

Methanesulfonic acid – Determination of methanesulfonic acid in workplace air using ion chromatography (IC)

Air Monitoring Method – Translation of the German version from 2023

Keywords

air analyses; analytical method; workplace measurement; hazardous substance; ion chromatography; quartz fibre filter; conductivity detector

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Abstract

The analytical method described here permits the determination of methanesulfonic acid [75-75-2] occurring as inhalable particles and vapour in workplace air. The concentration range covers one tenth up to twice the currently valid Occupational Exposure Limit Value (OELV) in Germany of 0.7 mg/m³ (inhalable fraction). The short-term exposure limit (STEL) with an excursion factor of 1 for the inhalable fraction and vapour can also be checked. Sampling is performed using a flow-regulated pump to draw a defined volume of air through a quartz fibre filter, which is alkaline-impregnated with sodium carbonate and inserted into a GSP sampling system. The volumetric flow rate is 10 l/min. Two hours or 15 minutes are suitable sampling periods. Alternatively, an intake cone suitable for a flow rate of 3.5 l/min can also be used for a sampling period of 2 hours. The methanesulfonic acid deposited on the filter is extracted with an aqueous sodium carbonate/sodium hydroxide solution and analysed by means of ion chromatography using a conductivity detector. Quantitative determination is based on multiple-point calibrations with external standards. For an air sample volume of 1200 litres, the relative limit of quantification (LOQ) is 0.021 mg/m³. At an LOQ of less than 0.17 mg/m³, the measurement of the STEL is also possible with an air sample volume of 150 litres. The recovery is 100% and the expanded uncertainty is 22% for a 2-hour sampling period and 35% for a 15-minute sampling period.

Method number	1
Application	Air analysis
Analytical principle	Ion chromatography with conductivity detection (IC)

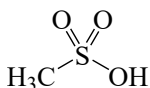
1 Characteristics of the method

Repeatability for control solutions:	Standard deviation (rel.): $s = 1.8\%$ at a concentration of 94 $\mu\text{g/ml}$
Repeatability for sample solutions:	Standard deviation (rel.): $s = 1.8\%$ at a concentration of 177 $\mu\text{g/ml}$
Reproducibility:	Standard deviation (rel.): $s = 0.9$ to 1.8% at a spiking level of 0.884 mg of methanesulfonic acid/filter and 0.103 mg of methanesulfonic acid/filter
Recovery:	$\eta = 100\%$
Limit of quantification:	5 $\mu\text{g/ml}$ (0.5 $\mu\text{g/ml}$ analytically possible) 0.021 mg/m^3 for an air sample volume of 1200 l and a sampling period of 2 h 0.06 mg/m^3 for an air sample volume of 420 l 0.17 mg/m^3 for an air sample volume of 150 l, an extraction volume of 5 ml and a sampling period of 15 min
Expanded uncertainty:	$U = 21$ to 22% in a range of 0.07 to 1.4 mg/m^3
Sampling recommendations:	Sampling period: 2 h Air sample volume: 1200 l or 420 l Flow rate: 10 l/min or 3.5 l For short-term measurements: 15 min; 150 l

2 Description of the substance

The Occupational Exposure Limit Value (OELV) is 0.7 mg/m^3 for the sum of vapours and aerosols according to TRGS 900, measured in the inhalable fraction. The substance is classified in Peak Limitation Category I with an excursion factor of 1 (AGS 2022).

Methanesulfonic acid [75-75-2]



Synonyms: Methylsulfonic acid, MSA

Methanesulfonic acid is the simplest sulfonic acid and is a colourless and odourless viscous liquid. It is a strong acid ($\text{pK}_a = -1.9$) and is miscible with water in any proportion and can be used as a solvent. The molar mass is 96.11 g/mol, the density is 1.48 g/cm^3 . The melting point is 19 °C and the boiling point is 167 °C at 13 hPa. Methanesulfonic acid has a vapour pressure of 0.100 Pa at 23 °C. The salts are known as methanesulfonates or mesylates (IFA 2021).

Methanesulfonic acid can, for example, be produced by oxidation of methanethiol with nitric acid. The conversion of methane with sulfur trioxide is also described for the large-scale technical production. Methanesulfonic acid serves

as a solvent and a catalyst for different organic reactions such as alkylations and polymerisations. It is increasingly used in cleaning agents, as well as in galvanisation baths (RÖMPP-Redaktion and Jahn 2019).

3 General principles

This analytical method permits the determination of methanesulfonic acid in workplace air in a concentration range of one tenth up to twice the currently valid OELV, as well as for checking the short-term exposure limit.

For sampling, a flow-regulated pump draws a defined volume of air through an alkaline-impregnated quartz fibre filter which is located in a GSP sampling system with an intake cone for a flow rate of 10 l/min. Alternatively, an intake cone suitable for a flow rate of 3.5 l/min can also be used for measurement of concentrations in the range of the OELV. After elution, the methanesulfonic acid deposited on the filter is analysed by means of ion chromatography and conductivity detection. The quantitative evaluation is based on two external calibration curves with 5 or 7 calibration points.

4 Equipment, chemicals and solutions

4.1 Equipment

For sampling:

- Pump for personal sampling, flow rate of 10.0 l/min (e.g. SG10-2, from GSA Messgerätebau GmbH, 40880 Ratingen, Germany)
- Personal sampling system for hazardous substances (PGP) with a GSP sampling head for the inhalable particle fraction with an intake cone for a flow rate of 10 l/min and 3.5 l/min and suitable filter cassettes (e.g. from DEHA Haan & Wittmer GmbH, 71296 Heimsheim, Germany)
- Rotameter (e.g. influx 1-13 litres, from DEHA Haan & Wittmer, 71296 Heimsheim, Germany)
- Quartz fibre filter, diameter 37 mm (e.g. T293, from Sartorius AG, 37079 Göttingen, Germany)

For sample preparation and analysis:

- Volumetric flasks made of polypropylene, 10 ml, 100 ml and 1000 ml
- Plastic screw-capped vessels, 30 ml (e.g. sample vessel made of polypropylene with screw caps, from Buddeberg GmbH, 68219 Mannheim, Germany)
- Variable piston pipettes, 5 µl to 10 ml (e.g. Multipette stream, from Eppendorf SE, 22366 Hamburg, Germany)
- Syringe filters (e.g. IC ACRODISC from PALL Ø 13 mm, 0.45 µm Supor (PES) Membrane, from VWR International GmbH, 40764 Langenfeld, Germany)
- Autosampler vials, 0.5 ml and 5 ml with caps (e.g. GC 000044, from Buddeberg, 68219 Mannheim, Germany and from ThermoFisher Scientific GmbH, 63303 Dreieich, Germany)
- Ion chromatograph with degasser, autosampler, suppressor and conductivity detector (e.g. ASSY Aquion DG with AS-DV, Dionex ADRS 600 Suppressor 4 mm, from ThermoFisher Scientific GmbH, 63303 Dreieich, Germany)
- Separation column (e.g. Dionex Ion Pac AS 22, inner diameter (ID) 4 mm, length (L) 250 mm with Dionex Ion Pac AG 22 Guard precolumn, ID 4 mm, L 50 mm, from ThermoFisher Scientific GmbH, 63303 Dreieich, Germany)

4.2 Chemicals

- Methanesulfonic acid, 99.6% (e.g. Article No. 59510, from Merck KGaA, 64271 Darmstadt, Germany)
- Methanesulfonic acid, 99.69% as a control standard (e.g. Article No. 471356, from Merck KGaA, 64271 Darmstadt, Germany)
- Sodium bicarbonate, 99% for HPLC (e.g. Article No. 446232500, from VWR International GmbH, 40764 Langenfeld, Germany)
- Sodium carbonate, anhydrous, 99.999% (e.g. Article No. 1.06395.0050, from Merck KGaA, 64271 Darmstadt, Germany)
- Ultrapure water ($\rho \geq 18.2 \text{ M}\Omega \times \text{cm}$ at 25 °C)
- Alternatively, the AS 22 IC eluent concentrate (Article No. 63965, from ThermoFisher Scientific GmbH, 63303 Dreieich, Germany) can be used.

4.3 Solutions

Stock solution of the eluent: (450 mmol sodium carbonate/l and 140 mmol sodium bicarbonate/l):

4.77 g of sodium carbonate and 1.18 g of sodium bicarbonate are weighed and transferred into a 100-ml volumetric flask. The volumetric flask is then filled to the mark with ultrapure water and shaken.

Eluent: (4.5 mmol sodium carbonate/l and 1.4 mmol sodium hydrogen carbonate/l):

10 ml of the eluent stock solution are pipetted into a 1000-ml volumetric flask into which approx. 900 ml of ultrapure water have been placed. The flask is then filled to the mark with ultrapure water and shaken.

The storage stability of the eluent is limited, it should be freshly prepared after at latest 5 days.

Impregnation solution for the quartz fibre filters: (1.0 mol sodium carbonate/l):

10.6 g of sodium carbonate are weighed into a 100-ml volumetric flask and dissolved in ultrapure water. The volumetric flask is then filled to the mark and shaken.

4.4 Calibration and control standards

Calibration stock solutions:

Calibration stock solution 1: (14.74 mg methanesulfonic acid/ml):

Ultrapure water (approx. 7 ml) is placed into a 10-ml volumetric flask. After 100 μl of methanesulfonic acid (density 1.48 g/ml) have been added, the volumetric flask is filled to the mark with ultrapure water and shaken.

Calibration stock solution 2: (1.474 mg methanesulfonic acid/ml):

Ultrapure water (approx. 7 ml) is placed into a 10-ml volumetric flask. After 1 ml of calibration stock solution 1 has been added, the volumetric flask is filled to the mark with ultrapure water and shaken.

Calibration stock solution 3: (0.1474 mg methanesulfonic acid/ml):

Ultrapure water (approx. 7 ml) is placed into a 10-ml volumetric flask. After 1 ml of calibration stock solution 2 has been added, the volumetric flask is filled to the mark with ultrapure water and shaken.

The calibration standards for the higher concentration range are prepared in plastic screw-capped vessels as outlined in [Table 1](#).

Tab. 1 Preparation of the seven methanesulfonic acid calibration standards (C1 to C7) for the higher concentration range

	C1	C2	C3	C4	C5	C6	C7
Addition of calibration stock solution 2 in μl	40	80	150	300	500	750	1200
Addition of impregnation solution in μl	500	500	500	500	500	500	500
Addition of elution agent in μl	4460	4420	4350	4200	4000	3750	3300
Concentration of methanesulfonic acid in $\mu\text{g/ml}$	11.79	23.58	44.22	88.44	147.4	221.1	353.8

The calibration standards for the lower concentration range are prepared as outlined in [Table 2](#). Methanesulfonic acid concentrations up to 20 $\mu\text{g/ml}$ are evaluated using the calibration curve in the lower range.

Tab. 2 Preparation of the five methanesulfonic acid calibration standards (C1 to C5) for the lower concentration range

	C1	C2	C3	C4	C5
Addition of calibration stock solution 2 in μl	15	30	45	60	75
Addition of impregnation solution in μl	500	500	500	500	500
Addition of eluent in μl	4485	4470	4455	4440	4425
Concentration of methanesulfonic acid in $\mu\text{g/ml}$	4.42	8.84	13.27	17.69	22.11

Control stock solutions:

Control stock solution 1: (11.8 mg methanesulfonic acid/ml):

80 μl of methanesulfonic acid are added into a 10-ml volumetric flask, into which ultrapure water (approx. 7 ml) has been placed. The flask is then filled to the mark with ultrapure water and shaken.

Control standard 1: (94.4 μg methanesulfonic acid/ml):

80 μl of control stock solution 1 and 500 μl of impregnation solution are added into a 10-ml volumetric flask into which ultrapure water (approx. 7 ml) has been placed. The flask is filled to the mark with ultrapure water and shaken.

Control standard 2: (9.4 μg methanesulfonic acid/ml):

1 ml of control standard 1 and 450 μl of impregnation solution are added into a 10-ml volumetric flask, into which ultrapure water (approx. 7 ml) has been placed. The flask is filled to the mark with ultrapure water and shaken.

5 Sampling and sample preparation

5.1 Preparation of the sample carriers

The quartz fibre filter is impregnated before sampling. For this purpose, 500 μl of the impregnation solution ([Section 4.3](#)) are uniformly distributed over the filter. The filter should then be left to dry in air overnight. The sample carriers are prepared by inserting a supporting sieve followed by the dried impregnated filter. Until sampling, the filter capsule remains sealed with the pertinent screw cap and is stored at room temperature. The prepared sample carriers can be used for two weeks.

The batch of quartz fibre filters used must be checked for possible blank values.

5.2 Sampling

The filter cassette, containing a supporting sieve and an impregnated quartz fibre filter, is inserted into the GSP sampling head ([Riediger 2001](#)) and equipped with an intake cone for 10 l/min or 3.5 l/min (for a sampling period of two hours). The time-weighted average is measured over a sampling period of 2 hours and results in an air sample volume of 1200 litres or 420 litres. A sampling period of 15 minutes is suitable for the measurement of the short-term

exposure limit, which results in an air sample volume of 150 litres. Sampling can be carried out stationary or personal. After sampling, the flow rate must be tested for constancy. If the deviation from the adjusted flow rate is greater than $\pm 5\%$, it is advisable to discard the sample (DIN 2023 a). The filter cassette with the loaded filter is sealed with the caps designated for the purpose and transported to the laboratory.

It is advisable to include one blank sample ('field blank') per sample series. This differs from the analytical sample only in that no sample air was drawn through the filter. This blank sample is then stored and analysed in the same manner as the other samples.

5.3 Sample preparation

The loaded filters are removed from the filter cassette and placed in a screw-capped plastic vessel. 5 ml of the eluent are added to the filters in each case, which are then treated for 15 minutes in the ultrasonic bath. The sample solutions are subsequently left to cool to room temperature for approx. 15 minutes. An aliquot of the sample solution is withdrawn with a disposable syringe and filtered into an autosampler vial through a syringe filter. The remaining sample solution is kept as a retention sample and is stored at room temperature.

It is advisable to additionally prepare and analyse a reagent blank value ('lab blank'). For this purpose, 500 μl of the impregnation solution and 4.5 ml of the eluent are pipetted into a screw-capped vessel, which is shaken and a Pasteur pipette is used to fill an aliquot into an autosampler vial.

6 Operating conditions

Apparatus:	Ion chromatograph with degasser and autosampler
Detector:	Conductivity detector
Detector temperature:	35 °C
Precolumn:	Dionex Ion Pac AG 22 Guard, ID 4 mm, L 50 mm
Separation column:	Dionex Ion Pac AG 22, ID 4 mm, L 250 mm
Mobile phase:	4.5 mM of sodium carbonate + 1.4 mM of sodium hydrogen carbonate, isocratic
Flow rate:	1.2 ml/min
Injection volume:	10 μl
Suppressor:	Dionex ADRS 600 4 mm; 31 mA
Flow time:	6 min

7 Analytical determination

For the analytical determination, 10 μl of the samples prepared as described in [Section 5.3](#) are each injected into the ion chromatography system and analysed under the conditions described in [Section 6](#). Depending on the methanesulfonic acid concentration to be determined, the relevant calibration curve is used for evaluation. Concentrations greater than 20 $\mu\text{g}/\text{ml}$ are evaluated by means of the calibration curve in the higher concentration range. If the measured concentrations are above the calibration range, then suitable dilutions using the eluent must be prepared and the analysis must be repeated.

8 Calibration

External calibration:

The calibration standards prepared according to [Section 4.4](#) are analysed as described in [Sections 6](#) and [7](#) in order to obtain the calibration functions.

The retention time for methanesulfonic acid is 3.6 minutes.

The resulting peak areas are plotted versus the corresponding concentrations.

The control standards described in [Section 4.4](#) are analysed to check the calibration functions every working day.

9 Calculation of the analytical result

Taking the air sample volume, the eluate volume, the dilution and the recovery into account, the concentration of methanesulfonic acid in the workplace air is calculated according to [Equation 1](#).

$$\rho = \frac{((c \times f_v) - c_{blank}) \times V \times 100}{V_{air} \times \eta} \quad (1)$$

where:

ρ is the mass concentration of methanesulfonic acid in the air sample in mg/m³

c is the concentration of methanesulfonic acid in the sample solution in µg/ml

c_{blank} is the concentration of the field blank value in µg/ml

f_v is the dilution factor

V is the volume of the eluate in ml (5 ml in this case)

V_{air} is the air sample volume in litres

η is the recovery in %

10 Reliability of the method

The characteristics of the method were determined as stipulated in DIN EN 482 (DIN 2015), DIN EN ISO 23861 (DIN 2023 b), DIN EN ISO 21832 (DIN 2020) as well as DIN 32645 (DIN 2008).

10.1 Repeatability

The repeatability was ascertained by analysing one prepared sample solution and one standard solution of intermediate concentration in the higher calibration range on six days. The concentrations and the relative standard deviations determined are listed in [Table 3](#).

Tab. 3 Repeatability over 6 days

	Concentration [µg/ml]	Relative standard deviation [%]
Sample solution	177	1.8
Standard	94	1.8

Additionally, the repeatability of a standard in the lower concentration range was determined by a six-fold measurement on one day. This was 1% for a concentration of 23.6 µg/ml.

10.2 Recovery and reproducibility

The recovery was determined by spiking six impregnated filters with 60 µl of calibration stock solution 1 each. After the filters had been dried overnight, clean air was drawn through the filters for 2 hours at a flow rate of 10 l/min. The samples were prepared according to [Section 5.3](#) and analysed according to [Sections 6 and 7](#).

Six reference solutions were also prepared in the same manner. For this purpose, 4.44 ml of the eluent, 500 µl of the impregnation solution and 60 µl of calibration stock solution 1 were each pipetted into a screw-capped vessel. After mixing, these solutions were also analysed according to [Sections 6 and 7](#).

The recovery was 100.8%, based on the mean value for the reference solutions. There is no statistically significant difference between the two measurement series. Therefore, the recovery is 100%. The reproducibility is 0.9%.

The recovery with low methanesulfonic acid concentrations was determined by loading six impregnated filters with 70 µl of calibration stock solution 2 each. After the filters had been dried overnight, clean air was drawn through the filters for 15 minutes at a flow rate of 10 l/min. The samples were prepared as described in [Section 5.3](#) and analysed according to [Sections 6 and 7](#).

Six reference solutions were also prepared in the same manner. For this purpose, 4.43 ml of the eluent, 500 µl of the impregnation solution and 70 µl of calibration stock solution 2 were each pipetted into a screw-capped vessel. After mixing, these solutions were also analysed according to [Sections 6 and 7](#).

The recovery, based on the mean value for the reference solutions, was also 100.8%. There is no statistically significant difference between the two measurement series. Therefore, the recovery is 100%. The reproducibility is 1.8%. The results are summarised in [Table 4](#).

Tab. 4 Recovery and reproducibility of the entire method based on the reference solutions for sampling periods of 2 h and 15 min

	Spiked quantity [mg]	Concentration in air [mg/m ³]	Recovery [%]	Reproducibility [%]
Methanesulfonic acid	0.884	0.74	100.8	0.9
	0.103	0.69	100.8	1.8

10.3 Expanded uncertainty of the entire method

The uncertainty was obtained by estimation of all the relevant influencing parameters (DIN 2008). This consists of two important steps, the uncertainty components for sampling and for analysis. In order to estimate the uncertainty components of sampling the uncertainty associated with the air sample volume as well as that of the sampling devices and their effectiveness for capturing inhalable dusts were determined according to Appendix C 3.4.1.2 in DIN EN ISO 21382 (DIN 2008).

The combination of all uncertainty contributions results in the concentration-dependent combined uncertainty. The corresponding expanded uncertainties that represent the concentration-dependent uncertainty of the entire procedure are obtained by multiplication with a probability factor $k=2$ (for 95% confidence level).

[Table 5](#) summarises all the determined uncertainty contributions, whereby a differentiation is made between high, intermediate and low concentrations in air and sampling periods of 2 hours and 15 minutes.

Tab. 5 Determination of the uncertainty in %

Uncertainty	Sampling period of 2 hours	Concentration in air	Sampling period of 15 minutes	Concentration in air
<i>u</i> sampling (air sample volume, sampling apparatus)	8.9		9.1	
<i>u</i> extraction	0.4		0.4	
<i>u</i> recovery and storage	4.8		4.8	
<i>u</i> measurement value for high concentrations	3.0		7.5	
<i>u</i> measurement value for intermediate concentrations 1	3.4		14.5	
<i>u</i> measurement value for intermediate concentrations 2	4.5			
<i>u</i> measurement value for low concentrations	3.0		4.3	
<i>u</i> combined high concentrations	10.5		12.7	
<i>u</i> combined intermediate concentrations 1	10.7		17.7	
<i>u</i> combined intermediate concentrations 2	11.0			
<i>u</i> combined low concentrations	10.5		11.1	
<i>U</i> expanded high concentrations	21	$c = 1.4 \text{ mg/m}^3$	25	$c = 1.4 \text{ mg/m}^3$
<i>U</i> expanded intermediate concentrations 1	21	$c = 0.7 \text{ mg/m}^3$	35	$c = 0.67 \text{ mg/m}^3$
<i>U</i> expanded intermediate concentrations 2	22	$c = 0.35 \text{ mg/m}^3$		
<i>U</i> expanded low concentrations	21	$c = 0.071 \text{ mg/m}^3$	22	$c = 0.33 \text{ mg/m}^3$

The expanded uncertainties for the analytical method described here were from 21% to 22% in the measurement range of one tenth up to twice the limit value for a sampling period of 2 hours. The expanded uncertainties for the measurement range of half to twice the short-term exposure limit for a sampling period of 15 minutes were from 22 to 35%.

10.4 Limit of quantification

The LOQ was determined by means of an equidistant 10-point calibration in each case as stipulated in DIN 32645 (DIN 2008) in the range of 1.2 to 6.5 µg/ml.

The resulting absolute LOQ of is 0.5 µg/ml. As this is very low and is not required for checking the OELV for this method, it is set at 5 µg/ml. Taking the extraction volume of 5 ml into account a relative LOQ of 0.021 mg/m³ is obtained for the time-weighted average for an air sample volume of 1200 litres or 0.06 mg/m³ for 420 litres and 0.17 mg/m³ for the STEL for an air sample volume of 150 litres.

10.5 Storage stability

The storage stability of the loaded impregnated quartz fibre filters at room temperature is ensured for a spiking quantity of 884 µg (see Table 4) for 2 weeks, the prepared sample solutions and standards are stable for 2 weeks at room temperature.

10.6 Selectivity

Anions such as fluoride, bromide, chloride, nitrite, nitrate, phosphate and sulphate do not interfere with the analysis. Methanesulfonic acid is readily separated by chromatography (Figure 1).

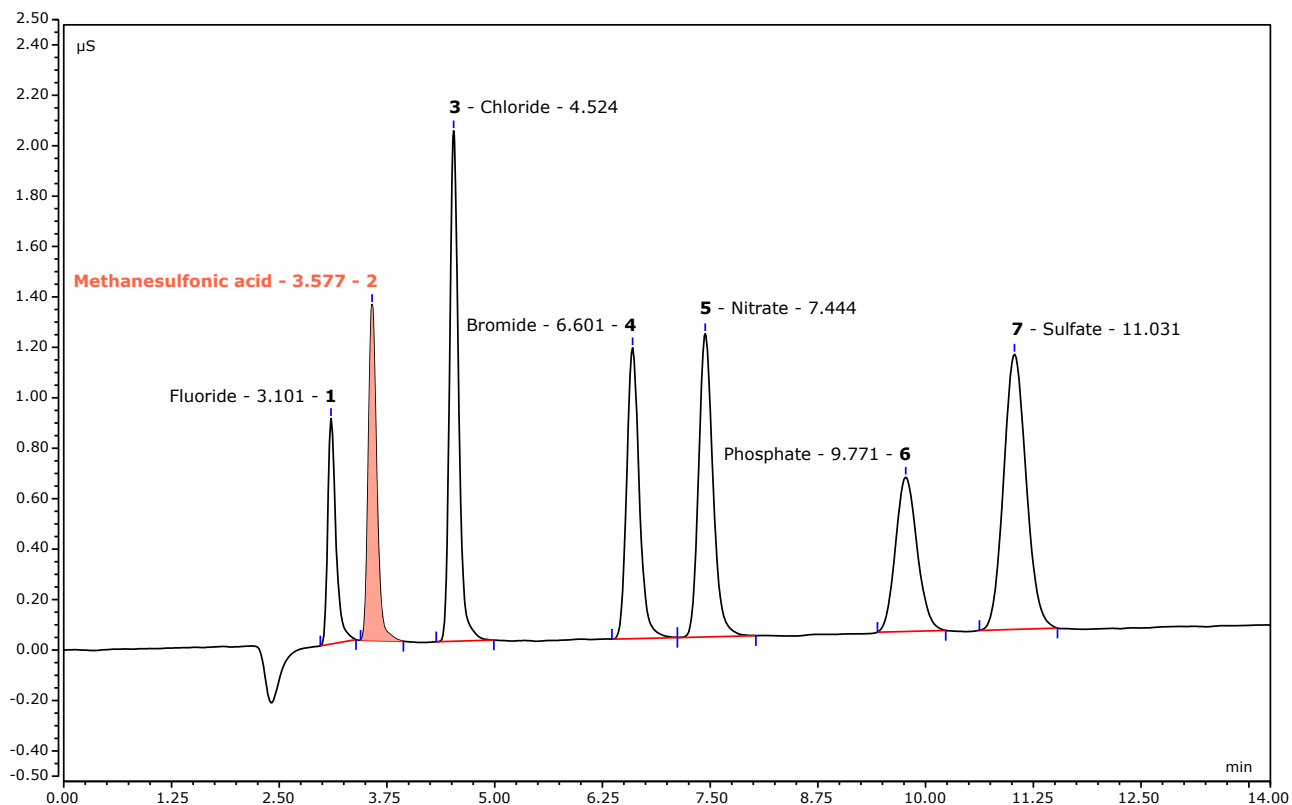


Abb. 1 Example of a chromatogram for the chromatographic separation of methanesulfonic acid (11.8 ppm), fluoride (1.5 ppm), chloride (5 ppm), bromide (10 ppm), nitrate (10 ppm), phosphate (15 ppm) and sulphate (10 ppm)

Blank values are taken into account by the parallel sample work-up of the prepared field blanks. The blank values of the filter and the eluent were below the LOQ.

11 Discussion

The analytical procedure described here permits the determination of methanesulfonic acid in workplace air in a concentration range from a tenth to twice the currently valid OELV of 0.7 mg/m³. By using an impregnated filter, methanesulfonic acid occurring as aerosol as well as vapour is bound to the filter. Furthermore, the analytical method is suitable for monitoring the STEL.

The influence of humidity was not checked, all experiments were carried out at room temperature and at approx. 40% humidity. If conditions are markedly different, it is advisable to check the influence of 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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