Determination of Formaldehyde from Vehicle Interior with Modified Flask Method VDA 275

Moulded composites and fleeces for vehicles
Determination of formaldehyde release
Test procedure called modified flask method

1. Purpose and application

The present test method describes a necessary procedure to the method of the formaldehyde release from the vehicle interior.
The procedure is applied to the check of the equability of the production of form parts. It can be applied restrictedly for the material development and to the receiving control.

2. Reference to other testing methods

VDA 276- Measuring procedure of formaldehyde, ammonia and phenols release form the vehicle interior with the use of 1 m^2 chamber.

3. Name

Name of the procedure of the formaldehyde release method. VDA 275.

4. Principle

In the test, samples of certain masses and dimension are fastened in a closed 1 litred Polyethylene flask with distilled water and are stored in steady temperature for a defined time. The bottles are then cooled and the dissolved formaldehyde in the distilled water is determined. The result is expressed in weight (mg/kg).

5. Reagents

For the analysis, reagents of analytic grade and distilled or demineralized water must be used. The formaldehyde content is determined photometry after the Acetylaceton method. Nevertheless, other procedures should be allowed if they correspond in specifications to the reference procedure for the analysis

5.1 Acetylaceton p.a.
5.2 Ammonium acetate p.a.
Remark: Commercially pre-prepared solutions can be used when the identical result is achieved.

6. Test equipment

6.1 Balance with range $\pm$ 1 mg
6.2 Well ventilated warm chamber which is able to hold a temperature from 103$\pm$2ºC.
6.3 Circulating air warm chamber which is able to hold a temperature from 60$\pm$2ºC
6.4 Spectrophotometer
6.5 Test apparatus
---1 litred Polyethylene flask with a lid (see appendix picture 1)
---Hooks from high-grade steel with poetries, used in the lid of the test bottle

6.6 Lab. Device
--Precision burette 50ml, calibrated at 20ºC
--2 Measuring cylinder, 1000ml, calibrated at 20ºC
--6 Measuring cylinder, 100ml, calibrated at 20ºC
--Pipette, 5ml, calibrated at 20ºC
--Pipette, 10ml, calibrated at 20ºC
--Pipette, 15ml, calibrated at 20ºC
--Pipette, 20ml, calibrated at 20ºC
--Pipette, 25ml, calibrated at 20ºC
--Pipette, 50ml, calibrated at 20ºC
--Pipette, 100ml, calibrated at 20ºC
--Conical flask, 250ml
--Bottle, 50ml with stop
--Watch glass with diameter of 120mm
--Desiccator
--Cuvette with suitable thickness for the spectrophotometer
--Stop watch

7. Specimen

7.1.1 Specimen: The test bodies should be taken from suitable and representative places of the vehicle interior parts

7.1.2 The size of the test bodies in each case should be 40 mms x 100 mms x thickness. At least 6 test bodies are taken, of it 5 parts for the test and 1 for the moisture content test

8. Realization of the check

8.1 Number of the check: These are professional method to be carried out

8.2 Determination of the moisture content

The moisture content is determined with DIN EN 322

8.3 Regulation of the formaldehyde release

The test bodies become precise-weighed out before the beginning of the analysis on the analysis scales on 0.01 g. In each of the Polyethylene flask become 50 ml distilled water is pipetted.

For the connection, the test bodies will provide 10 mms of the upper edge concentric with 1 to 2 mms of drilling for the purpose of connection in the hanging hook.

After fixing of the test bodies in the hook (see appendix: picture 1) the vessel is closed and is kept more than 3 hours in the warm chamber (6.3) with a steady temperature at 60ºC.

At the end of the test time the vessels from the warm chamber are taken. After 60 minute stabilizing time at ambient temperature the test bodies from the test bottle are removed.

8.4 The control test

The attempt is carried out without test body in parallel.

8.5 Regulation of the formaldehyde concentration in the absorbent solution

The formaldehyde content of the absorbent solution is detected by photometry after the Acetylaceton method.

8.5.1 The principle

The chemical reaction (see appendix on the so-called Hantzschen: picture 2) with which dissolved formaldehyde reacts in solution react with Ammonia and Acetylaceton to form Diacetyldihydrolutidin (DDL). The absorption maximum of DDL is 412 nm. The reaction is specific for formaldehyde.

8.5.2 Reagent

8.5.2.1 Acetylaceton solution

4ml of Acetylaceton are dispensed in a 1000 ml to measuring cylinder and is made up to 1000 ml with distilled water. The solution air-tighted and can be used for 4 weeks if kept away from light.

8.5.2.2 Ammonium acetate solution

200 g of ammonium acetate is dispensed in a 1000ml measuring cylinder and make up to 1000ml with distilled water.

8.5.3 Reaction

10ml of the absorbent solution (8.3) are taken with a pipette and are moved in a 50ml bottle with 10 ml Acetylaceton solution (8.5.2.1) and 10 ml ammonium acetate-solution (8.5.2.2). The bottle is closed, shaken and warmed up for 15 minutes in a waterbath at 40ºC. The greenish-yellow solution is then cooled at ambient temperature, protected from the sunlight (about one hour). The optical density of this solution is determined with a wavelength of 412 nm by distilled water with a Spectrophotometer (6.4). A control is determined in parallel with distilled water and is taken into consideration with the calculation of the bottles value (9.2).

Remark: After every test, the PE flasks are cleaned in a lab washer. Should this not be sufficient, it is advisable that the flasks are kept at 70ºC openly in a circulating air warm chamber for approximately 16 h.

8.5.4 Calibration curve

The calibration curve (see appendix: pictures 3 and 4) is constructed by means of a formaldehyde standard solution whose concentration was determined by titration with iodine. The calibration curve must be checked at least once per week.

8.5.4.1 Formaldehyde-standard solutions reagent solutions

Iodine $c(I_2)=0.05$ mol/l

Sodium thiosulfate $c(Na_2S_2O_3)=0.1$ mol/l

Sodium hydroxide $c(NaOH) = 1$mol/l

Sulfuric acid $c(H_2SO_4)= 1$ mol/l

Indicator solution 1% m/m

The Titer of the solutions must be checked before use.

About 1 g of formaldehyde solution (concentration of 35 to 40%) are solved in a 1000 ml measuring cylinder with distilled water and are filled in up to mark. The exact formaldehyde concentration is determined as follows:

20ml of the formaldehyde-standard solution are mixed with 25ml iodine solution and 10ml mixed sodium hydroxide solution. After 15 minute stabilizing time under light, 15ml sulfuric acid is added to the mixture. The excess iodine reacts with the thiosulfate solution. At the end of the titration some drops of indicator

solution are added. The control is carried out with 20ml to distilled water in parallel.
The formaldehyde content is calculated with the following formula:
c (HCHO) = (Vo-V)x15 [$c(Na_2S_2O_3)$] x 1000/20
Where:
c (HCHO): Formaldehyde concentration in mg/l
Vo: Volumes of the thiosulfate solution for the control in ml
V: Volumes in thiosulfate solution in ml
$c(Na_2S_2O_3)$: Concentration of the thiosulfate solution in mol/l
Remark: 1ml 0.1 mol/l thiosulfate solution corresponds to 1 ml 0.05 mol/l of iodine solution and 1.5 mg of formaldehyde
8.5.4.2 Formaldehyde calibration solution (with thickness of the cuvette: 1 cm)
From the formaldehyde standard solution, determined in 8.5.4.1, becomes a volume which contains 15 mg of formaldehyde from a microburette in a 1000ml measuring cylinder and filled with distilled water up to mark filled in. 1 ml of this solution contains 15 µg formaldehyde.
8.5.4.3 Calibration curve
0, 5, 10, 20, 50 or 100 ml are filled in by the formaldehyde calibration solution (8.5.4.2) in a 100ml measuring cylinder and with distilled water up to mark. 10ml of solution are photometrically analyzed with the same procedure, as that of 8.5.3. The optical densities are applied against the formaldehyde concentration (between 0 and 15 µg/ml) on standard curve. The upward gradient is determined graphically or is calculated.
8.5.4.4 Formaldehyde calibration solution (with thickness of the cuvette: 5 cm)
From the formaldehyde-standard solution, determines under 8.5.4.1, becomes a volume which contains 3 mg of formaldehyde with a microburette in a 1000ml measuring cylinder and make up to mark with distilled water. 1ml of this solution contains 3 µg of formaldehyde.
8.5.4.5 Calibration curve
0, 5, 10, 20, 50 or 100 are filled in by the formaldehyde calibration solution (8.5.4.4) ml in a 100ml measuring cylinder and with distilled water up to mark. 10ml of solution is photometrically analyzed with the same procedure, as that of 8.5.3. The optical densities are applied against the formaldehyde concentrations (between 0 and 3 µg/ml) on standard curve. The upward gradient is determined graphically or is calculated.

9. Calculation of the results

9.1 Moist content

The moist content H (in masses % m/m) of the form part arises from the following formula:

$$H = \frac{m_1 - m_0}{m_0} \times 100$$

Besides, is:
m_1: the masses of the test bodies before the drying in gram
m_0: the masses of the test bodies after the drying in gram

9.2 Formaldehyde release with flask method

The whole amount of dissolved formaldehyde becomes from the upward gradient factor of the photo-metrical regulation and the whole volume of the absorbent liquid (here: 10ml) investigates. This Formaldehyde is calculated with the weight corrected on dry weight of the test body in mg / kg.

Calculation of the formaldehyde delivery in mg / kg:

$$\frac{(As - A_B) \times f \times V \times (100+H)}{m \times 1000} \times F = \text{mg HCHO / kg dry test material}$$

As: the optical density of the analyzed solution

A_B: the optical density of the analysis with distilled water

f: the upward gradient factor of the calibration function (in µg/ml)

m: the masses of the test body in g

H: the moisture content of the test material in percent

V: the volume of the absorbent solution (50 ml)

atro: absolutely dry test material

F: Factor to the calculation of the result of analysis in kg [mg / kg]; F=10

10. Test report

In the test report the following information should be given with reference to this test method, if necessary:

- Origin of the test material
- Place, position and state of the material at the time of the sampling, in particular the humidity
-date and production of the material
-date of the sampling
-date of the test performed
-moisture content (%) at the time of the check (according to 8.2)
-formaldehyde release after the flask method (mg of formaldehyde / kg dry test material)
-description of other details [1)]

[1)] Report on all occurrences which are not according to this test regulation (withdrawal of the test bodies, conditioning etc.)