GM	WORLDWIDE ENGINEERING STANDARDS	Test Procedure	GMW15635

Determination of Aldehyde and Ketone Emissions from Interior Materials

1 Introduction

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 Test Procedure describes a measuring process for determining the aldehyde and ketone emissions from vehicle interior materials.

1.1.1 For this test, test pieces are fastened above deionized water in a sealed bottle and stored in an oven for a specific period. Afterwards, the bottles are cooled and the aldehydes and ketones absorbed in the demineralized water are determined.

1.1.2 The concentrations of aldehydes and ketones are determined by 2,4-dinitrophenylhydrazine (DNPH) derivatization followed by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection.

1.1.3 The quantity of aldehydes and ketones are ascertained as a proportion of the dry weight in µg/g (micrograms per gram) of the test piece.

1.2 Applicability. This Test Procedure is applicable to materials and components used in vehicle interiors that may contain carbonyls (e.g., polyoxymethylene, parts containing wood, natural or laminated fibers, leather, artificial leather, adhesives, sealants, polyurethane, cotton fleece, joint fabric fleece, phenol/formaldehyde resins, etc.) The results will provide complementary information to GMW14444.

1.3 Remarks. The determination of aldehydes and ketones in vehicle materials and components is essential to meet customer requirements and regulatory requirements in some global markets.

2 References

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

2.1 External Standards/Specifications.

DIN 55531	ISO 16979	ISO/IEC 17025

2.2 GM Standards/Specifications.

GMW3059	GMW14444	GMW16853

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.

3.2 Equipment.

3.2.1 Test Apparatus.

3.2.1.1 Precision weighing device with scale graduation ±1 mg.

3.2.1.2 Air circulating oven capable of maintaining the required temperature within ±3 °C.

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3.2.1.3 One liter (1 L) polyethylene or glass bottle with an integrated hook implemented within the lid (see Figure 1).

3.2.1.4 High performance liquid chromatograph with binary pump, solvent degasser, heated column compartment, autosampler (optional), variable wavelength or diode array detector (DAD), and personal computer (PC)-based controller and data evaluation. System must be capable of gradient operation to 3.5 mL/minute and 350 bar (maximum ceiling pressure capacity), and UV detection at (360 to 365) nm (e.g., Agilent 1100 Series and Agilent ChemStation for system control and data evaluation, or comparable equipment).

3.2.1.5 HPLC column capable of separating and resolving the 2,4-dinitrophenylhydrazine derivatives of the specified aldehydes/ketones using gradient elution conditions. The separation of the DNPH derivatives depends on the HPLC column used. See Appendices A, B and C for example conditions.

3.2.2 Reagents.

3.2.2.1 Acetonitrile. HPLC grade.

3.2.2.2 Tetrahydrofuran. HPLC grade containing no antioxidant additive.

3.2.2.3 Water. HPLC grade.

3.2.2.4 Phosphoric Acid. Concentrated, reagent grade.

3.2.2.5 Derivatizing Reagent.

3.2.2.5.1 DNPH Solution. ≈ 250 mg of 2,4-dinitrophenylhydrazine dissolved in 50 mL of acetonitrile.

3.2.2.6 Calibration standards containing the components of interest already derivatized in appropriate concentration range, typically (0.1 to 15) µg/mL in acetonitrile.

3.2.2.6.1 Calibration standards, among others, are available from, e.g.:

Supelco: TO11/IP-6-A Carbonyl-DNPH-Mix, 15 components in acetonitrile, 15 µg/mL of each component (aldehyde equivalent), Sigma-Aldrich (Supelco), 47285-U.

3.3 Test Vehicle/Test Piece.

3.3.1 Test Sample Handling and Preparation. If possible, the test sample shall be taken out of the finished part of the material or the component.

3.3.1.1 In exceptional cases (e.g., if only one material of a component is to be tested, or if the complete component is not available), tests can be conducted on specially prepared parts.

3.3.1.2 Test pieces should be obtained from suppliers as soon as possible after production. The production date and sampling should be documented. Care should be taken to prevent material from thermal desorption before analysis (package, refrigerate).

Open Storage before testing shall be done according to GMW16853, "Preparation" section.

3.3.1.3 A representative material sample (smaller samples) should be cut out from large components to a size of 10 cm x 15 cm. The sample shall be handled with protective gloves and the cutting shall be done with an oil-free tool such as a disposable razor blade. The sample should be packed by aluminum composite films (e.g., VACUPAC or any other material according to DIN 55531) prior to shipping. In the exceptional case, the sample can be packed twice into thick silicone-free aluminum foil (pre-cleaned with n-pentane). The edges should be folded multiple times to prevent contamination from outside and to ensure an air-tight packing. The aluminum foil packed sample should then be packed additionally and placed in a polyethylene bag. The bag shall be labeled with sample identification (ID) number; final sample name (cannot be changed or altered afterwards), production date and sampling date shall be recorded in Appendix D, Data Sheet D1. If this information is missing, the sample should not be analyzed.

3.3.2 Removing Test Pieces. The test pieces shall be removed from suitable and representative areas spread evenly across the width of the part. They shall be taken 50 mm from the edge of the part.

3.3.3 The size of each test piece is nominally 40 mm x 100 mm x thickness. If the size of the molded part is smaller or the distance of 50 mm to the edge of the part is not possible, this test may also be performed using smaller test pieces. Care must be taken to ensure that all the edges of test pieces are freshly cut. To fasten the test piece, drill a hole corresponding to the diameter of the hook near its center and 10 mm from the upper edge.

3.3.3.1 For small parts, at least 3 samples must be taken from each part. Two (2) parts are used for determining aldehydes/ketones and 1 part is used to determine the moisture content.

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3.3.4 If the testing of the samples cannot take place immediately, they shall be stored in cool, air-tight conditions with a minimal air volume. Storage time should not exceed 5 days.

3.4 Test Time. Sampling of the test pieces requires 3 h in the oven and 1 h of cool down. Sampling is best conducted for several materials in parallel in individual bottles in the oven. The determination of aldehydes/ketones by derivatization and HPLC is conducted serially with each sample requiring (30 ± 5) minutes.

3.5 Test Required Information. Material component information such as vendor name, production date, pretreatment, etc., should be recorded in Appendix D, Data Sheet D1.

3.6 Personnel/Skills. Proper training is required to process the samples and operate the HPLC instrument. A professional degree in chemistry, chemical engineering or the health sciences, and familiarity with the operation of HPLC instrumentation is required.

4 Procedure

4.1 Preparation.

4.1.1 Determining the Moisture Content. The moisture content is determined in accordance with ISO 16979.

4.1.1.1 The moisture content H% of the part in percent is derived from the following formula:

$H (\%) = 100\% \times ((m_1/m_0) - 1)$

Where:

m_1 = mass of the test piece (g) before drying

m_0 = mass of the test piece (g) after drying

4.1.1.2 The mass constancy is obtained if the results of two successive tests do not deviate more than 1% from one another.

4.2 Conditions.

4.2.1 Environmental Conditions.

4.2.1.1 Instrument. Set up the HPLC instrument with the correct conditions, for example see Appendices A, B or C.

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.

4.3 Instructions.

4.3.1 Determining the Release of Aldehydes and Ketones from the Test Samples. Prior to the chemical analysis, the samples are weighed on the analytical balance to ± 0.01 g. 50 mL aliquots of deionized water are pipetted into each of a clean 1 L polyethylene or glass bottle. As soon as the test sample has been attached to the hook, the bottle is sealed and stored for (180 ± 15) minutes in a heated cabinet at (+60 ± 3) °C. Additionally a blank test shall be run without test piece present.

4.3.1.1 When the test time is completed, the containers are removed from the heating cabinet and left to stand at (+23 ± 5) °C for (60 ± 10) minutes. The samples are removed from the bottles.

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