Alternative Method of Analysing Odour Intensity

Method of static gas dilution in the head space flask

Many possible applications • By Gerd Scharfenberger

Odour is not a physical dimension, but a sensory perception and thus not directly measurable. Odour can be qualitatively and quantitatively characterised. The qualitative assessment in particular is very individual and closely inked with sensitivity (7).

And apparatus is available to assist in the analysis of odour intensity. The values given in the literature fluctuate widely. Among the instruments used are - so called olfactometers, constructed to VDI regualtion 3881. These devices operate by dynamic dilution, that is a gas jet pump is used to mix an odour intensive stream of gas with a constant, neutral-smelling stream of air. This mixture is tested by smelling and then progressively diluted until the odour can no longer be delected (1). Using these devices levels of delution for extemal and internal air can be set fromm 1:l to 1:10,000. In such cases the olfactometer has proved inself in practice. As a general rule an olfactometer requires an volume of air at least 6 litres.

For sensory testing of solids, there trends to be in practice little test material available. A hundred square centimeters of board or several grams of granular plastic material are often not sufficient to generate an adequate level of odour in the volume of 6 litres mentioned above. If a large number of samples is to be tested for odour such as, for example, in production control problems arise with the time required to carry out the tests.

A current method is to keep several grams of the solid or a defined area i a 250 ml ground wide neck bottle at a temperature of 40°C for two hours. The sample is then assessed by being smelled at room temperature. For this type of odour resting we have developed a method of analysing the intensity of the odour

Method of Measuring

The first dilution experiments were carried out with precision gas injectors and plunger-type testers ("Kolbenprober"). All the experiments carried out using these dilution systems failed on reproducibility. The experiments were modified a number of times, but no significant improvements were obtained.

The best reproducibility with small amounts of sample material was finally obtained by another technique, static gas dilution.

The sample was placed in a 100 ml head space flask. Nitrogen was then added via a metering loop to generale 1 bar excess pressure in the flask. The pressure was measured using an electronic manometer. The pressure was then released with the aid of a metal hypodermic needle with an attached nose mask and the odour intensity was determined using a mask placed over the nose. The pressure was relieved in stages until the odour could no longer be detected (now, look up to Olfactomat - www....). The nitrogen could also be replaced by synthentic air. The odour tests were carried out with six people.

While in the first dilution experiments with the precision gas injectors, the spread of the results obtained was as much as three evaporation levels, with static dilution broad agreement was reached by the testers. If variations occured these were normally only of one extraction step. In several of the experiments the static gas dilution was checked in parallel after each dilution step on a gas chromatograph. There was a good corrlation between the computed values and those obtained by analysis.

Equipment and Aids

For the analysis of odour intensity nitrogen (degree of purity 99.999 percent) is required. If necessary the nitrogen can be repurified by filtering.

The 100 ml head space flask contains an aluminium coated septum. (The tests can also be carried out in 250 ml head space flasks. In this case there is a greater volume available for the odour testing). On safety grounds this flasks is placed in a metal basket Preliminary experiments showed that the flask can withstand 3 bar excess pressure. For safety, the head space flask can be checked for stress in the glass with a polaristion device before the odour testing.

For the nitrogen bottle a precision pressure reducing valve with a 1/8 inch connection piece is required, to which the merering loop is attached with a threaded joint. This is a high grade steel capillary tube with a connected hypodermic needle. The hypodermic needle has a laterally positioned hole so that particles from the septum cannot enter the needle. The pressure in the injektion flask is measured by means of an electronic manometer (effective range up to 0,01 bar). For the odour testing a metal hypodermic needle with a laterally positioned hole and attached glass nose mask are also required. The measuring eauipment described here is currently undergoing technical development.

The head space flasks can also be heated. For this a thermoblock was prepared. This raises the limit of detection for samples with difficult volatility.

For solids experimentes must be carried out beforehand to determine the themperature and the time for equilibration. These can vary from material to material.

With such materials it is not only possible to determine the intensity of the odour from the number of extraction steps, bus also to determine alterations in the nuance of the odour during the individual extraction steps. It is therefore possible to de-termine a profile for the odour, which is however individual to the parlicular person.

Examples of Determination of Odour Thresholds (GS)

To determine the odour threshold of toluol, 0,5 µl were injected into a 100 ml head space flask which was placed in a metal basket. The method of measuring described above was then applied.

With toluol it was still possible to discern a clear odour at the eighth pressure relief step. In the ninth step the odour was no longer discernible. The odour threshold value was found from the following equation:

$$GS = \frac{X}{2^n}$$

GS = odour threshold in mg/m³

X = quantity in mg/m³

n = number of extractions

For toluol the odour threshold was 17 mg/m³.

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3 Table: Odour threshold values determined using the head space flask (4)

Solvent	Level (µl) (1)	Concen-tration (mg/m³) (2)	Number of extractions (3)	Experimental odour thresehold (mg/m³) (4)	Odour threshold, values in the literature (mg/m³) (5)
Toluol	0,5 µl	4360	8	17	0,6 to 153
Ethylacetate	1,0 µl	9000	6	141	0,2 to 183
Denatured ethanol (2% with cyclohexane)	1,0 µl	7900	6	123	
Ethanol p.a.	1,0 µl	7900	3	988	19 to 672
Methylketone	1,0 µl	8060	6	126	30 to 80
Isopropanol	1,0 µl	7850	4	491	80 to 250
Benzine 80/110	1,0 µl	7160	4	448	3300
Xylol	0,5 µl	4325	10	4	1 to 100
Isopropylacetate	1,0 µl	8700	7	68	140
Methanol	1,0 µl	7900	2	1976	6,6 to 7800
n-propanol	1,0 µl	8035	9	16	30 to 250
n-butanol	1,0 µl	8100	14	0,5	0,36 to 77
n-butylacetate	1,0 µl	8820	11	4	0,35 to 48
Methylacetate	1,0 µl	9270	4	579	154 to 550

The values in the literature vary between 0,6 and 153 mg/m³. The threshold values for a number of other solvents, apart from toluol, were also determined (c.f. table).

In addition to the analysis of individual solvents, determination of the odour thershold value for mixtures of solvents is particularly interesting.

The testing method can be extended by the use of 3 special sampling technique.

The bottles can be evacuated and remain under vacuum for days. These evacuated bottles provide an elegant method of taking external and internal air samples on the spot. A good vacuum can be easily detected by relieving the pressure under water. Only a small air buddle should remain. The evacuated head space flak for measurement of odours can be used to extract the air even from materials enclosed in packaging. For this a double sided hypodermic needle is required. This is two metal hypodermic needles soldered together at the Luer connector.

Special Applications

The apparatus can also be used for the production of calibration gas. By dosing via micro-litre injectors (control by means of an analytical balance) and using the appropriate number of extraction steps, calibration gas or mixtures of calibration gases can be produced very quickly in very low concentrations. The method is also quick and cheap.

Since the samples for the extraction steps are under excess pressure, the escaping gas can also be subjected to wet chemical investigation. Small absorption vessels with a hypodermic needle are particularly suitable for this (for the detection of aldehydes, hydrogen chlonde, nitrous gases, amnionia, for example).

Good results were also given using Dräger tubes. The Dräger tube is attached to hypodermic needle by means of a short hoses connection. Since there are a number of testing tubes, the investigation can be extended to a broad range of inorganic and organic substances (3).

The method described here for analysis of odour intensity gives reproducible results with very little aparatus. Since 2,5 l pressure gas bottles are also available , the apparatus is portable when packed in a suitable case.

I would like to thank Mrs. Elisabeth Kopp and Mrs. Heide Lehmler for their collaboration in these wide ranging experiments.

Literature:

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