FLTM BZ 157-01

DETERMINATION OF ORGANIC EMISSIONS FROM NON-METALLIC MATERIALS IN VEHICLE INTERIORS BY HEADSPACE GAS CHROMATOGRAPHY

Application

This test method is used to determine emissions of organic compounds (TVOC = total volatile organic compounds) which are given off from non-metallic materials in the immediate vicinity of or inside vehicle interiors.

The sum of individual substances according to a gas chromatography analysis and detection with a flame ionization detector (GC/FID) or mass selective detector (GC/MS) is used as a measure of the total emission of a material.

It is preferred to use GC/MS as this technology delivers more accurate and more reliable results. Usage of GC/FID must be agreed by Ford Materials Engineering prior to testing.

The test is performed using Head-Space technique after tempering at 120 °C.

The relevant sampling points, requirements and special provisions relating to the execution of the test can be found in the appropriate specifications and drawings, etc.

If components are made up of a number of different materials, separate tests may be required for each individual material.

Apparatus and Materials Required

Acetone p.a.

Butanol p.a.

Gas Chromatograph for capillary column operation with Head-Space test vessel, flame ionization detector (FID) or mass selective detector (MSD) and computer/integrator.

WCOT capillary column with a separation phase of 100 % polyethylene glycol (so-called wax type, e.g. DB wax, Carbowax, etc.), 0.25 mm internal diameter, 0.25 um film thickness and 30 m length.

Analytical balance with an accuracy of 0.1 mg

Micro liter syringe, 5 µL

Beakers

Graduated flasks

Date	Action	Revisions	Rev. 1
2011 03 11	Revised	Added preferred use of GC/MSS.	S.Riewer, FOE
2009 01 03	Activated		S.Riewer, FOE

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Conditioning and Test Conditions

Transport and storage of the samples shall take place in an aluminium-clad polyethylene bag.

Sampling shall take place immediately after delivery or in a condition equivalent to that status. The dates of delivery and sampling shall be recorded.

Conditioning of the sample is not normally required. One exception is natural materials (cotton, wood, leather, wool). These materials must be divided up into small pieces, dried for 24 h over calcium chloride ($CaCl_2$) and then weighed. The sample must be taken at the stipulated point over the entire section of the component. It must be broken (without causing it to heat up) into pieces weighing between 10 mg and 25 mg. Other types of sample preparation may be indicated, if necessary, on the drawing or in the Technical Regulation (TR). This must be stated in the report of the results.

The test quantity to be weighed depends on the size of the Head-Space vessel, which shall have a minimum volume of 5 ml. For a 10 ml Head-Space vessel, 1,000 g ± 0.001 g (i.e. a maximum weighing error of 0.1 %) of the sample shall be weighed. Metal parts shall be removed before weighing.

Organic substances such as paint and adhesive, etc., which are firmly attached to metal parts shall be removed by mechanical means and then weighed.

The sample is weighed in the Head-Space vessel (at least three Head-Space vessels per sample). The Head-Space vessel is then sealed in a gas-tight fashion with the help of a septum, with the Teflon coating on the side facing the interior of the Head-Space vessel.

Method

Immediately before the measurement, the Head-Space vial is tempered to 120 + 1 °C. The test period in the Head-Space is 5 h ± 5 min at 120 ± 1 °C, and the analysis shall take place immediately thereafter. At least three samples of each material shall be analyzed.

The blind value is determined by means of the mean value from at least three measurements with empty Head-Space vessels.

Metering shall take place in an identical and reproducible fashion in conjunction with all analyses of samples, blind values and calibration solutions.

The column shall be burned off once a week by heating the column to its maximum operating temperature for 15 minutes.

GC oven temperature Program:

3 min isothermal at 50 °C

Heating to 200 °C at a rate of 12 °C/min

4 min isothermal at 200 °C

Injector temperature: 200 °C

Detector temperature: 250 °C

Split ratio: approx. 1 : 20

Carrier gas: helium

Mean carrier gas velocity: approx, 22 - 27 cm/s

Note: The substance 2.6-di-tertiary-butyl-methylphenol (BHT) shall exhibit a retention time of less than 16 minutes.

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