Determination of Organic Emission of Non-metallic materials from vehicles Interior VDA 277

Contents
1. General
2. Test preparations
3. Testing sets and conditions
4. Attempt realization
5. Calibration
6. Evaluation

1. General

1.1 Purposes

According to this test method, the organic emission from non-metallic materials is determined with direct or indirect influence for the passenger's cell of automobiles stand.

It measure the emission potential of a material, the sum of all release values of the emitted substances with gaschromatograph and detection with a Flame ionization detector. The test operated by means of steam space analysis (Head Space technology) at temperature of 120ºC.

1.2 Requirements

The accompanying sample, the respective requirements as well as special method is of the suitable specification to take drawing or the like.

If materials are composed of different components, a separate check is required for every single material.

2. Test preparations

Transport and storage of the tests has to occur in an aluminum coated Polyethylenbeutel.

The sampling has immediately after incoming goods or in a condition, which corresponds to this, to take place. The times of the incoming goods and the sampling are to be labelled. There is no conditioning of the test as a rule. Except of it are physical materials (cotton, wood, leather, wool). These materials are dried before the weighted sample in the chopped up state for 24 hours with calcium chloride ($CaCl_2$).

The test is to be inferred at the agreed place about the whole test cross section from the component. The samples are chopped up in pieces with a weight more than 10 mg and less than 25 mg, without the test warms itself up. If necessary, another test preparations can be also undertaken and such deviation from standard should be mentioned in the final result.

The test amount to be rocked to sleep is directed after the size of the Head Space of little glass whose least contents 5 ml must amount. Per 10 ml little glass volumes are to be rocked to sleep 1.000 g $\pm$0.001 g (i.e. maximum content error 0.1%) test material. Metal are to be removed before weighting. When metal parts are liable to organic substances, for example, varnish, pastes, should be separated mechanically at first and then to rock to sleep.

The weighted test particles become in a Head Space little glasses (least 3 little glasses per test), then air-tighted with the use of a septums with the Teflon coating which points to the interior of the little glass.

3. Testing sets and conditions
3.1 testing sets
Gaschromatograph for capillary column with Head Space Tester, flame ionization detector (FID) and calculator/ Integrator.
WCOT-capillary dividing column with a separating phase from 100% Polyethylene glycol (so-called Wax type, e.g., DBWax, Carbowax...)
0.25 mm I.D., 0.25 μm film thickness, 30-m length
Analytic balance, in the order of 0.1 mg
Microliter syringe, 5 μl, sample in vitreous body
3.2 measuring conditions
Stove temperature program GC: 3 minutes isotherm at 50ºC
                                Heating at 200ºC with a rate of 12 K / min
                                4 minutes isotherm at 200ºC

Injector temperature: 200ºC
Detector temperature: 250ºC
Split ratio: approx. 1: 20
Carrier gas: helium
Middle carrier gas speed: approx. 22-27 cm /s.
Remark: the substance 2,6-Di-tert-butyl-4-methylphenol (BHT) must show a retention time smaller than 16 minutes.

4. Attempt realization
The little glasses are tempered to the enrichment of the substances in the air standing about the test directly before the measurement 5 hours $\pm$ 5 minutes with 120 $\pm$ 1 ºC in the Head Space Tester and are analyzed directly afterwards. At least 3 tests are to be analyzed in each case.
The control value is determined by average signal value of at least 3 measurements with empty test little glasses.
The dosage must run off in all analyses of the tests, the control value and the calibration solution identically and reproduceable.
The separating column must be brought to the bake once per week for 15 minutes at maximum temperature.

5. Calibration
For the quantitative determination of the total carbon emission as well as the amount of special single substances, calibration curve are compared with the method of the external standard.
For the total carbon emission, acetone serves as a calibration substance, for the single substances and the respective materials.
After installation of a new column and after changes in the device a basic calibration with 7 calibration concentration is to be carried out. In addition, a control calibration with at least 3 concentrations has to be carried out at least every 4 weeks.

For basic calibration, 7 calibration solutions of acetone with concentration of 0.1/0.5/1/5/10/50/100grams of acetone in per liter of n-Butanol is made. For the control calibration solution at least three concentrations 0.5/5/50 grams of acetone per liter are required. It is to be guaranteed that in the used n-Butanol show no Peaks at the same time as acetone.

For the single substances, calibration solution are to be produced in the same concentration like for the acetone-control calibration and in each case, a solvent is to be used which shows no peaks with the retention time of the relevant single substance and boiling point lies under 120ºC.

All substances used for the calibration procedure should be checked at least each year to show quality.

With the calibration measurement with one 5 μl - syringe in each case 2 μl $\pm$ 0.02 μl (i.e. maximum injection error 1%) per 10 ml little glass volume in an empty, unlocked Head Space little glasses squirted. Specific attention should be paid to the fact that no air bubbles are in the cylinder when drawing the syringe. The little glass is closed directly after as under 2 described.

The calibrated sample is kept at a moderate temperature for 1 hour with 120ºC in the Head space Tester and then analyzed in accordance with the general test specification, whereby the temperature program of the gaschromatograph can be broken off after elution of the solvent. At least 3 measurements are to be carried out for each calibration solution. With the concentrations of the calibration solutions (in g/l), determined for the respective calibration substance, a straight line can be drawn, whose upward gradient represents the calibration factor k (k(G) for the least squares to take place. The coefficient of correlation K must be thereby larger than 0,995.)

6. Evaluation

From the data of the gaschromatogram the total peak area as well as the surfaces of the peaks belonging to the single substance indicated in the design and/or TL must have to be extracted. For the computation of the total peak area only peaks are consulted,

-their height greater than 10% of the triple value of the baseline is

And

-their area greater than 10% of the area of the Acetone peaks in the calibration solution with the concentration 0.5 g/l is

The detection limit of the analysis procedure must deliver a peak area and a peak height which is in each case smaller than 10% of the respective value which will receive for acetone in analysis of a calibration solution concentration of 0.5 g/l acetone.

The desired emission values result from the results of measurement as follow:

Total carbon emission E_G:

Ascertained from the total peak area which has arisen in the analysis of the tests, and the calibration factor k (G) from the acetone calibration

$$E_G = \frac{\text{Total peak area} - \text{Peak area of control}}{K\,(G)} \times 2 \times 0.6204$$

The unit " μg carbon per g of sample ".

The factor two is originated from the relation of " µg of sample " and arises by the fact that 1 g of sample, 2 µl calibration solution are given in 10 ml little glass.
The factor 0.6204 shows the weight proportion of carbon in acetone.

<u>Single material – Emission Ei</u>:
Ascertained from the Peak area which has arisen in the analysis of the tests for the single material in request i, and the calibration factor k (i) from the single material calibration

$$E_i = \frac{\text{Peak area of the single material i} \times 2}{K(i)}$$

The unit " µg substance i per g of sample ".
The factor two is originated from the relation of " g of sample " and arises by the fact that for 1 g of sample, 2 µl calibration solution are given in 10 ml little glass.
For the results of 3 measurements of a part, not the average value, but all 3 measured values must fulfill the requirement. This is necessary to guarantee that at all places of the construction meet the requirements.