Emission test chambers: Standards, requirements and challenges

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What are VOC‘s?

- VOC = **V**olatile **O**rganic **C**ompounds

- VOC are present in various forms, occur everywhere and can emerge both natural and artificially
  - e.g. natural or artificial flavors
  - serve e.g. as a messenger for plants (Discover Magazine, April 2002 "Talking Plants")
  - are necessary for processes (e.g. cleaning of metal sheets), processing of products (e.g. paints) or the improvement of properties (e.g. plasticizers)

➢VOC cannot be avoided in the environment and are required for many industrial processes and products

Definitions of VOC

- Definition of VOC not clear:
  - „[…] organic compound which has at 293.15 K a vapor pressure of 0.01 kPa or more [...]“ (Directive 1999/13/ EC (VOC Directive), Article 2, paragraph 17)
  - „[…] Organic compound having an initial boiling point not exceeding 250 °C at a standard pressure of 101.3 kPa;“ (Directive 2004/42 / EC (Decopaint Directive), Article 2, paragraph 5)
  - Definition according to DIN EN ISO 16000-6 and the WHO („Indoor air quality: organic pollutants“; Report on a WHO meeting in Berlin, August 23-27, 1987, ISBN 92 890 1277 3, p. 4)

Description	Abbreviation	Boiling point [°C]
Very Volatile Organic Compounds	VVOC	< 0 to 50-100
Volatile Organic Compounds	VOC	50-100 to 240-260
Semi Volatile Organic Compounds	SVOC	240-260 to 380-400

Definition of TVOC

- Often, however, not only the concentration of a specific VOC is of interest, but also the total content of all VOC => TVOC
  (sum of all total volatile organic compounds)

- TVOC is defined according to DIN EN ISO 16000-6 and 13999-2 as:
  "sum of volatile organic compounds collected on Tenax TA® and eluted between and including n-hexane and n-hexadecane. The compounds are verified with a flame ionization detector (TVOCFID) or the mass spectrometer as detector (TVOCMS)."

- The measured TVOC value depends on the sampling and analytical procedure used. Any interpretation and comparison of the values should therefore always be made taking these parameters into account.

Why VOC Measurements?

- Many VOC have an odor which people notice e.g. as "artificial", "unpleasant" or “stinging”. For example:
  - limonene
  - Diethylamine
- VOC may have an irritant effects on the respiratory system and other mucous membranes. For example:
  - toluene
  - styrene
- Some VOC are carcinogenic or mutagenic. For example:
  - benzene

Why VOC Measurements?

- Because of the aforementioned properties, the use of many VOCs is severely restricted or prohibited:
  - VOC Council Directive (1999/13/EC) dated March 11, 1999
  - Stockholm Convention dated May 22, 2001 => „dirty dozen“
  - Decopaint Directive (2004/42/EC) dated April 21, 2004
  - REACH Regulation (2006/1907/EC) dated December 30, 2006

- Manufacturers are partly obligated and also want to ensure that their products comply with legal requirements and do not represent health hazard for customers.

Standards for the Measurement of VOCs

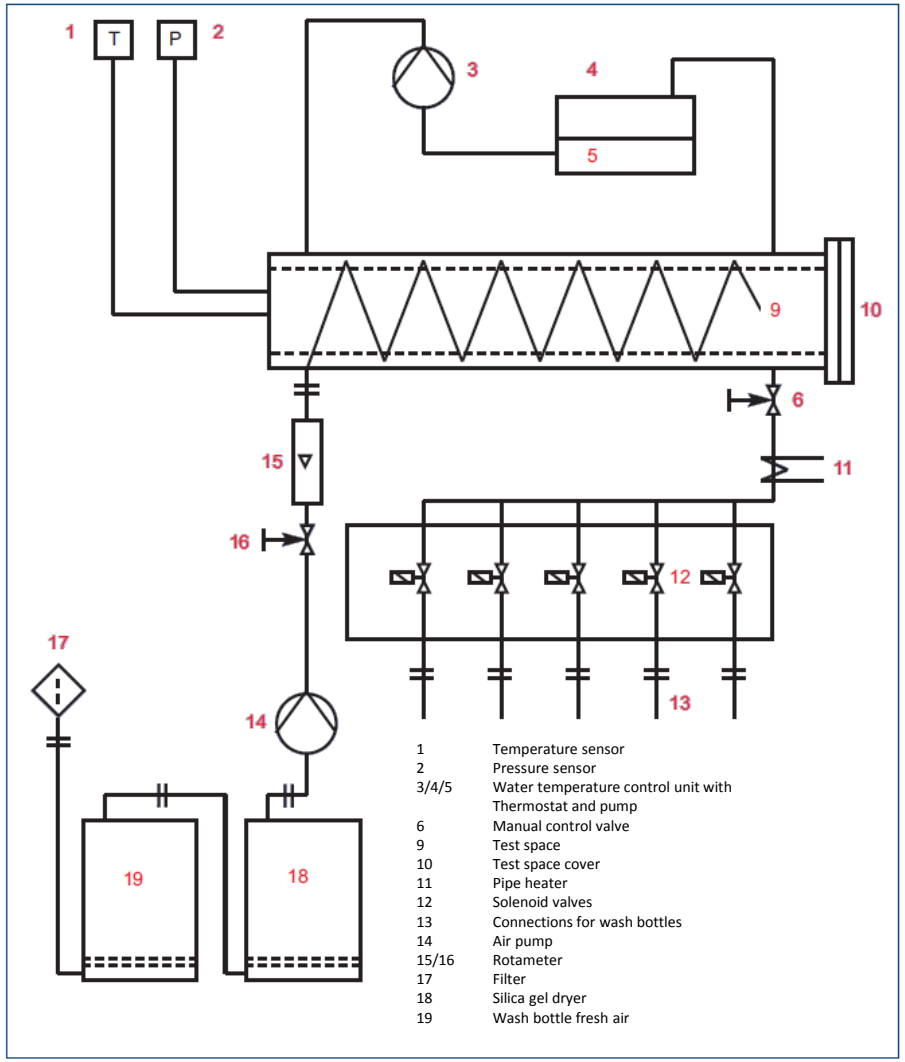
- Today there are all kinds of standards for the measurement of VOCs.
- For example:
  - DIN EN ISO 10580: Resilient, textile and laminate floor coverings - Test method for volatile organic compound emissions
  - DIN EN 13999-2: Adhesives - Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application - Part 2: Determination of volatile organic compounds
  - DIN EN ISO 22155: Soil quality - Gas chromatographic determination of volatile aromatic and halogenated hydrocarbons and selected ethers - Static headspace method
  - …..

VOC Measurement in Test Chambers

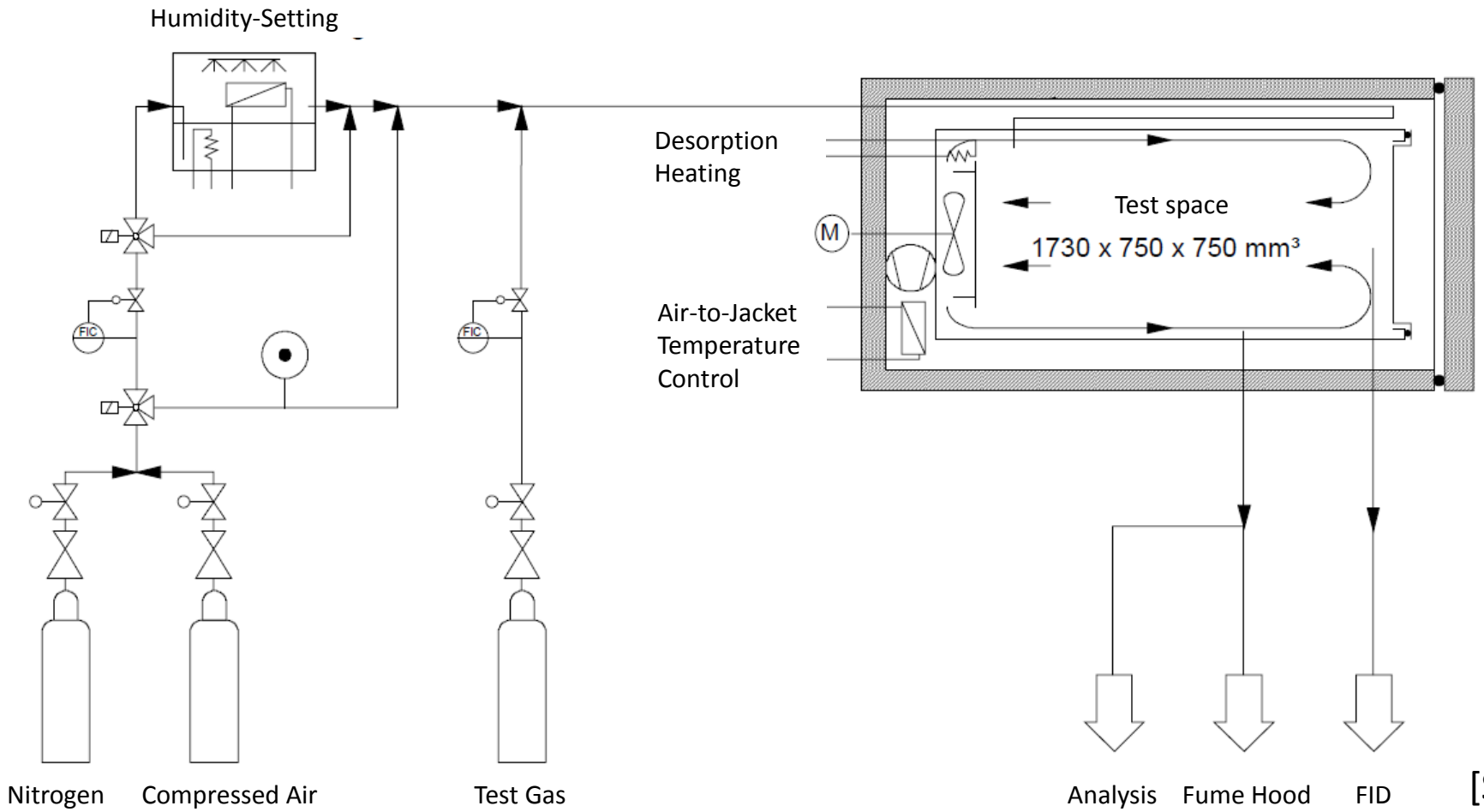
- If you want to do a qualitative and quantitative analysis under defined environmental conditions, the test material must be placed in a test chamber and here the sample must be taken.
- Standards which require a test chamber:
  - DIN EN 717-2 Wood-based materials - Determination of formaldehyde release - Part 2: Formaldehyde emission by the gas analysis method
  - DIN EN ISO 16000-9 Indoor air pollutants - Part 9: Determining the emissions of volatile organic compounds from building products and furnishings - Emission test chamber method
  - VDA 276 Determining organic emissions from components for vehicle interiors with a 1m³ test chamber
  - DIN ISO 12219-1 Indoor air of road vehicles - Part 1: Whole vehicle test chamber - Specification and method for the determination of volatile organic compounds in cabin interiors

Test Chamber for Formaldehyde Testing According to DIN EN 717-2

- Test space volume 4 l (9)
- Water temperature control unit with thermostat and pump (3,4,5)
  - constant 60 °C
- Air conditioning (17,18,19)
  - free of formaldehyde
  - dew point approx. -6 °C
- Manual control valve for gas analysis (6)



Flow Diagram of Emission Test Chamber



[Source: VDA 276]

EmissionEvent WKE 1000

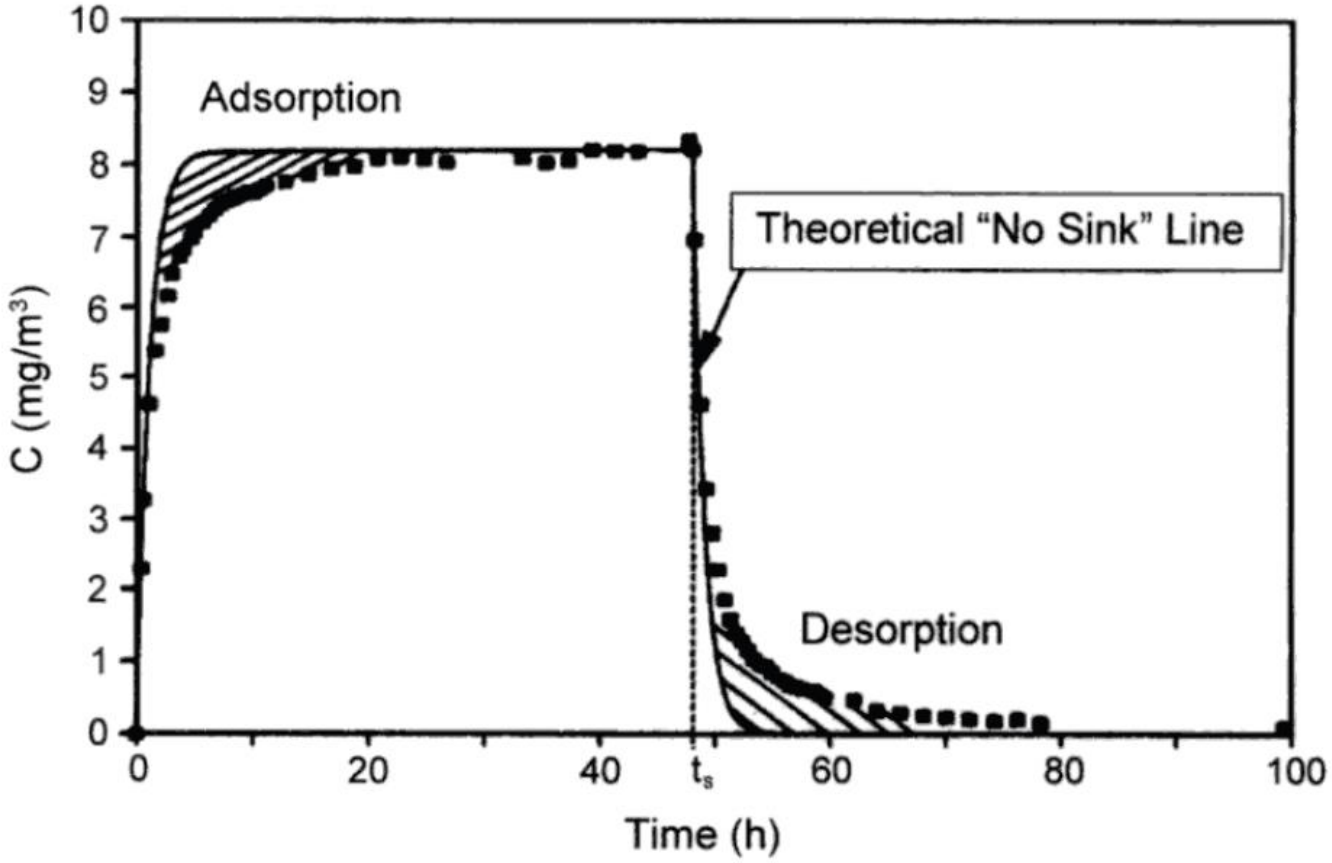
Challenges

- Design
  - walls
  - components
  - volume compensation
- Environment
  - fresh air
  - compressed air (free of oil and dust)
  - water for humidification
  - nitrogen
- Cleaning
  - grooming
  - desorption
- User

Background Emissions

- Many standards require a background emission of the system of
  - < 20 µg/m³ TVOC
  - < 2 µg/m³ for individual VOC
- Others require
  - < 50 µg/m³ TVOC
  - < 5 µg/m³ for individual VOC
- When testing for formaldehyde, the background emission must be
  - < 6 µg/m³ formaldehyde
- SHED tests require a background emission of
  - < 1 ppm

Test to Determine the Sink Effect



In a good chamber the theoretical curve and the measured curve are nearly equal.

[Source: Tichenor et al., Indoor Air 1 (1), 1991, p. 23-35]

Requirements in Test Chambers for VOC Measurements

- May not emit VOCs
  - shaft bushings and bearings must be designed accordingly
- Adsorption and absorption of VOCs should be minimized
  - small surface (preferably smooth material)
  - sealed surface
  - metals shall be preferred to plastics
- Permeation tight
- Easy to clean
  - mechanical cleaning
  - desorption at T > 200°C

Test Space Cleaning

- Mechanical cleaning
  - Water and alkaline detergent
  - Wipe with very volatile solvents (e.g. distilled water and alcohol)
  - Steam cleaner (oil-free) with osmotic water

- Thermal desorption cleaning
  - Heating up the test space for several hours to > 200 °C
  - The higher the temperature, the better/faster the desorption
  - Not all components tolerate the heat exposure well

Volume/Pressure Depending on Temperature

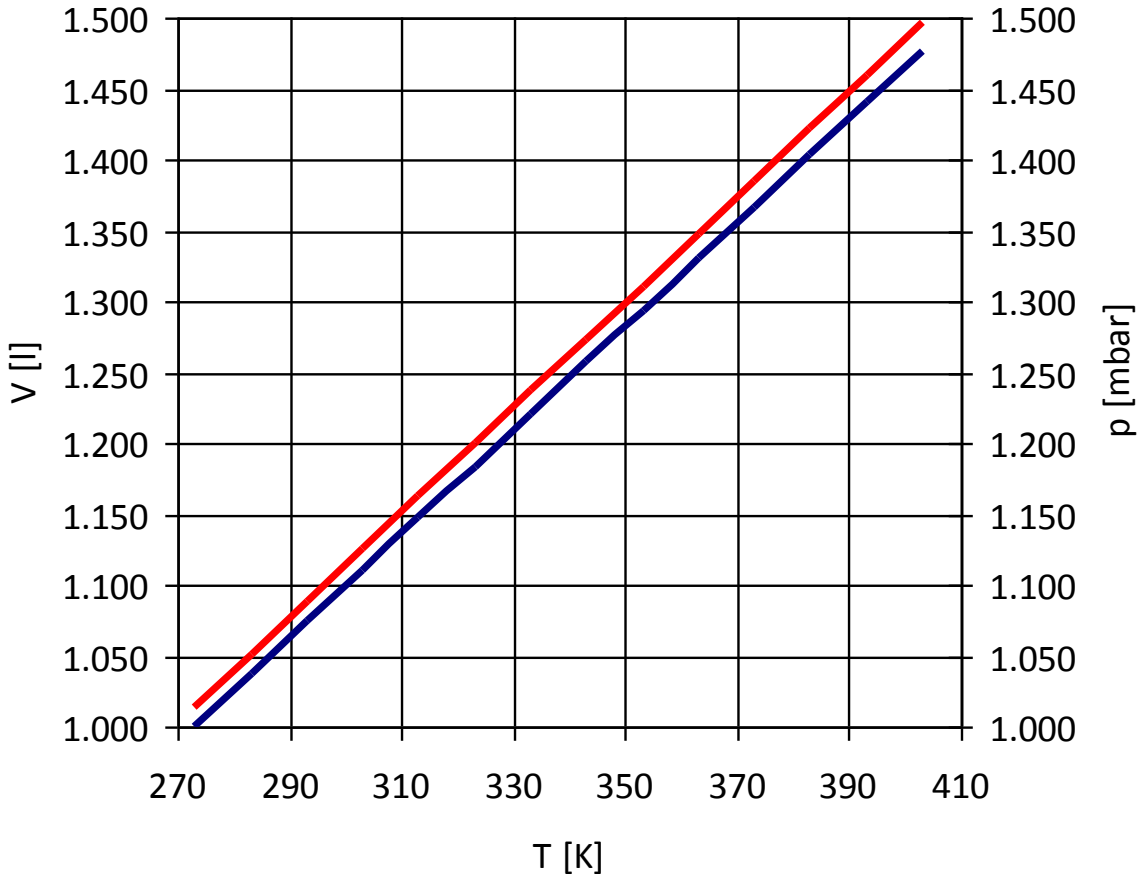
- Assumption of an ideal gas:
- constant pressure

$$V = \frac{n R T}{p}$$

- constant volume

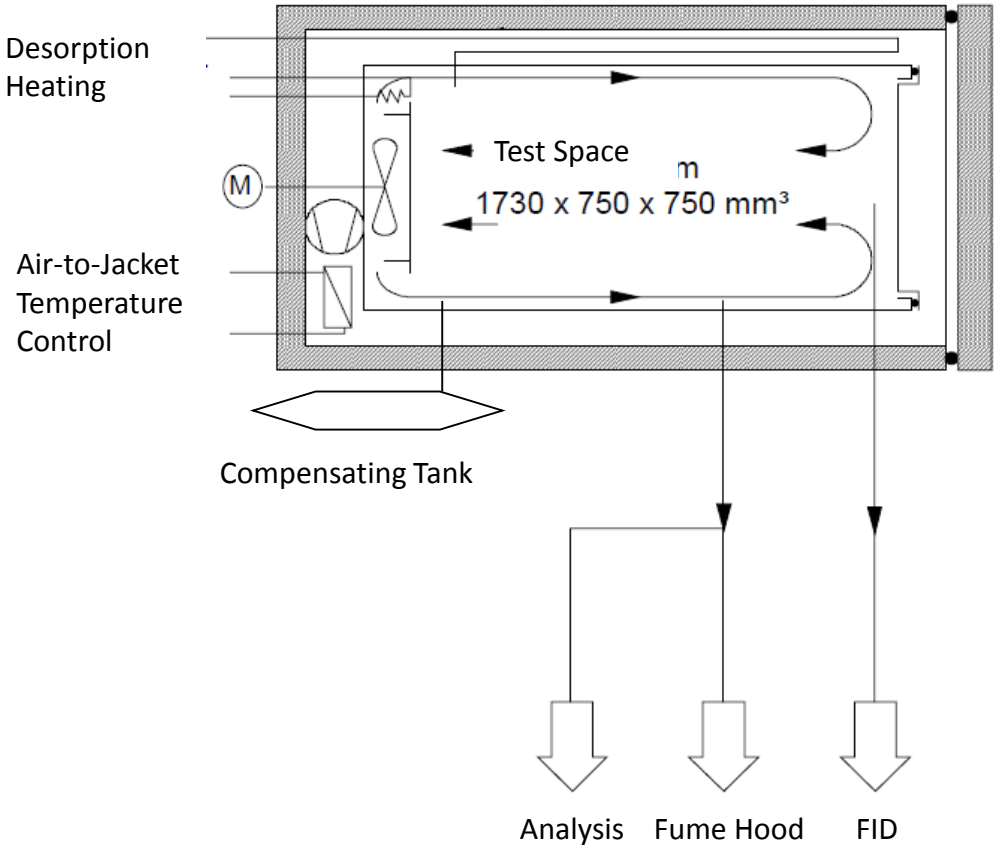
$$p = \frac{n R T}{V}$$

- V = volume [l]
- n = quantity of material [mol]
- R = universal gas constant [J/mol K]
- p = pressure [mbar]



Volume Control Via Compensation Tank

- Chamber is connected to an external bag
  - $p_i = p_o$
  - n = constant
- Usually bag is made of PVF
  - inert material
  - stable up to 130 °C
  - not 100% permeation tight



Requirements for Supply Media

- Also by supply media VOCs can be brought into the test space, which are not caused by specimen.

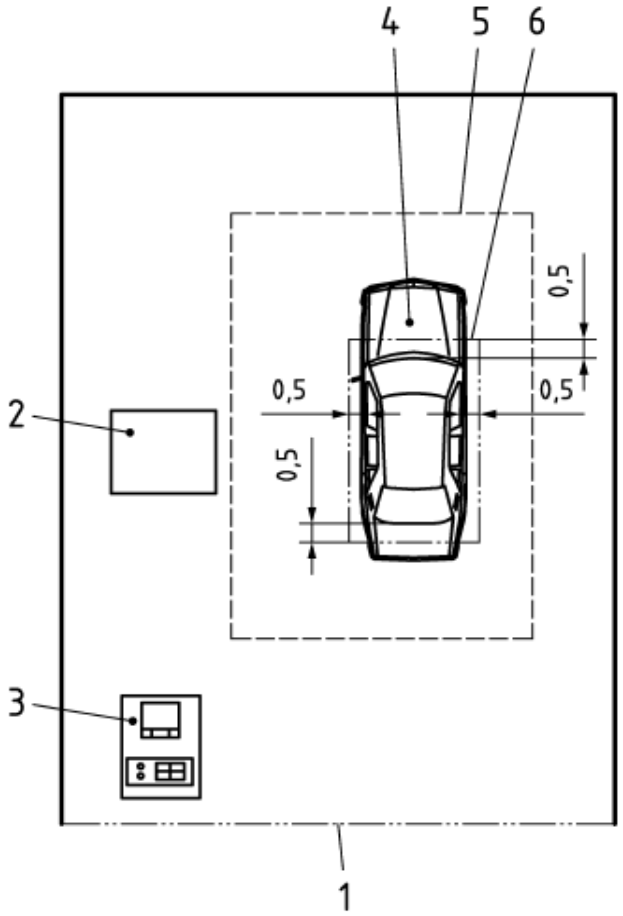
- ➢ Use of compressed air filters with residual concentrations c(HC) < 0.003 mg/m³ and particle concentrations < 0.01 mg/m³
- ➢ Nitrogen from central supply (see above)
- ➢ Nitrogen from bottle quality 5.0 or higher (pure gas 99,999990%)
- ➢ Water for dew point bath should be distilled water

Challenge Automotive Interior Testing According to DIN ISO 12219-1

- Requirements for the chamber as such are very low
  - Tiled area with heating and air condition is sufficient
  - 23 °C ± 2 °C
  - 50 % r. H. ± 10 % r. H.
  - < 20 µg/m³ for single VOC and < 200 µg/m³ TVOC
- Challenge: to reach limit values also with vehicle in the chamber
- VOC emissions in the automotive interior:
  - New car: > 4.000 µg/h
  - After 20 days of ageing: > 800 µg/h
- Values tend to be even higher, since plastic parts are used in the bodywork and also the wheels and the fuel system potentially emit VOC

Challenge Automotive Interior Testing According to DIN ISO 12219-1

- VOC emissions of new cars are very high
  - Constant purging of the chamber with clean air is required
  - Location of the test chamber is critical
  - Closed area with predefined environmental influences such humidity



1 Gate of vehicle test chamber
2 Sample device
3 Control and data processing equipment
4 Test vehicle
5 Test space
6 Irradiation area
(all figures in m)

User Competence

- VOC measurement is „Science“
  - One need to know what to do
  - Understanding of the system must be existing
- Even the simplest errors have a large effect on the results, for example:
  - Used filter cartridges
  - Improper handling
  - Incorrect sample preparation
- Interpretation of the measurement results of GC/MS, FTIR, etc. takes a lot of experience

➢ Only well trained personnel should work with VOC measurement equipment

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