorbed from the 1st Tube or the SVOC/FOGs desorbed from 2nd Tube are transferred to the focusing trap under splitless conditions and are captured and contained by the sorbents in the trap which is maintained at -5 °C.

Note that trap temperatures up to +30 °C may be used to minimize water retention if the automotive material under test has high water content. Any such deviation from standard conditions must be agreed in advance by the sample submitter.

4.3.4.3 Secondary (Trap) Desorption. The VOCs and SVOCs desorbed from the Solid Absorbent Media Glass Thermodesorption Tubes were collected on the focusing trap at -5°C under splitless conditions (i.e. All the compounds desorbed from Solid Absorbent Media Glass Thermodesorption Tubes were transferred to the focusing trap). Prior to secondary desorption the focusing trap is exposed to a 1.0 minutes pre-trap fire purge with GC carrier gas flowing through the trap in the desorption direction. The trap is then heated to +375 °C (see note below) using the maximum rate and held for at this temperature for 7.5 minutes under a 38 ml/minute He flow rate, while maintaining a (30:1) split ratio. The temperature for the transfer line, and heated valve are kept at +200 °C.

Note: A trap packed with robust carbon sorbents is normally recommended, but an alternative trap packed with Tenax TA backed up by Carbograph 5TD may also be used (see Appendix A, Table A1) if reactive compounds are of interest. The trap desorption temperature shall not be taken > +335 °C, if the alternative trap is used.

Other equipment can also be used when compatibility has been shown.

4.3.5 GC/MSD Conditions. A constant 1.3 ml/minute flow of He is set through the GC column. The GC oven is allowed to equilibrate for ≥ 0.5 minutes at +40 °C before trap desorption. Trap desorption automatically initiates the GC run. At the start of the run, the GC oven is held at +40 °C for an additional 2 minutes. The GC oven is then ramped at 3 °C/minute to +92 °C for 0 minutes. This is followed by a GC oven ramp of 5 °C/minute to +160 °C for 0 minutes. Finally, the GC oven is ramped at 10 °C/minute to +310 °C and held for 15.07 minutes. The GC cycle time is set at 0.0 minutes and total GC run time is 63.0 minutes. The data is acquired using positive electron ionization (EI+) mass spectrometry. The MSD transfer arm is held at +300 °C, the source at +230 °C and the quadrupoles at +150 °C. The scan range is set from (33 to 800) u (unified atomic mass unit).

4.3.6 The VOC value is measured from the 1st Tube and the SVOC/FOG value is measured from the 2nd Tube.

4.3.7 A quantitative determination of the emissions expressed as ppm can be obtained by calibration with reference substances. Deuterated toluene-D8 is used as a reference substance for VOC and deuterated n-hexadecane-D34 is used as reference substance for the SVOC/FOG value. Unknown substances can be identified from the mass spectra and the RT.

5 Data

5.1 Calculations.

5.1.1 Qualitative Analysis. During this procedure, identification of individual VOC and SVOC components is accomplished by operating the mass filter in SCAN mode. Several target compounds may be quantitatively determined with an extracted ion chromatogram, e.g., when there are overlapping peaks, or when full scan identification is otherwise compromised. Usually the molecule's base ion (highest m/z abundance) or the molecule's parent ion (if available: $m/z = M^+$) serves as the target ion (quantitative m/z). A known corresponding fragment ion serves as a qualifier ion (m/z). With this information and each analyte RT, confirmation of component identity is possible. If a substance cannot be clearly identified, a possible identification (marked with a question mark) or a reference to the substance class can be given, provided that the appropriate reference points (e.g., typical mass fragments) permit such conclusions to be drawn. See Appendix D for notation examples.

5.1.2 Quantitative Analysis.

5.1.2.1 Total Amount of VOCs and SVOCs. The total area of all of the peaks occurring in the chromatogram (excluding artifacts) is determined first. The integration parameters for the Gas Chromatography / Mass Spectrometry (GC/MS) software shall be set so that peaks with a concentration of ≥ 1 ppm are detected reliably. For the total amount of VOCs and SVOCs, all peaks in the corresponding chromatogram have to be integrated, including peaks with a concentration < 1 ppm.

5.1.2.2 If the chromatogram includes so called "oil-mountains", consisting of unresolved species, these are integrated as one peak, in which the baseline is laid from the start to the end of the "oil-mountain". If clearly identified peaks of one or more individual substances occur in this zone, they must be integrated and specified separately.

5.1.2.3 Example calculations for using liquid phase ISCMs from a solvent are found in Appendix E1.

5.1.2.4 Example calculations for using gas phase ISCMs from a pressurized cylinder are found in Appendix E2.

5.1.2.5 The total amount of VOCs is calculated as deuterated toluene-D8 equivalent and the total amount of SVOCs as deuterated n-hexadecane-D34 equivalent. The comparison of the peak areas to the peak area of the individual calibration standard is calculated. For the VOC, only the peak areas from n-pentane (n-C5) to n-eicosane (n-C20) will be calculated. For the SVOC, only the peak areas from n-hexadecane (n-C16) to n-dotriacontane (n-C32) will be calculated.

5.1.3 Individual Amounts of VOCs and SVOCs.

5.1.3.1 Each analyte with a concentration >1 ppm shall be identified and its concentration calculated. At a minimum, the concentrations of the analyte(s) specified in a relevant regulation must be calculated.

5.1.3.2 The individual VOCs and SVOCs are quantified by comparing the SCAN quantitative ion peak area amounts to those of known standard solutions (ISCM).

5.1.3.3 If there are individual resolved peaks in the "oil-mountains," they shall be integrated and evaluated manually.

5.1.4 Calculated Result Disclaimer. The calculated quantitative results obtained are from individual organic compounds thermally stripped and detected under these particular methods and corresponding sampling conditions. This quantitative method should not be compared to other semi-quantitative or quantitative methods.

5.2 Interpretation of Results. Some VOCs and SVOCs are frequently cited as odorous compounds and may require additional testing in accordance with GMW3205.

5.3 Test Documentation. The results are displayed in a report. See Appendix F for details. In addition, the chromatograms for the VOC/SVOC determination and associated substance lists shall be attached to the results report. The full set of analytical data must be supplied electronically. It should include:

5.3.1 Microsoft Excel files containing the detailed results of the VOC/SVOC analysis shall be supplied together with a copy of the relevant total ion chromatogram for each sample.

5.3.2 Information on the TD-GC/MSD and the Micro-Scale Chamber instrumentation used for these measurements shall be supplied.