Determination of Organic Substances as Emitted from Automotive Interior Products Using a 1 m^3 Test Chamber VDA276

Preface

The present testing methods represent a possibility of quantitative determination of organic chemical substances released from the automobile interior components under described conditions.
The results, which can be obtained with these methods, are not suitable for:

- to effect further evaluation of health judgment of the emitted substances
- As a basis for the estimation of concentrations to serve, as those in the interior of a complete vehicle, in driving or in a driving-similar condition found warden can

The available set of rules contains the following parts:

Part 1: Standard emission examination [1]
Part 2: Determination of the delivery of formaldehyde, ammonia and phenols according to the method of the equilibrium concentration from parts for the vehicle interior [2]

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1. Introduction

In this VDA recommendation an emission chamber procedure for the determination of volatile organic emissions from construction units of the vehicle interior. It contains remarks to the characteristics and general quality assurance measures for a 1-m^3 test chamber for the standard emission examination of products of the vehicle interior. Further, the general operational procedure of the standard emission examination is described by the supply of the test property to the sample delivery. For the following analysis methods should be used in the context of the FAT/BMBF - project of the technical control board, for short, (TUV) North, Hamburg and Institute for plastic examination and plastic customer (IKP), Stuttgart provided analysis methods recommended. In general, the recommended procedure should be followed and any deviations should be documented in the test report.

2. Tips to further standards, literature

The following are specified standards quoted for separate reference. With no data concerning date or expenditure of such a standard are made, in each case, the up-to-date valid version application should be used.

- European Collaborative Action (ECA) ”Indoor Air Quality And Its Impact on Man”, Report No.8 “Guideline for the Characterization of Volatile Organic Compounds Emitted from Indoor Materials and Products Using Small Test Chambers”
- European Collaborative Action (ECA) “Indoor Air Quality And Its Impact on Man”, Report No. 18 “Evaluation of VOC Emission from Building Products”
- DIN EN 45001, general criteria for the testing laboratories and companies
- DIN EN ISO 9001, quality management system: quality assurance model in Design development, production, assembly and maintenance
- DIN 50011-11, climates and their technical application: Climatic testing facility; General terms and Requirements
- DIN 50011-13, climates and their technical application; Climatic testing facilities; Climatic conditions: Air humidity and air temperature

3. Definitions

In the context of this VDA recommendation, the following definitions apply:

Exhaust air stream: the air flow loading leaving the inspection room by a given

Loading: the relationship of volumes, surface, length or mass of the construction unit to the volume of the test chamber

Emissions: organic substances released from the construction unit under the described test conditions

Emission rate: emission emitted from a construction unit for each time unit: the indication effected related to mass, volume, surface, length or absolutely test part

Fogging: Precipitation from condensable volatile, usually organic substances

Air change: Quotient from supply air stream and test space and measure for the air interchange for each time unit air

Air speed: medium speed of the air flow inside test space, measured in center, caused by the circulation

Air sample: the withdrawal of representative, quantitative volume of the test space atmosphere of ideal mixing: in an ideally mixed area a supplied material without time delay distributes itself completely and homogeneous in the entire area

Condensate sample: the enrichment of condensable substances (Fogging) from the test chamber atmosphere at a coolable collecting device

Concentration in the test chamber: the concentration of one or several chemical substances in a gaseous air sample, those in a representative place of the test space, determined by chemical or physical means

Surface of the construction unit: the surface inspection room passable formed by the outlines of the construction unit for organic substances

Test chamber: air-tight lockable area for the determination of volatile organic emissions under defined climatic conditions

Test chamber volumes: Volume of the test chamber less all volumes taking technical building and devices in the test chamber

Recovery: proportional relationship de rim exhaust air stream intended mass of a substance for the mass loss of this substance in the inspection room, determined in the same period

Supply air stream: sum of all gaseous flow rates for each time unit led into the test chamber

4. Symbol and units

C	[µg/m^3]	Test space-concentration of a substance (at 1.013×10^5 Pa and 293K)
R	[µg/h]	Emission rate
R_A	[µg/m^{2}h]	Emission rate related to the construction unit surface
R_V	[µg/m^{3}h]	Emission rate related to the construction unit volume
R_m	[µg/kgh]	Emission rate related to the construction unit mass
N		Air change: changes of air per hour
L_A	[m^2/m^3]	Surface loading
L_V	[m^3/m^3]	Volume loading
L_m	[kg/m^3]	Mass loading

5. Basis of the procedure

A construction unit is put into an approximately ideally mixed 1 m^3-test chamber and stored there under given temperature, humidity and of air flow. Organic substances, which escape from the construction unit, enrich themselves in the inspection room and the air flow is delivered. At selected times air or condensate samples are taken, from which with chemical analysis, the concentrations of gaseous air contents materials in the test chamber can be determined qualitatively or quantitatively.

6. Emission tester

It is used for the determination of the gaseous emissions and contains the following functional elements:

- Test chamber
- Air agitator
- Test chamber keeping at a moderate temperature
- Air humidifier

- Pure air supply
- Control and regulation
- Entire-CH-monitor
- Fogging cooling device

Concerning the design, system, combination and technical execution of these individual functional elements, there should not be binding defaults occurred. In the following sections, to the extent necessary, minimum requirements which can be fulfilled are specified. The proof is additional to furnish (e.g. by interlaboratory tests) that with the used emission chamber comparable results for the chemical substances which can be determined can warden obtained. References to continuous quality assurance measures are in chap.7.

6.1 Test chamber

The test chamber is an airtight lockable space with a volume of 1± 0.05 m^3. Inside it, a device for air mixing and a rack which guarantee the storage of the construction unit without wall contact are required. About the attitude of the air exchange: for air sampling procedures, an inlet and an outlet pipe (Ø < 50 mm) should be diametrically arranged to avoid short-circuit flows.

Materials

The wall surfaces of the test chamber and the rack for holding the construction unit must be made of high-grade steel or glass. With building or parts in the test chamber inside, for technical reasons, the two materials mentioned are preferred. Materials are to be selected based on exhibition of small emission and adsorption of organic substances and do not exceed with the test chamber wall surface.

Tightness

To avoid uncontrolled air exchange, it should be guaranteed that the leakage current caused by leakages in air change examinations at 1000 Pa positive pressure is less than 1% of the test chamber volume per minute and/or 1% of the total supply air stream. To prevent foreign air inflow, the emission procedure should always operate with a small positive pressure in the test chamber in related to the atmospheric pressure in the laboratory and/or a volume overcurrent. This is to be considered in particular during the air sampling.

Air mixing

A substantial basis of this procedure is the ideal mixing of the test chamber. This must be achieved by a suitable device for air mixing and guaranteed its fulfillment of this condition even with large volume, bulky test sample. The air flow rate in the centre of the test chamber should be above 1 m/s.

Note: In Report No.8 and No.18 of the European Collaborative Action (ECA) “Indoor air Quality and Its Impact on Human ", methods are described for the regulation of tightness and air mixing.

Cleaning

With a suitable cleaning method e.g. the thermodesorption at high temperatures it must be guaranteed that a thorough cleaning between examinations takes place. A detailed description is given in chap.9.1. The success of the cleaning should be ensured before the start of an examination by a blank value sample.

6.2 Test chamber temperature

Due to the strong influence of the temperature on the emission behavior, the high requirement of temperature control in the test chamber must be fulfilled. A broad temperature range is also required to suit the requirements of different test temperature. The spatial and temporal temperature derivation may not exceed ±0.5 K.

6.3 Air humidity

Humidification of the test chamber supply air it is to be accomplished in such a way that the formation is excluded from steam and aerosols. By the humidification unit a dew point must be able to reach 10.4 ºC to 65 ºC in the supply air stream. This corresponds to a relative humidity of 45% with 23 ºC and/or 5% with 65 ºC.

6.4 Pure air supply

Examinations with air change as well as the withdrawal of air samples makes the supply of reinstall necessary. For adjustment the fixed air change of 0.5 per hour a volumetric air flow must be regulated by 6.67±0.35 l/min. under the test conditions (65 ºC, ambient pressure) with an accuracy of ±3%. The sum concentrations of volatile organic hydrocarbon compounds of the supply air before the start of an examination should not be more than 50 $\mu g/m^3$ as a total and 5 ug/m^3 for individual substance. The fine dust portion must be reduced by a particle filter of 7 µm to avoid adsorption.

6.5 Measurement of the sum concentration at hydrocarbon connections

For the qualitative evaluation of the sum concentration of hydrocarbon compounds in the test chamber during a test cycle, an on-line analyzer with flame ionization detector (FID) is used, in which the composite signal of a broad spectrum of organic substances is seized. The calibration takes place with a test gas mixture of approx.100 ppmv propane in synthetic air. As a zero-gas, nitrogen of the purity 5.0 is used.

A conversion formula for the indication of a mass concentration is in chap.10.

Note: Conditions according to device for the determination of quantitative determination on the basis of a FID composite signal as indicated in "determination of the sum concentration at hydrocarbon compounds with a flame ionization detector (FID)”.

6.6 Fogging cooling

Qualitative determination of condensable components of the organic emissions of a construction unit takes place via radiator box an attached inside the test chamber. The radiator box must be able to hold on a temperature of a 21±1 ºC in a 100 ºC test chamber. It is to be noted that the use of a radiator box can affect the recording of temperature in the test chamber. And material selection section 6.1 is to be used.

Fig. 1 The assembly line of an emission test chamber

7. General quality assurance measures

The integration of numerous technical functions in an emission chamber creates the possibility of error, which require a regular and conscientious examination of the entire system. Since these errors can affect the part directly and hence the inspection result, the emission chamber is to be merged into an accreditable quality assurance system or a comparable continuous monitoring measure. Described below are some important testing methods for the measurement of test parameters.

Tightness: The tightness of the inspection room is examined with a positive pressure of 1000 Pa by measurement of the decrease of pressure with an interval of 2 hours. The sensitivity of the pressure absorbers is selected to be smaller than 100 Pa, with an accuracy of ±5%. The average leakage rate in this period is also calculated.

$$V[\%/h] = \frac{100\%}{t[h]} \times [\,(P_1/P_2)-1]$$

V: specific leakage rate related to the test chamber volume in parts per thousand per hour
P_1: absolute pressure in the test chamber at the beginning of the examination in Pa
P_2: absolute pressure in the test chamber at the end of the examination in Pa
t: period of the leakage rate in h

Alternative procedure: The tightness of the test chamber is determined with a positive pressure of 1000 Pa by measurement of the radioactive half-life $t_{1/2}$ of the pressure acceptance in the test chamber. The radioactive half-life is the period, in which the positive pressure reached half of its initial value. Thus the specific leakage rate is calculated according to the formula

$$V[\%/h] = \frac{100\% \times (\Delta p/p)}{t[h]} \times (\mathrm{In}\ 2/t_{1/2})$$

V: specific leakage rate related to the test chamber volume in per cent per hour
$\Delta p/p$: relative positive pressure related to ambient pressure
$t_{1/2}$: time interval up to the reduction of the positive pressure on half of the initial value in h

In both procedures the tightness is determined at test chamber at temperature of 65 °C.

Air speed:

Air speed is measured in the centre of the empty test chamber. For measurement, hot wire -, film or impeller anemometers are used.

Supply air stream:

Due to the strong influences on the test result, the supply air stream of regular intervals is examined. The inflowing volume in approximate atmospheric pressure is measured directly at the inlet of the inspection room with a gas meter, which exhibits an accuracy of ± 2%, and the range is certified as specified in section 6.4. The minimum volume which can be determined amount to the 200-times of the reading precision of the gas meter. The supply air stream can then be calculated as quotient from inlet volumes and measuring period. For the calculation of the change of air under test conditions, the determined supply air stream must be corrected according to the ideal gas law with the dominant temperature and pressure values.

Control values:

Control values in supply air and room air, as well as other sources of emission, which stand with the test chamber atmosphere, are used for assistance of regular examinations. With control substance, its quantitative methods of analysis are proceeded with specific procedure and measurement. For the half-quantitative control value monitoring, a screening procedure e.g. Tenax/ Thermodesorption is consulted, which exhibits a sensitivity < 2 µg/m^3 for single substances. The sum concentration of VOC, determined with this procedure, should be below 50 µg/m^3, whereby the concentrations of single substances are not to exceed 5 µg/m^3.

Temperature and humidity:

The adherence to the tolerances for temperature and relative humidity is determined with a combined temperature moisture meter on ±0.5K and/or ±5% exactly. If sufficient experiences are present over possible condensation effects of the moistened supply air, the humidity can also used in the supply air stream measurement.

8. Construction unit

Before the determination of volatile organic emissions, the construction unit was exposed to condition which can affect the quantitative investigation and hence the results substantially. It is necessary to standardize the pre-examination of construction unit as far as possible.

8.1 Withdrawal of the construction unit

For standardized examinations, in which the emission behavior of a new part is to be seized, the construction units in the delivering condition are to be examined with incoming goods. The possible adsorption of substances from the environment which were not originally in contained in the construction unit also counts. The pre-examination of the construction unit should be documented as complete as possible. If it is necessary to dismantle the parts for inspection purposes, the separation of the construction unit or any changes, those procedures should be documented. Anschmutzungen are to be avoided.

8.2 Packing, transport, storage of the construction unit

The construction unit should be packed after withdrawal until the start of the examination that the contamination of the construction unit by chemical substances from the environment is prevented, the emission potential of the test sample is conserved and material changes by radiation (e.g. sun exposure) to be excluded.

Note: For this purpose a weldable sandwich foil with an intermediate layer of aluminum is suitable, concerning gas and diffusion tightness. It also protects against transportation damages offers (e.g. company Flöter, 71735 Eberdingen nut village). During the temporary storage and transport of the construction unit, the temperature of 23 ºC should not be exceeded.

9. Procedure of the standard emission examination

In this section, the proceedings, definitions and requirements of the standard emission examination in the chamber are described. This examination gives a broad overview of the intensified emission conditions for the construction units.

The test temperature amounts to the first conditioning phase 65 ºC with a supply air humidity of approx. 5% R.H. (45% correspond R.H. at 23 ºC). The change of air amounts to 0,4 l/h.

The method determines the air concentration of BTXE/S: aromatics, aldehydes and ketone by GC-MS analysis. When necessary, further analytic procedures can be used for air determination e.g. amines, glykolethern, phthalate or n-nitrosamine can within 2.5 the hour sampling window beprobt.

In a second conditioning phase, the test chamber is raised to a temperature of 100 ºC is raised (without supply air humidification), while at the same time, the cooling device of the fogging condensate sample is kept at a moderate temperature of 21 ºC. The test sequence is in minutes form to document (for example see appendix 1). During the entire test period, the temperature, humidity and total concentration of organic compounds in the test chamber is monitored by mean of FID.

9.1 Cleaning

Before the start of a construction unit investigation, all pollutant-liable surfaces of the test chamber must be in cleaned condition. If necessary, existing background concentrations must be so small that the quality of the results from air-analytic regulation methods remains uninfluenced.

Test chamber

The test chamber should be first freed of particles or the like. Mechanical cleaning methods are used to remove the remains of the construction unit (e.g. industrial vacuum cleaner). Drying up of condensate arising in the edges may be removed by steel wool or volatile solvents. In the case of a thermal cleaning, the test chamber are baked under scavenging air at a temperature > 200 ºC which can reach all surfaces and in contact with the test chamber atmosphere. The cleaning procedure is terminated after 10 changes of air starting from reaching the baking temperature. If the cleaning of the test chamber surfaces takes place via washing with alkaline cleaning agents, it should be accomplished with two washing processes of demineralized water. Afterwards the test chamber is dried with rinsing air flow at test temperature.

Others

All parts in contact with the test chamber atmosphere, which are not directly included in the cleaning procedure of the test chamber, must be supplied to a separate purification process comparable from the technical view.

Note: With the test chamber outfits parts, the maximum temperature of the drying process is 200 ºC with120 ºC over 2-3 hours is considered sufficiently.

9.2 Examination

The actual examination extends from inserting the construction units into the emission test chamber to the withdrawal after conclusion of all conditioning and sampling procedures.

9.2.1 Preconditioning

The test chamber is conditioned before inserting the construction unit at a temperature of 70±1 ºC and a relative humidity from 5±2%.

9.2.2 Preparation

The entire emission chamber should be examined carefully for all necessary functions before the start of a test, so that the test can be completed with high security. For that, the pure air production, the test chamber, the climatic regulation, the entire CH monitor, data recording and all other necessary accessory equipment are to be included in the functional check.

Note: Appendix 2 contains an example checklist of the activities before the test start.

9.2.3 Inserting the construction unit

The construction unit is put in the test chamber concentrically and avoids wall contact by the rack. A sentence from several construction units is to be arranged in such a way that the all-round surge that result in the best possible way which can be reached by the circulation. It is to be made sure that the construction units cannot shift over the entire testing period. The test chamber is to be locked immediately after inserting the construction units.

9.2.4 Conditioning phase

Immediately after locking the emission chamber, the examination is started by selection of an automated test program or by manual control of the test conditions. In the following table the climatic parameters and sampling procedures of the individual test phases are summarized. Fig. 2 Plot of temperature gradient and the sampling windows:

Tabular operational sequence of the standard emission examination

Row	Time [h:min]	Temperature [ºC]	Air change [l/h]	Incoming air humidity [ºC/%R.H.][1)]	Process
Conditioning					
1		70	maximal	10.4/4.0	
2	-0:30	70	maximal	10.4/4.0	If necessary, prepare the test chamber
Testing					
3	0:00	65	0.40	10.4/5.0	1. Conditioning phase
4	2:00	65	0.40	10.4/5.0	Start testing -BTXE/S-Aromatic -Aldehyde and ketone -Clearanalysis -If necessary, further testing

5	4:30	100	0.44	out	2. Conditioning phase -Fogging test start -Fogging test end
6	8:00	100	0.44	out	

1) Dew point temperature e.g relative humidity at 65/ºC u.1.013x10^5 Pa

Supplementing notes:

Note 1: In the test chamber before beginning of the examination, the demanded humidity prevails, must be begun with the humidification of the supply air in time.

Note 2: The preparation of the test chamber must take place so promptly that at the latest to test beginning the conditions of the conditioning are again fulfilled within the accuracy borders.

Note 3: It is to be guaranteed that the inspection temperature of 65±0.5 ºC in the test chamber is again reached at the latest after 30 min.

Note 4: The given air sampling phase is laid out that after conclusion of the prescribed air sampling procedures, there is still time for supplementing air sampling remains for the testing of e.g. amines, glykolethern, phthalate or n-Nitrosamine.

Fig. 2 test cycle of the standard emission examination for regulation easily and heavy volatile substances from construction units with a 1 m^3 emission test chamber

Air sampling

The withdrawal of air samples is to take place with standardized emission examinations at a given time. Therefore the duration and sequence of the air sampling procedures are constantly bleached to hold during an air sampling phase of several analysis. The execution of the air sampling is described in the respective air-analytic testing methods.
Note: The number of air sampling procedures running off at the same time is limited by the adjusted change of air on maximum 61/min (65 ºC, ambient pressure).

Condensate sampling

The condensate sampling begins with the supply of radiator box kept at a moderate temperature on 21 ºC. Since the test chamber must be open in order to withdraw the condensate sample, the examination has to be terminated.
The execution of the condensate sampling is operated as described in a standard working instruction.

9.2.5 Completion of the examination

An emission examination is completed with the switching off of the climatic regulation and/or the opening of the emission chamber. A cleaning procedure is required right after the emission examination (see chap. 9.1).

10 Calculation and presentation of the inspection results

Upon completion of the test, one receives quantitative statements of air concentration in the test chamber and from this emission rate or qualitative statements about the presence of chemical substances in the test chamber atmosphere can be calculated.
The determination and calculation of these inspection results are a component of the respective chemical analysis procedure according to the respective standard working

instruction. As reference volumes, the volume of the test chamber in the **normal state** [1] (standard temperature and pressure) is used, which can leads to a lower, calculated concentration value than the actual one.

Test chamber concentration
Standard emission test 65ºC Toluene (108-88-3) 10µg/m^3
Test Phase Substance (CAS-Nr) Concentration [µg/m^3]

Qualitative substance verification
Standard emission test 65ºC Toluene (108-88-3) Identification
Test Phase Substance (CAS-Nr) Verification

Total carbon emission-mass concentration with FID
Standard emission test 180 min Total-CH 10mg $_{CH}$/m^3
Test Time Substance Concentration [mg$_{CH}$/m^3]

Since the recording takes place continuously, the concentration profile in the test chamber is shown by a concentration-time diagram. Time t =0 min is to set as the time locking the test chamber after inserting the construction unit. The total carbon concentration is calculated from the volume concentration of a FID reading according to the formula (calibration with a propane gas mixture).

$$\text{Mass concentration} = \frac{\text{Mol mass}_{\text{Propane}}}{\text{Mol volume}_{\text{Propane}}} \text{ x Volume concentration}$$

$$m_{CH}\ [\text{mg/Nm}^3] = \frac{44.094\ [\text{kg/kmol}]}{24.055\ [\text{m}^3/\text{kmol}]} \text{ x } C_{FID}\ [\text{ppmv}]$$
$$= 1.833 \text{ x } C_{FID}\ [\text{ppmv}]$$

1) as a normal state a pressure is based by 1.013x10^5 Pa and a temperature of 293 K for the gas volume

Emission rate
With the mass concentration, the emission rate can be calculated for the time of the sampling in the test chamber. This reads:

Emission rate = Air stream volume x Concentration
m_{CH} [mg/h]= V [m^3/h] x C_{CH}[mg/m^3]

Area-related emission rate

$$\text{Emission rate}_A = \frac{\text{Air stream volume x Concentration}}{\text{Surface of the test material}}$$

$$m_{CH;A}\ [\mathrm{mg/m^2h}] = \frac{V[\mathrm{m^3/h}] \times C_{CH}\ [\mathrm{mg/m^3}]}{A[\mathrm{m^3}]}$$

Mass-related emission rate

$$\text{Emission rate}_{m} = \frac{\text{Air stream volume x Concentration}}{\text{Mass of the test material}}$$

$$m_{CH}\ [\mathrm{mg/kgh}] = \frac{V[\mathrm{m^3/h}] \times C_{CH}\ [\mathrm{mg/m^3}]}{m[\mathrm{kg}]}$$

11 Test report

It should be in compliance with DIN ISO 9001 and/or the following data are given according to DIN EN 45001 in a test report:

- Name and address of the test laboratory
- Name and address of the client
- Input information of the construction unit and kind of the packing during delivery
- Clear notation(s) for the identification of the construction unit
- Detailed description of the construction unit
- Production date
- Storage and climate of entrance of the construction unit before the test start
- Changes made in the construction unit (e.g. disassembly, surface sealing...)
- Test parameters temperature, humidity and change of air during the entire test period
- Time, nature and duration of the sample/sampling
- Method of analysis and working instruction
- Short description of the analysis procedure
- Precision and correctness of the analysis procedure
- Recovery of the determined substance
- Reference to the available standards
- Deviations from the testing method and characteristics during the examination
- Date and signature

Appendix 1 **Lab. Protocol** Page 1 of 1	**Determination of Organic Substance as Emitted from Automotive Interior Products Using a 1 m^3 test cabinet** **Part 1: Standard Emission Test**	**VDA 276-1**

Order No.	Name of construction unit
Processor, Department	Pretreatment See section B
Date	Test condition See section C
Test No.	

A. Characteristics

Manufacturer: Manufacturing date:

B. Storage

Store in: Place:

Temperature: ºC Packaging: PE

PE-Al

Openly

C. Measurement

Measurement beginning: End:

EPS: Total-CH-File:

C1.Conditioning

Automatic: Manual:

Program No.: Temperature: ºC

Dew point bath: ºC

Incoming air: NI/min

C2.Test

Date/ Test name Substances

____________________ 1 2 3 4 5 6 7 8 9

____

____

D. Relocation/ outsourcing

Evacuated in: Place:

Date:____________________ Signature:_________________

Appendix 2: Check list of emission examination (example)

Regular check	i.o.
Air-filter for carrier gas supply (function, loading)	
Water stock for sufficient air humidification	
Berstsicherung (function, density)	
Visual check of the inspection room, air agitation	
Function of the Fogging cooling (circulation, tightness, temperature)	
Before insertion of construction unit	
Supply air stream (actual value) correctly adjusted	
Fogging cooling connected, discs put on	
Test chamber inserted and intact	
Total-CH-Analysis and fogging cooling attached	
At test beginning	
Necessary test chamber openings	
Activate data recording	
Keep test parameter	

Determination of Organic Substances as Emitted from Automotive Interior Products Using a 1 m^3 Test Chamber VDA276-2

Part 2: Determination of the release of formaldehyde, ammonia and phenol-measurement by method of steady-state concentration.

1. Purpose

The available VDA recommendation describes a measuring procedure for the determination of the release of formaldehyde, ammonia and phenol from shaped parts of the vehicle interior.

2. Range of application

The measuring procedure applied for the emission examination of shaped parts under extreme climatic conditions.

3. Reference to other testing methods

ENV 717-1 timber material determination of the formaldehyde discharge Part 1: Determination of the formaldehyde discharge according to the test chamber method

4. Principle

The determination of the release of air-contaminating pollutants such as formaldehyde, ammonia and phenol from shaped parts of automotive interior made of textile and wood fiber, using 1 m^3-Chamber-Method. Practical condition as in the automotive interior under unfavorable conditions is considered.

For the simulation of conditions, which can occur in motor vehicle interiors during sun exposure, the test chamber temperature was specified at 65ºC. Based on different preliminary investigations over the figure determination of air changes with closed ventilation flaps in automobiles as well as regarding the average more highly lying portion of the blocked textile fleeces in the vehicle interior, the space loading figure of air changes of 4 was selected for the test. Relative humidity is fixed at approx. 11%, i.e. it introduces air saturated with water vapor at ambient temperature in the test chamber kept a moderate temperature on 65ºC. The construction unit is generally air-retune inserted.

5. Sampling

The construction unit is to have total surfaces of 2 m^3. The construction unit is a larger shaped representative part taken from the complete part. The construction unit is to be packed immediately after the production or after the withdrawal, e.g. stored in polyethylene foil at a temperature of max.25 ºC until the examination. In order to avoid condensation on the surfaces, the sample material must exhibit a temperature > 20 ºC when bringing into the chamber.

6. Testing method

6,1 Test parameters

The sample material is stored in a chamber with a total volume of 1 m^3 under defined climate conditions:

Test temperature	(65±1)ºC

Relative humidity	(11±5)%
Change of air	0.5 h^{-1}
Space loading	$2m^2/m^{3*}$

* A smaller space loading is just as possible, if the figure of air changes is reduced, to reach the relationship of space loading/figure of air changes to 4.

6.2 Description of a 1 m^3 chamber

6.2.1 Glass chamber (example)

The inside dimensions of the chamber amounts to 1.00m x 0.80m x 1.25m (fig. 1). The outside walls are manufactured from 8 mm thick glass. The setting of the temperature occurs with the help of a heating thermostat manufactured from high-grade steel, the circulation achieved through two ventilating fans right in the thermostat.
By introduction of air into the test chamber, kept at a moderate temperature of 23 ºC, saturated with water, on 65 ºC the test chamber air with relative of (11±5)%.

Fig.1 Schematic construction of a 1 m3 chamber (e.g. Glass with external thermal insulation)

6.2.2 High-grade steel chamber

The test cabinet consisted of a conditioning area and the actual emission chamber (1.78m x 0.75m x 0.75m). The test chamber is a gas-tight welded adjustment tank with smooth surfaces made of high-grade steel with small roughness depth. The attitude of the humidity takes place in the air inlet stream according to the dew point principle. An accurate description of the high-grade steel test chamber is contained in VDA recommendation Part 1, Section 6.
Note: Other $1m^3$-chamber test checking facility is permissible, if it is proven to agree with available described measuring procedures.

6.3 Devices for the set up of the test chamber

- 1 m^3 chamber
- 1 air measurer, 50 to 300l/h
- Gas sampling equipments
- Thermal hydraulic graph
- Barograph
- 3 air washer compressor
- Air bleed port or pump compressor
- Activated charcoal tower
- Purifying oil separator
- Insulating material (only glass chamber)

7. Execution of the test

7.1 Preparation of the test equipments

The chamber is to be adjusted in such a manner that a test temperature is reached by (65±1) ºC. Fresh air can enter e.g. over an air supply line. For air pre-purification, a activated charcoal tower is used. Before the start of an examination the concentrations of the following single materials in test chamber air should not be exceeded:

	ppm	µg/m^3
Formaldehyde	<0.005	6.3
Phenol	<0.003	11.4
Ammonia	<0.01	13.9

7,2 Test

The construction units is removed from the packing and inserted into the chamber kept at a moderate temperature. Once the sample inserted in the chamber and during the test, the temperature and the relative humidity is documented by the thermal hydraulic graph. Two air washers switched into row for the humidity adjustment. The air volume in the closed chamber is regulated by transformer and throttle valve e.g. with a figure of air changes of 0.5 h^{-1} to (500± 5) l/h.

7.3 Sampling for the regulation contents of formaldehyde, ammonia and phenol

The measurements of the concentrations of formaldehyde, ammonia and phenol takes place daily up to reach the respective balance concentrations.

Note: At the beginning of the investigation, it is necessarily to accomplish 3 successive working-days 2x daily sampling. The loading of the chamber with the test samples should be according to the scheduled. For this masses of gas, in each case, 0.1m^3 is taken (fig. 2), with liquid-filled absorption and by means of different analytic methods with assistance of gas sampling equipments of the room air.

7.3.1 Assigned methods of analysis

7.3.1.1 Formaldehyde

For the determination of the formaldehyde concentration, test chamber air is passed through the gas wash bottle filled with distilled water at a sampling speed of 2 l/min. The formaldehyde content of the absorber solution is determined by photometry and/or fluorimetry using the acetyl acetone method (VDI recommendation 3484 sheets 2, draft 1999). The detection limit for the applied method is 0.005ppm formaldehyde (1ppm~ 1.25 mg HCHO/m^3).

Alternatively, formaldehyde content can be determined by the DNPH method according to the VDI recommendation 3862 sheets 3, draft October 1999.

7.3.1.2 Phenol

The phenol content can be determined with the help of the p-Nitranilin-method (VDI recommendation 3485, December 1988). With absorber solution of 0.1 Mol caustic soda solution, which at a sampling speed of 1 l/Minute. The detection limit of this analysis method for phenol under these sampling conditions is approximately 0,003 ppm. (1 ppm~ 3.8mg/m^3).

7.3.1.3 Ammonia

0.1M sulfuric acid at the speed of l/Minute serves as the absorber solution for the ammonia in the room air for the photometric test. In each case, 1 ml of the absorber solution react with 5 ml solution A (25mg sodium nitroprusside and 9.975g phenol dissolve in water and fill up to 500ml) and solution B (2.5g sodium hydroxide and 2.5ml sodium hypochlorite solution dissolved and make up to 500ml with water). It is transferred to the thermostatic water bath at 40ºC for 30 min. Subsequently, the extinction of the developing chromocomplexes Indophenol is determined photometrically at the wave-lengthen of 580nm. (detection limit: 0,01 ppm ammonia; 1 ppm~1.39 mg/m^3). Note: Other methods of analysis are permissible, if it is proven to agree with the results of the specified procedures.

8. Calculation of the results

Calculation of the corrected sampling volume (20C, 1013 hPa):

$$\mathbf{V_{korr} = \frac{V_P \times P_L \times 293}{(273 + t_G) \times 1013} \quad [m3]}$$

V_{korr}: corrected sampling volume (20ºC, 1013 hPa) [m^3]
V_p: sampling volume, read off [m^3]
P_L: medium air pressure during the measurement [hPa]
t_G: middle air temperature in the mass of gas counter during the measurement [ºC]

Calculation of the formaldehyde concentration:

$$\mathbf{K_{CH2O} = (Ex - Ex_{BW}) \times A \times 3 \quad [\mu g/30ml]}$$

$$\mathbf{K'_{CH2O} = \frac{K_{CH2O}}{1000 x V_{korr}} \quad [mg/m^3]}$$

$$\mathbf{K''_{CH2O} = \frac{K_{CH2O}}{1.248} \quad [ml/m^3] \text{ and/ or } [ppm]}$$

K_{CH2O} : formaldehyde content of the sample [µg]
Ex : extinction of the absorption solution
Ex_{BW} : extinction of the comparison solution
A : upward gradient factor of the calibration function [µg/10ml]
K'_{CH2O} : formaldehyde concentration [mg/m^3]
K''_{CH2O} : formaldehyde concentration [ppm] and/or [ml/m^3]

Calculation of the ammonia concentration:

$$\mathbf{(Ex_{St} - Ex_{BW}) \sim C_{St}}$$

$$\mathbf{(Ex_{NH3} - Ex_{BW}) \sim K_{NH3}}$$

$$\mathbf{K'_{NH3} = \frac{K_{NH3} \times 30}{V_{korr}} \quad [mg/m^3]}$$

$$\mathbf{K''_{NH3} = \frac{K_{NH3}}{1.39} \quad [ppm] \text{ and/ or } [ml/m^3]}$$

Ex_{St} : extinction of the standard solution
Ex_{NH3} : extinction of the analysis solution

Ex_{BW} : extinction of the comparison solution
K_{St} : concentration of the standard solution [mg/ml]
K_{NH3} : concentration of the analysis solution [mg/ml]
K'_{NH3} : ammonia concentration [mg/m^3]
K''_{NH3} : ammonia concentration [ppm] and/or [ml/m^3]

Calculation of the concentration of phenol

$$K_{Ph} = (Ex - Ex_{BW}) \times B3 \qquad [mg/30ml]$$

$$K'_{Ph} = \frac{K_{Ph}}{V_{korr}} \qquad [mg/m3]$$

$$K''_{Ph} = \frac{K'_{Ph}}{3.8}$$

Ex: extinction of the analysis solution
Ex_{BW} : extinction of the comparison solution
B: upward gradient factor of the calibration function [mg/10ml]
K_{Ph}: concentration of phenol [mg]
K'_{Ph}: concentration of phenol [mg/m^3]
K''_{Ph}: concentration of phenol [ppm] and/or [ml/m^3]

9. Test report

In the test report the following information should be given under reference to this VDA recommendation:

- Origin of the construction unit
- place, situation and condition of the material at the time of the sampling, humidity in particular
- date and production of the material
- date of the sampling
- date of the test
- test condition
- space loading
- determined concentrations of formaldehyde, ammonia and phenol (e.g. in ppm)
- description of further details [1)]

[1)] Report all procedures that are not in agreement with this VDA recommendation (withdrawal of the construction unit, conditioning etc..)