

2-Isopropoxyethanol – Determination of 2-isopropoxyethanol in workplace air using gas chromatography (GC-FID)

Air Monitoring Method – Translation of the German Version from 2022

Keywords

2-isopropoxyethanol; air analyses; analytical method; workplace measurement; hazardous substance; gaschromatography; flame ionisation detection; GC-FID; Chromosorb 106; thermal desorption

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Abstract

This analytical method is a validated measurement procedure for the determination of 2-isopropoxyethanol [109-59-1] in workplace air in a concentration range of one tenth up to twice the currently valid occupational exposure limit value (OELV) in Germany of 44 mg/m³. 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 2-isopropoxyethanol concentration of the calibration standard is plotted against the intensities, calculated over the internal cyclooctane standard. The limit of quantification is 4.48 mg/m³ based on an air sample volume of 600 ml. The mean recovery is 92% and the expanded uncertainty for the validation range of 4.62 to 83.08 mg/m³ is 28.4%.

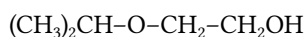
Method number	1
Application	Air analysis
Analytical Principle	Gas chromatography with flame ionisation detection (GC-FID)

1 Characteristics of the method

Precision:	Standard deviation (rel.):	$s = 2.9$ to 3.4%
	Expanded uncertainty:	$U = 24.8\%$
	in the concentration range from 4.62 to 83.08 mg/m^3 and for $n = 6$ determinations	
Limit of quantification:	$2.69 \text{ }\mu\text{g}$ absolute	
	4.48 mg/m^3 for an air sample volume of 600 ml and a sampling period of 2 h	
Recovery:	$\eta = 0.87$ to 0.97	
Sampling recommendations:	Air sample volume:	600 ml
	Sampling period:	120 min
	Volumetric flow rate:	5 ml/min
	For short-term measurements:	15 min
	Volumetric flow rate:	40 ml/min

2 Description of the substance

2-Isopropoxyethanol [109-59-1]



2-Isopropoxyethanol (2-isopropoxyethanol, ethylene glycol monoisopropyl ether, 2-(1-methylethoxy)ethanol, isopropyl glycol, 2-propan-2-yloxyethanol) is a colourless, flammable liquid with an ethereal odour that is miscible with water. 2-Isopropoxyethanol is used as a solvent in paints, varnishes and adhesives as well as in cleaning products for machines (ECHA 2022).

The physico-chemical data and the evaluation criteria for 2-isopropoxyethanol are listed in [Table 1](#).

Tab. 1 Substance data (ECHA 2022) and evaluation criteria for 2-isopropoxyethanol

Name	2-isopropoxyethanol
CAS No.	109-59-1
Molar mass [g/mol]	104.15
Physical state at 20°C	liquid
Density at 20°C [g/cm^3]	0.903
Vapour pressure at 25°C [hPa]	5.99
Melting point [$^\circ\text{C}$]	-60.2
Boiling point at 1013 hPa [$^\circ\text{C}$]	145
Flash point [$^\circ\text{C}$]	43 to 45

Tab. 1 (continued)

Evaluation criteria	
OELV, Germany (AGS 2022)	44 mg/m ³ a), 10 ml/m ³
MAK value, Germany (DFG 2022)	43 mg/m ³ , 10 ml/m ³
Peak limit category (excursion factor) (AGS 2022; DFG 2022)	I (2)

a) The discrepancy between the OELV and MAK value is due to rounding.

3 General principles

This analytical method permits the determination of 2-isopropoxyethanol in the workplace air in a concentration range of one tenth up to twice the currently valid OELV in Germany of 44 mg/m³ or 10 ml/m³ and the MAK value of 43 mg/m³ or 10 ml/m³ (AGS 2022; DFG 2022). The peak limit with an excursion factor of 2 (AGS 2022) can also be monitored (DIN 2021 a).

A sampling pump draws a defined volume of air from the breathing zone through an adsorption tube filled with Chromosorb 106. The 2-isopropoxyethanol is adsorbed onto the collection phase in this manner. After thermal desorption and gas chromatographic separation, detection is performed using a flame ionisation detector (GC-FID). A 10-point calibration with an internal standard (ISTD) is used for quantitative evaluation.

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 volumetric flow rates of 5 ml/min and 40 ml/min (e.g. Model LFS-113DC, from Gilian Inc., Carlsbad, California, USA)
- Sealing caps with PTFE seals (e.g. 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 Perkin Elmer 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 Technologies Germany GmbH & Co. KG, 76337 Waldbronn, Germany)
- Flow meter or stopwatch and soap bubble flow meter (e.g. 4100 Series, 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
- Piston burette (e.g. Titronic 110 plus, TA 01 with 1 ml syringe, from SCHOTT Instruments GmbH, 55122 Mainz, Germany)
- Analytical balance (e.g. Model AT250, from Mettler GmbH, Greifensee, Switzerland)

- 10 ml amber glass bottles with Mininert valves (e.g. bottles from Chromatographie Service GmbH, 52379 Langerwehe, Germany, with valves from Valco Instruments Co. Inc., Houston, Texas, USA)
- Microlitre syringes, 1000 µl, 2500 µl (e.g. from Hamilton Bonaduz AG, Bonaduz, Switzerland)
- Dispenser (e.g. Dispensette Digital 2 to 10 ml, from Brand GmbH + CO KG, 97877 Wertheim, Germany)

4.2 Chemicals

- 2-Isopropoxyethanol, purity $\geq 99\%$ (e.g. from Merck KGaA, 64293 Darmstadt, Germany, Order No. 107891)
- 1,3-Dioxolane, purity $\geq 99.9\%$ (e.g. from Merck KGaA, 64293 Darmstadt, Germany, Order No. 803553)
- Dimethoxymethane, purity $\geq 99.5\%$ (e.g. from Merck KGaA, 64293 Darmstadt, Germany, Order No. 806017)
- Cyclooctane, purity $\geq 99\%$ (e.g. from Merck KGaA, 64293 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 solutions are prepared using the chemicals listed in [Section 4.2](#):

Stock solution: (138.46 g of 2-isopropoxyethanol/l in 1,3-dioxolane/dimethoxymethane):

Microlitre syringes are used to fill 3 ml of 1,3-dioxolane, 2.5 ml of dimethoxymethane and 1 ml of 2-isopropoxyethanol into a 10-ml amber glass bottle via a Mininert valve and the bottle is briefly shaken.

Each dose is checked using a balance, which is accurate to 10 µg, whereby the deviations 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.

If the density is checked by weighing, then the errors in dosing and weighing could lie within the degree of deviation between the calculated and 'actual' density, in which case a possible slight contraction in volume can be considered negligible.

Various solvents are used to prepare the stock solution, on the one hand to dilute the components and on the other to take the interference, which may occur in the matrix (see [Table 2](#)), into consideration during chromatography and analysis.

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 gases 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 in each case is possible. The characteristic data of the method were checked and are comparable.

One possibility of producing test gases is by continuous injection (Figure 1).

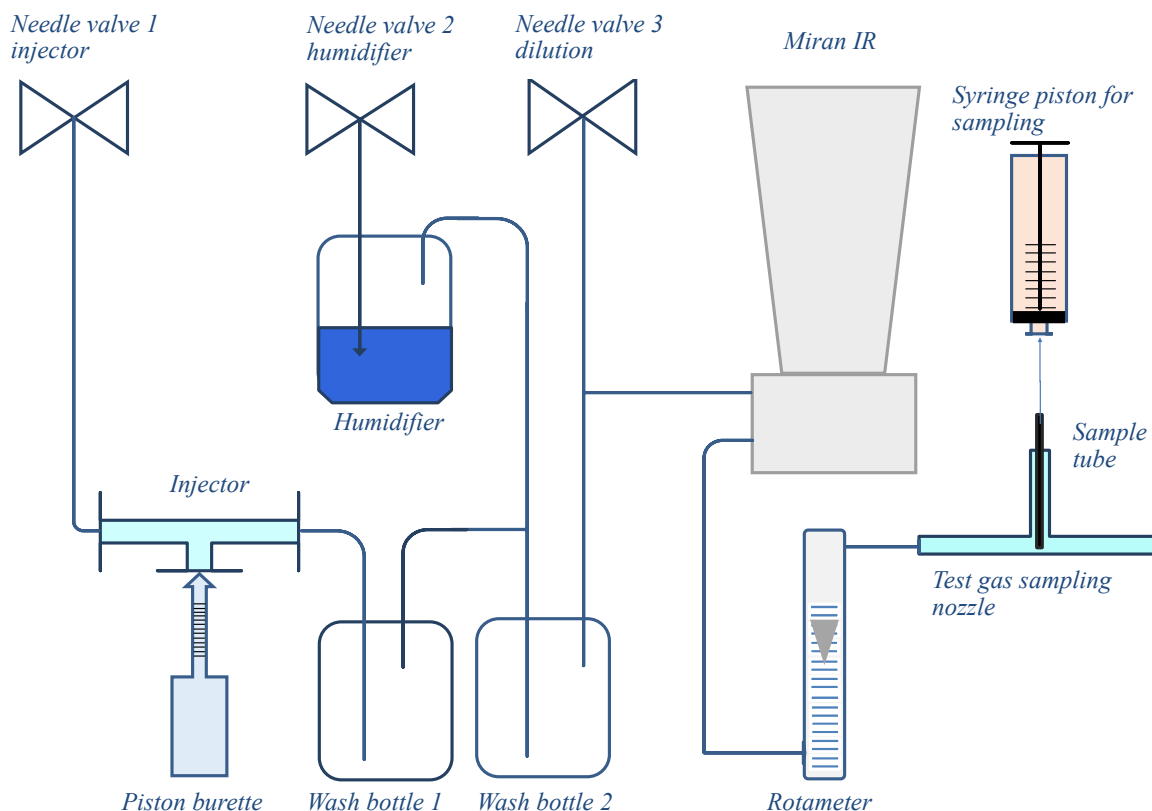


Fig. 1 Schematic illustration of the dynamic test gas facility

The stock solution (Section 4.3, Table 2) or the internal standard (ISTD) is continuously injected at a rate of 150 $\mu\text{l}/\text{h}$ 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 $\mu\text{l}/\text{l}$.

Adsorption tubes packed with Chromosorb 106 are connected to the suction side of a 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 dosing 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 generated 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

All samples are each loaded with 30 ml of ISTD gas atmosphere (concentration of 1 µl/l $\hat{=}$ 0.834 µg/ml) after test gas loading.

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 tested for blank values. The tubes are then tightly sealed with Swagelok caps with PTFE seals and stored at room temperature.

5.2 Sampling

The pre-treated adsorption tubes are used for sampling (Section 5.1). The sealing caps are removed 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.

To ensure that the amounts sampled are not outside the calibration range, a sample volume of 600 ml is desirable. This must be achieved by adjusting the volumetric flow rate and the sampling period. The volumetric flow rate of the pump should be set to 5 ml/min for a 2-hour sampling period and to 40 ml/min for the short-term measurements of 15 min. The volumetric flow rate must be checked before and after sampling. If the deviation from the adjusted flow rate is greater than \pm 5%, it is advisable to repeat the measurement (DIN 2014). The important parameters for 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 °C
Detector gases:	Hydrogen (40°ml/min), synthetic air (400°ml/min)
Heating rate:	5 min at 30 °C, increased by 40 °C/min up to 200 °C, 3 min

Thermal desorber

Apparatus:	Turbomatrix 650 (PerkinElmer LAS)
Desorption temperature:	170 °C
Desorption time:	15 min
Conditioning flow rate:	50 ml/min
Valve temperature:	200 °C
Temperature of transfer line:	200 °C
Transfer line:	1.5 m of deactivated, blank capillaries (ID of 0.32 mm)
Cold trap (adsorption):	5 °C
Cold trap (injection):	250 °C
Cold trap filling:	Air Toxics
Heating rate:	99 °C/s
Carrier gas / column flow:	Helium, 2.2 ml/min, constant flow rate
Split (in front of the cold trap):	80 ml/min (input split)
Desorption flow over the cold trap:	10 ml/min (desorption flow)
Split (behind 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, then the instrumental conditions must be adapted accordingly.

Under the conditions given here, the retention time for 2-isopropoxyethanol is approx. 10.4 minutes.

7 Analytical determination

The loaded adsorption tubes are heated in the thermal desorber, whereby the substances are transferred with 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. A calibration standard of known concentration must be analysed in each analytical series for this purpose.

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:

$$\rho = \frac{X}{V \times \eta} \quad (1)$$

[Equation 2](#) enables calculation of the value at 20 °C and 1013 hPa:

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

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 2021 a](#)), DIN EN ISO 22065 ([DIN 2021 b](#)) 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 two experimental series with 2-isopropoxyethanol 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](#). The first experimental series was carried out in the absence of any humidity for concentrations of one tenth, once and twice the OELV with six spiked tubes per concentration. The second series at 80% relative humidity was carried out with five spiked tubes a concentration of one tenth and twice the OELV, each. The analytical determination was carried out according to [Sections 6 and 7](#).

The expanded uncertainty was estimated taking all the relevant influencing factors into consideration as stipulated in DIN EN 482 (DIN [2021 a](#)) and DIN EN ISO 22065 (DIN [2021 b](#)) and estimated with the aid of the IFA-Excel-Sheets (IFA [n.d.](#)) for the calculation of the expanded uncertainty. It is 28.4% for 2-isopropoxyethanol. The results are summarised in [Table 4](#).

10.2 Recovery

The recovery of 2-isopropoxyethanol was calculated in the same manner as the precision from the same samples and is listed for three different concentrations in each case in [Table 4](#).

The mean recovery of 84% in the lower concentration range is approx. 15% smaller than in the intermediate and higher concentration ranges.

Tab. 4 Recovery, relative standard deviation and expanded uncertainty for three different concentrations

Concentration [mg/m ³]	Recovery at 0% humidity [%]	Rel. standard deviation [%]	Expanded uncertainty U [%]	Recovery at 80% rel. humidity [%]
4.62	87.0	3.4	28.4	81.0
41.54	96.6	2.9	28.4	–
83.08	100	2.9	28.4	101

10.3 Limit of quantification

As part of the development of the method, the limit of quantification was calculated in the same manner as calibration line method according to DIN 32645 (DIN [2008](#)) with a statistical certainty of P = 95% and k = 2. It is 2.69 µg of 2-isopropoxyethanol, absolute.

The relative limit of quantification is 4.48 mg/m³ 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 loading collection tubes at twice the OELV for 2 hours and 20 times OELV for 3 hours at a volumetric flow rate of 85 ml/min. A direct-reading measurement device is used, which is connected by a tube to the outlet of the adsorbent tube, to check whether a breakthrough occurs in real time. No breakthrough was observed for either of these measurements. In this method, the sample volume, the volumetric flow rate as well as the concentration of the substance are considerably below the capacity of the tubes filled with 370 mg of Chromosorb 106.

10.5 Storage stability

As described in [Section 4.4](#), 12 adsorption tubes were each spiked with 4.62 mg/m³, 41.54 mg/m³ and 83.08 mg/m³ of 2-isopropoxyethanol 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. The storage stability of the adsorption tubes over a period of four weeks is checked and confirmed.

Tab. 5 Recoveries at 0% humidity after 1, 2, 3 and 4 weeks

Concentration [mg/m ³]	1 day [%]	1 week [%]	2 weeks [%]	3 weeks [%]	4 weeks [%]
4.62	86.8	84.1	91.4	93.2	89.6
41.54	93.5	92.0	96.9	99.9	99.9
83.08	97.0	100.5	103.8	104.2	102.7

In addition, the influence of the humidity on storage stability was checked. For this purpose, 10 adsorption tubes were again each spiked with 4.62 mg/m³ (0.1 OELV) and 83.08 mg/m³ (2 OELV) of 2-isopropoxyethanol 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 the storage stability due to the humidity could be detected.

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

Concentration [mg/m ³]	0% rel. humidity		80% rel. humidity	
	1 day [%]	3 weeks [%]	1 day [%]	3 weeks [%]
4.62	78.2	84.7	81.0	90.0
83.08	100.3	100.1	100.7	104.1

10.6 Interference

The analytical procedure by GC-FID is specific and robust under the operational conditions stated here. There was no evidence of interference from other solvents.

11 Discussion

The analytical method described here permits the determination of 2-isopropoxyethanol in workplace air in a concentration range from a tenth to twice the currently valid OELV of 44 mg/m³. The method is suitable for checking the short-term exposure limit. No negative influence could be detected on the measurement results and storage stability due to the humidity (of up to 80% relative humidity).

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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