The MAK Collection for Occupational Health and Safety

1,3-Dioxolane – Determination of 1,3-dioxolane in workplace air using gas chromatography (GC-FID)

Air Monitoring Method – Translation of the German version from 2022

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Keywords

1,3-dioxolane; air analyses; analytical method; workplace measurement; hazardous substance; gas chromatography; GC-FID; Chromosorb 106; thermal desorption

Abstract

This analytical method is a validated measurement procedure for the determination of 1,3-dioxolane [646-06-0] in workplace air in a concentration range of one tenth up to twice the currently valid Occupational Exposure Limit Value (OELV) in Germany of 150 mg/m^3. For sampling, a defined volume of air is drawn through a sorbent tube (for thermal desorption) filled with Chromosorb 106. The flow rate is set to 5 ml/min and sampling is performed over 2 hours (which corresponds to a sampling volume of 600 ml). The samples to which cyclooctane is added as internal standard are thermally desorbed. The quantitative determination is based on a calibration function, whereby the 1,3-dioxolane concentration of the calibration standard is plotted against the intensities, calculated over the internal cyclooctane standard. The limit of quantification is 12.9 mg/m^3 based on an air sample volume of 600 ml. The mean recovery is 97% and the expanded uncertainty for the validation range of 16.3 to 293 mg/m^3 is 11.5 to 12.1%.

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Method number	1
Application	Air analysis
Analytical principle	Gas chromatography (GC-FID)

1 Characteristics of the method

Precision:	Standard deviation (rel.):	$s = 1.4$ to 1.8%
	Expanded uncertainty:	$U = 11.4$ to 12.1%
	in the concentration range from 16.3 to 293 mg/m^3 and for $n = 6$ determinations	
Limit of quantification:	7.76 µg/l	
	12.9 mg/m^3 for an air sample volume of 600 ml and a sampling period of 2 h	
Recovery:	$\eta = 0.96$ to 0.98	
Sampling recommendations:	Air sample volume:	600 ml
	Sampling period	120 min
	Volumetric flow rate:	5 ml/min
	For short-term measurements:	15 min; 40 ml/min

2 Description of the substance

1,3-Dioxolane [646-06-0]

1,3-Dioxolane (also known as: glycol methylene ether, glycolformal, 1,3-dioxacyclopentane; Figure 1) is a readily volatile, colourless liquid that is miscible with water. It is used for instance in coating products, adhesives and sealants, cleaning products as well as in products for the treatment of leather (ECHA 2020 c). Furthermore, it is used in the production of modified polyoxymethylenes and in paint strippers. 1,3-Dioxolane is an aprotic solvent and is used in formulations, production processes and as a reactant. It is used in coating processes, as it is capable of rapidly dissolving polar polymers such as polyester, epoxides and urethanes due to its small molecular size, thus enabling their fast application (ECHA 2020 b). As a paint stripper 1,3-dioxolane is often used in combination with dimethoxymethane [109-87-5] (AGS 2006; RPA 2007).

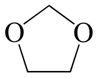

Fig. 1 Structural formula of 1,3-dioxolane

Due to concerns regarding consumer usage, exposure of sensitive population groups, large (aggregated) tonnage and widespread use as well as possible hazards caused by mutagenic and reprotoxic properties, 1,3-dioxolane was included in the Community Rolling Action Plan (CoRAP) by the EU in 2016. A re-evaluation of the effects of the substance on human health and the environment is being carried out and, if necessary, follow-up measures will be initiated (ECHA 2020 b).

1,3-Dioxolane has an Occupational Exposure Limit Value (OELV) of 150 mg/m^3, the short-term exposure limit is assigned to Peak Limitation Category II with an excursion factor of 2 (AGS 2020). In the list of MAK and BAT values, the MAK value for 1,3-dioxolane is the same as the OELV (DFG 2020). Substance data for 1,3-dioxolane can be found in Table 1.

Tab. 1 Substance data for 1,3-dioxolane (ECHA 2020 a)

Name	1,3-Dioxolane
CAS No.	646-06-0
Molar mass [g/mol]	74.08
Physical state at 20 ℃	liquid
Density at 20 ℃ [g/cm^3]	1.06
Vapour pressure at 20 ℃ [Pa]	105
Melting point [℃]	−95
Boiling point at 1013 hPa [℃]	76
Flash point [℃]	2.5
Type and category of hazard (according to GHS)	Reprotoxic (Repr. 1B) Flammable liquid (Flam. Liq. 2) Serious eye damage/eye irritation (Eye Irrit. 2)
Toxicological effect	3000 mg/kg body weight (LD$_{50}$, rat oral) 68.4 mg/l/4 h (LC$_{50}$, rat inhalation: vapour)
Evaluation criteria	
OELV, Germany (AGS 2020)	150 mg/m^3
MAK value, Germany (DFG 2020)	150 mg/m^3

3 General principles

This analytical method permits the determination of 1,3-dioxolane in workplace air in a concentration range of one tenth up to twice the currently valid OELV of 150 mg/m^3 (AGS 2020). The peak limit with an excursion factor of 2 can also be monitored (AGS 2020; DIN 2015).

A sampling pump draws a defined volume of air from the breathing zone through an adsorption tube filled with Chromosorb 106. The 1,3-dioxolane is adsorbed onto the collection phase in this manner. After thermal desorption and gas chromatographic separation, detection is performed using a flame ionisation detector (FID). Quantitative evaluation is based on multiple-point calibration with an internal standard.

4 Equipment, chemicals and solutions

4.1 Equipment

For sampling:

- Adsorption tubes made of stainless steel (6.3 mm × 90 mm, 5 mm ID), packed with 370 mg of Chromosorb 106 (e.g. from PerkinElmer LAS, 63110 Rodgau, Germany)
- Pump for personal sampling, suitable for a flow rate of 5 ml/min and 40 ml/min (e.g. Modell LFS-113DC, from Gilian Inc., Carlsbad, California, USA)
- Sealing caps (e.g. Swagelok with PTFE seals, from Swagelok Company, Solon, Ohio, USA)
- Sampling cap made of aluminium, which has a hole with an ID of 1 mm

For sample preparation and analysis:

- Gas chromatograph with thermal desorber, FID and evaluation system (e.g. Clarus 680 with FID, from PerkinElmer LAS, 63110 Rodgau, Germany)
- Capillary columns (e.g. DB-5 and DB-1701: 30 m, film thickness of 1.0 µm and ID of 0.32 mm, from Agilent, 76337 Waldbronn, Germany)
- Flow meter or stopwatch and soap bubble flow meter (e.g. TSI model 4100, from TSI Inc., Shoreview, Minnesota, USA)
- Direct-reading measurement device, photo-ionisation detector (e.g. MultiRAE 3000, from RAE Systems Inc., San José, California, USA)
- Dynamic test gas facility
- Syringe pump (e.g. Titronic 110 plus, TA 01 with 1-ml syringe, from SCHOTT, 40880 Ratingen, Germany)
- Analytical balance (e.g. Modell AT250, from Mettler GmbH, Greifensee, Switzerland)
- 10-ml amber glass bottles with Mininert valve
- Microlitre syringes, 1000 µl, 2500 µl (e.g. from Hamilton, 52379 Langerwehe, Germany)
- Dispenser (e.g. Dispensette Digital 2–10 ml, from Brand, 97877 Wertheim, Germany)

4.2 Chemicals

- 1,3-Dioxolane, purity ≥ 99.9% (e.g. from Merck, Darmstadt, Germany, Order No. 803553)
- Dimethoxymethane, purity ≥ 99.5% (e.g. from Merck, Darmstadt, Germany, Order No. 806017)
- 2-Isopropoxyethanol, purity ≥ 99% (e.g. from Merck, Darmstadt, Germany, Order No. 107891)
- Cyclooctane, purity ≥ 99% (e.g. from Merck, Darmstadt, Germany, Order No. C109401)
- Helium 5.0 (carrier gas)
- Nitrogen 5.0
- Hydrogen 5.0
- Synthetic air (free of hydrocarbons)

4.3 Solutions

The following stock solution is prepared using the chemicals listed in Section 4.2:

Stock solution (489.23 g of 1,3-dioxolane/l in dimethoxymethane/2-isopropoxyethanol):

Microlitre syringes are used to fill 3 ml of 1,3-dioxolane with 2.5 ml of dimethoxymethane and 1 ml of 2-isopropoxyethanol into a 10-ml amber glass bottle. The bottle is sealed with a Mininert valve and briefly shaken. Filling is controlled using a balance, which is accurate to 10 µg, whereby the deviation should be less than 1%.

The density of the stock solution is calculated on the basis of the sum of the individual masses and the sum of the individual volumes. A possible contraction in volume is not taken into consideration. The error between the calculated and the actual density is negligible due to the small quantities used.

Different solvents were used to prepare the stock solutions, on the one hand to achieve a dilution of the components and on the other to measure possible interference that might occur in the matrix.

Tab. 2 Composition of the stock solution and in the test gas

Substance	Density [g/ml]	Volume [ml]	Stock solution [g/l]	Test gas [mg/m^3]
1,3-Dioxolane	1.06	3	489.23	489.23
Dimethoxymethane	0.86	2.5	330.77	330.77
2-Isopropoxyethanol	0.90	1	138.46	138.46

4.4 Test gas and reference standards

In the case of thermal desorption processes it is advisable to calibrate with test atmospheres. There are different procedures for the preparation of test gases (Flammenkamp and Risse 1994). Furthermore, spiking with 1 µl of calibration solutions in methanol is possible in each case. The characteristic data of the method were checked and are comparable.

One possibility of producing test gases is by continuous injection (Figure 2).

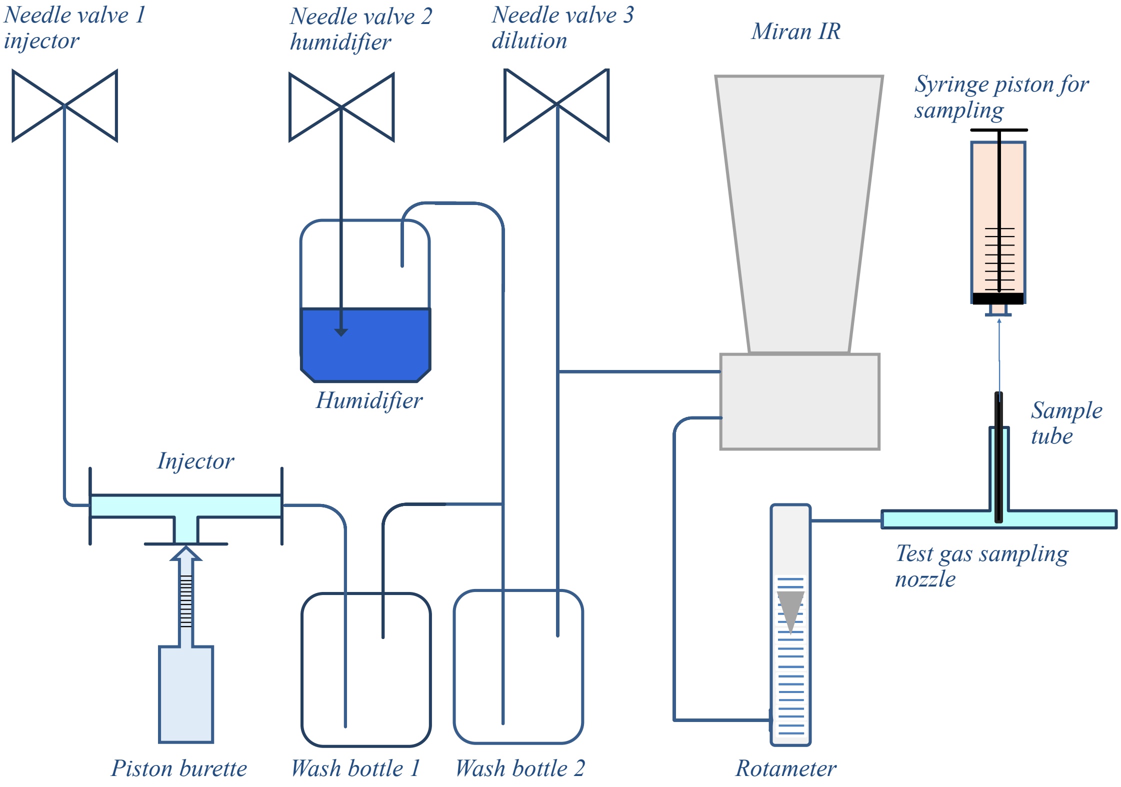

Fig. 2 Schematic illustration of the dynamic test gas facility

The stock solution (Section 4.3, Table 2) or the internal standard (ISTD) are continuously injected at a rate of 150 µl/h into an injector nitrogen gas flow of 250 ml/min in a dynamic test gas facility. The calculated concentrations of the individual components are obtained by diluting the resulting total test gas flow of 2500 ml/min with a nitrogen gas flow of 2250 ml/min. The concentration of the stock solution in the test gas is 1 µl/l.

Adsorption tubes packed with Chromosorb 106 are connected to the suction side of the syringe injection pump and aliquots in the range of 15 to 370 ml of this calibration atmosphere are drawn through 20 sample tubes (determination in duplicate, 10-point calibration). Table 3 lists the corresponding masses of the individual calibration points. The stated doses and the volumetric flow rates of the injector have proved to be the most stable and without fluctuations for the production of test gases in the apparatus.

A test gas with a relative humidity of 80% can be produced using the humidifier by adding it with a volumetric flow rate of 2000 ml/min (with a relative humidity of 100%) through the pertinent needle valve. The diluting nitrogen gas flow is then lowered to 250 ml/min, in order to maintain the total test gas flow rate of 2500 ml/min.

Tab. 3 Test gas volumes and calibration masses

Test gas [ml]	1,3-Dioxolane [µg]	Dimethoxymethane [µg]	2-Isopropoxyethanol [µg]	ISTD gas cyclooctane [ml]	[µg]
15	7.34	4.96	2.08	30	25
50	24.46	16.54	6.92	30	25
90	44.03	29.77	12.46	30	25
130	63.60	43.00	18.00	30	25
170	83.17	56.23	23.54	30	25
210	102.7	69.46	29.08	30	25
250	122.3	82.69	34.62	30	25
290	141.9	95.92	40.15	30	25
330	161.4	109.2	45.69	30	25
370	181.0	122.4	51.23	30	25

Each sample that is spiked with the stock solution by means of the test gas facility is then loaded with 30 ml of ISTD atmosphere (with a concentration of 1 µl/l ≙ 0.834 µg/ml) in the test gas facility.

5 Sampling and sample preparation

5.1 Pre-treatment of the adsorption tube

Before use, the adsorption tubes are heated for approx. 15 minutes in the thermal desorber at 170 °C in a stream of inert gas (e.g. helium) and then tested for blank values. The tubes are then sealed with Swagelok caps with Teflon seals and stored at room temperature.

5.2 Sampling

Pre-treated adsorption tubes are used for sampling (Section 5.1). The sealing caps are removed for sampling and the adsorption tube is connected to the sampling pump. An aluminium sampling cap with a 1-mm hole is fitted on the intake side of the tube in order to minimise additional substance adsorption by diffusion.

In order to ensure that the values measured during sampling are not outside the calibration range, a sample volume of 600 ml is desirable. This is achieved by adjusting the volumetric flow rate and the sampling period. The volumetric flow of the pump should be set to 5 ml/min for a 2-hour sampling period. For short-term measurements of 15 min, the volumetric flow rate of the pump should be set to 40 ml/min. The volumetric flow rate must be checked before and after sampling. If the deviation from the adjusted flow rate is greater than ± 5%, it is advisable to repeat the measurement (DIN 2014). The parameters relevant to the determination of the concentration in air (air sample volume, temperature, air pressure and relative humidity) are documented in the sampling record.

Sampling is carried out in the breathing zone and can be performed as stationary or personal sampling, whereby the aperture of the adsorption tube must not be obstructed. On completion of sampling, Swagelok caps with PTFE seals are used to seal the loaded adsorption tubes. The samples are transported and stored at room temperature.

6 Operating conditions

Gas chromatograph

Apparatus:	Clarus 680 gas chromatograph with FID, PerkinElmer LAS
Column:	Material: Fused silica capillary Stationary phase: DB-5 and DB-1701 Length in each case: 30 m (connected in series) Inner diameter (ID): 0.32 mm Film thickness: 1.00 µm
Detector:	FID
Detector temperature:	300 ℃
Detector gases:	Hydrogen (40 ml/min), synthetic air (400 ml/min)
Heating rate:	5 min at 30 ℃, increased by 40 ℃/min up to 200 ℃, 3 min

Thermal desorber

Apparatus:	Turbomatrix 650 (PerkinElmer LAS)
Desorption temperature:	170 ℃
Desorption time:	15 min
Conditioning flow rate:	50 ml/min
Valve temperature:	200 ℃
Temperature of transfer line:	200 ℃
Transfer line:	1.5 m of deactivated, blank capillaries (ID of 0.32 mm)
Cold trap (adsorption):	5 ℃
Cold trap (injection):	250 ℃
Cold trap filling:	Air Toxics
Heating rate:	99 ℃/s
Carrier gas / column flow:	Helium, 2.2 ml/min, constant flow rate
Split (before the cold trap):	80 ml/min (input split)
Desorption flow over the cold trap:	10 ml/min (desorption flow)
Split (after the cold trap):	20 ml/min (output split = recollect)
Purge drying phase:	1 min at room temperature at 50 ml/min

If other thermal desorption devices are used, the instrumental conditions must be adapted accordingly.

Under the conditions given here, the retention time for 1,3-dioxolane is approx. 8.90 minutes.

7 Analytical determination

The loaded adsorption tubes are heated in the thermal desorber, where the substances are transferred by means of a carrier gas into a cold trap packed with Air Toxics. After complete desorption from the adsorption tube, the cold trap is rapidly heated, so that the substance mixture is transferred to the separation column as a narrow substance plug. If the sample volume of 600 ml is adhered to, the measurement range of one tenth up to twice the limit value is covered.

8 Calibration

The calibration solutions described in Section 4.4 are used to obtain the calibration function. The samples are analysed under the operating conditions described in Sections 6 and 7 and the resulting peak areas are plotted versus the corresponding calibration masses with respect to the ISTD.

The calibration functions are linear in the investigated concentration range and should be regularly checked during routine analysis. The calibration must be performed anew if the analytical conditions change or the quality control results indicate that this is necessary.

9 Calculation of the analytical result

Based on the peak areas the corresponding mass X in µg per sample is obtained from the calibration function. The corresponding mass concentration (ρ) is calculated according to Equation 1 as follows:

$$\rho = \frac{X}{V \times \eta} \tag{1}$$

Equation 2 enables for the calculation of the value at 20 °C and 1013 hPa:

$$\rho_0 = \rho \times \frac{273 + t_a}{293} \times \frac{1013}{p_a} \tag{2}$$

where:

ρ	is the mass concentration of the substance in the air sample in mg/m^3 at t_a and p_a
ρ_0	is the mass concentration of the substance in mg/m^3 at 20 °C and 1013 hPa
X	is the mass of the substance in the analytical sample in µg
V	is the air sample volume in l (calculated from the volumetric flow rate and the sampling period, in this case 0.6 l)
η	is the recovery
t_a	is the temperature during sampling in °C
p_a	is the atmospheric pressure during sampling in hPa

10 Reliability of the method

The characteristics of the method were calculated as stipulated in DIN EN 482 (DIN 2015), DIN EN ISO 22065 (DIN 2021) and DIN 32645 (DIN 2008). A complete validation of the method was carried out. For this purpose, test gas mixtures in concentrations of one tenth, once and twice the OELV at relative humidities of 0 and 80% were prepared in a dynamic test gas facility.

10.1 Precision and expanded uncertainty

The precision was determined by carrying out three experimental series with 1,3-dioxolane concentrations in the range of one tenth to twice the OELV. For this purpose, the adsorption tubes were spiked with the respective concentrations as described in Section 4.4. Six tubes per concentration were spiked. Two experimental series were carried out. The first was run in the absence of any humidity for all three concentrations; the second at 80% relative humidity for one tenth and twice the OELV (5 spiked tubes in each case). The analytical determination was carried out according to Sections 6 and 7.

The expanded uncertainty was estimated taking all relevant influencing factors into consideration as stipulated in DIN EN 482 (DIN 2015) and DIN EN ISO 22065 (DIN 2021) and calculated using the IFA-Excel-Sheets (IFA n.d.) to yield the expanded uncertainty. It lies between 11.4 und 12.1% for 1,3-dioxolane. The results are summarised in Table 4.

10.2 Recovery

The recoveries for 1,3-dioxolane were determined in the absence of any humidity for one tenth, once and twice the OELV. For this purpose, the adsorption tubes were spiked with corresponding concentrations of 1,3-dioxolane. Six tubes per concentration were spiked. The mean recoveries were between 95.6 and 98.0%. The influence of the humidity was checked by also spiking tubes with one tenth and twice the OELV at 80% humidity (n = 5). The mean recovery for the low concentration was 98.4% and 100.0% for the high concentration. The setup of the apparatus, the operating conditions and the analysis are described in Sections 4.4, 6 and 7. The results are listed in Table 4. No influence on the recovery due to the humidity could be detected.

Tab. 4 Recoveries, relative standard deviation and expanded uncertainty

Concentration [mg/m^3]	Recovery at 0% humidity [%]	Rel. standard deviation [%]	Expanded uncertainty U [%]	Recovery at 80% rel. humidity [%]
16.31	95.6	1.64	12.1	98.4
146.7	96.6	1.79	11.7	–
293.5	98.0	1.45	11.4	100.0

10.3 Limit of quantification

The limit of quantification was calculated according to the calibration line method as stipulated in DIN 32645 (DIN 2008) with a statistical certainty of P = 95% and k = 3. It is 7.76 µg of 1,3-dioxolane, absolute.

The relative limit of quantification is 12.9 mg/m^3 for an air sample volume of 600 ml (equivalent to a sampling period of 2 hours at a volumetric flow rate of 5 ml/min).

10.4 Capacity of the adsorbent

The capacity of the adsorbent was checked by spiking collection tubes at 2.4 times the OELV (353 mg/m^3) at a volumetric flow rate of 46 ml/min (almost ten times the volumetric flow rate that should be used during sampling) for 2 hours. A direct-reading measurement device was used, which was connected by a tube to the outlet of the adsorbent tube, to check whether a breakthrough occurred in real time. No breakthrough was observed over a period of 120 minutes. Similar experiments with 80% relative humidity were also carried out. No breakthrough over a period of 120 minutes with constant loading was observed in this case either.

10.5 Storage stability

As described in Section 4.4, 12 adsorption tubes each were spiked with 16.31 mg/m^3, 146.7 mg/m^3 and 293.5 mg/m^3 of 1,3-dioxolane in the test gas facility to determine the storage stability. The sample carriers were sealed with Swagelok caps and stored at room temperature. After 1, 2, 3 and 4 weeks, three samples of each concentration were analysed as described in Sections 6 and 7. The results can be found in Table 5. Storage stability of the adsorption tubes over a period of four weeks was checked and confirmed.

Tab. 5 Results of the recovery at 0% humidity after 1, 2, 3 and 4 weeks

Concentration [mg/m³]	1 day [%]	1 week [%]	2 weeks [%]	3 weeks [%]	4 weeks [%]
16.31	95.6	101	100	102	98.3
146.7	96.6	92.0	91.3	92.5	91.6
293.5	98.0	96.4	95.0	95.5	94.3

In addition, the influence of the humidity on the storage stability was checked. For this purpose, 10 adsorption tubes were again each spiked with 16.31 mg/m^3 (0.1 OELV) and 293.5 mg/m^3 (2 OELV) of 1,3-dioxolane at 0% and 80% relative humidity and 5 samples were each analysed after one day and 3 weeks. The setup of the apparatus, the operating conditions and the analysis are described in Sections 4.4, 6 and 7. The samples were stored at room temperature for 3 weeks. The results can be found in Table 6. No negative influence on storage stability due to the humidity could be detected.

Tab. 6 Comparison of storage stability over three weeks at 0% and 80% humidity

	0% rel. humidity		80% rel. humidity	
Concentration [mg/m³]	**1 day [%]**	**3 weeks [%]**	**1 day [%]**	**3 weeks [%]**
16.31	95.6	98.5	98.4	101
293.5	99.8	94.1	100	97.1

10.6 Interference

The analytical procedure using gas chromatography and flame ionisation detection is specific and robust under the operating conditions stated here. There was no evidence of interference from other solvents, such as dimethoxymethane, which is often used together with 1,3-dioxolane in paint strippers.

11 Discussion

The analytical procedure described here permits the determination of 1,3-dioxolane in workplace air in a concentration range from one tenth to twice the currently valid OELV of 150 mg/m^3. The method is suitable for checking the short-term exposure limit. No negative influence on the measurement results and storage stability due to the humidity (up to 80% relative humidity) could be detected.

Notes

Competing interests

The established rules and measures of the Commission to avoid conflicts of interest (www.dfg.de/mak/conflicts_interest) ensure that the content and conclusions of the publication are strictly science-based.

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