

Dicarboxylic acids, short-chained – Determination of oxalic acid, malonic acid, succinic acid, glutaric acid and adipic acid in the workplace air using ion chromatography (IC)

Air Monitoring Method – Translation of the German version from 2022

Keywords

dicarboxylic acid; air analyses;
analytical method; workplace
measurement; hazardous
substance; IC; glass fibre filter;
conductivity detector

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Abstract

The analytical method described here permits the determination of five linear chain aliphatic dicarboxylic acids with 2 to 6 carbon atoms and terminal carboxy groups occurring as inhalable particles in workplace air. The concentration range covers one tenth up to twice the currently valid Occupational Exposure Limit Value (OELV) in Germany, which is 1 mg/m³ for oxalic acid and 2 mg/m³ for succinic, glutaric and adipic acid (inhalable fraction). The peak limitation with an excursion factor of 2 can also be checked. At the moment, there is no OELV for malonic acid, so the same concentration range has been considered as for oxalic acid. Sampling is performed using a flow-regulated pump to draw a defined volume of air through a glass fibre filter, which is alkaline-impregnated with sodium carbonate and inserted in a GSP sampling system. The volumetric flow rate is 10 l/min. For sampling, 2 hours or 15 minutes can be used. The collected dicarboxylic acid deposited on the filter is extracted by means of 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 in the range from 0.0002 mg/m³ for oxalic acid and 0.0009 mg/m³ for succinic acid. With LOQs less than 0.0076 mg/m³,

the measurement of the short-term exposure limit (STEL) is also enabled with an air sample volume of 150 litres. The recoveries of the five dicarboxylic acids range from 96% to 110% and the expanded uncertainty is less than 29%.

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

1 Characteristics of the method

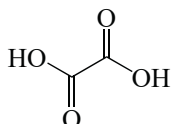
Repeatability:	Standard deviation (rel.):	
	Oxalic acid	s = 1.4%
	Malonic acid	s = 0.68%
	Succinic acid	s = 0.67%
	Glutaric acid	s = 0.59%
	Adipic acid	s = 0.53%
Expanded uncertainty:	In the concentration range from 0.1 to 2.0 mg/m ³	
	Oxalic acid	21 to 29%
	Malonic acid	20 to 21%
	In the concentration range from 0.2 to 4.0 mg/m ³	
	Succinic acid	20 to 22%
	Glutaric acid	20 to 21%
	Adipic acid	20 to 22%
Limit of quantification:	For an elution volume of 10 ml and an air sample volume of 1.2 m ³ :	
	Oxalic acid	0.0002 mg/m ³
	Malonic acid	0.0004 mg/m ³
	Succinic acid	0.0009 mg/m ³
	Glutaric acid	0.0005 mg/m ³
	Adipic acid	0.0007 mg/m ³
	For an elution volume of 10 ml and an air sample volume of 0.15 m ³ :	
	Oxalic acid	0.0020 mg/m ³
	Malonic acid	0.0032 mg/m ³
	Succinic acid	0.0076 mg/m ³
	Glutaric acid	0.0040 mg/m ³
	Adipic acid	0.0052 mg/m ³
Recovery:	Oxalic acid	100 to 110%
	Malonic acid	99 to 103%
	Succinic acid	96 to 102%
	Glutaric acid	101 to 102%
	Adipic acid	100 to 103%
Sampling recommendations:	Sampling period	2 h
	Air sample volume	1200 l
	Flow rate	10 l/min
	For short-term measurements	15 min; 150 l

2 Description of the substances

The straight-chain aliphatic dicarboxylic acids described here are the homologous series of the C₂ to C₆ dicarboxylic acids with terminal carboxyl groups.

With the exception of malonic acid, Occupational Exposure Limit Values (OELV) have been assigned to the other four dicarboxylic acids in the TRGS 900.

Oxalic acid (anhydrous) [144-62-7] or oxalic acid dihydrate [6153-56-6]



Synonym: Ethanedioic acid

Oxalic acid is the simplest dicarboxylic acid, which usually occurs in its anhydrous form or as a dihydrate. Oxalic acid is a colourless and odourless crystalline solid with an acidic taste. The strong acid has notable reductive properties. The molar mass of the anhydrous oxalic acid is 90.04 g/mol. The melting point of oxalic acid dihydrate is approx. 101.5 °C and the boiling point is approx. 150 °C. When heated, decomposition of anhydrous oxalic occurs at temperatures above 110 °C, 157 °C or 189.5 °C (depending on the literature). The vapour pressure at 25 °C is in the range of $2 \cdot 10^{-4}$ Pa to $3 \cdot 10^{-2}$ Pa (Cai et al. 2015; Henk 2005; Soonsin et al. 2010). The solubility in water is 90 to 100 g/l (20 °C) (IFA 2022). The salts are known as oxalates.

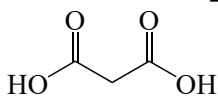
Oxalic acid occurs naturally in e.g. rhubarb, sorrel, common wood sorrel, Swiss chard, spinach and turnip leaves. It is a metabolic product of amino acids and ascorbic acid and is excreted in the urine (RÖMPP-Redaktion and Schmidt 2008).

Oxalic acid can be synthetically produced from sodium formate in the presence of an alkaline catalyst (RÖMPP-Redaktion and Schmidt 2008).

It is used e.g. for the removal of rust spots, as a bleach, as a reducing agent in analytical chemistry (manganometry), for the production of inks, metal cleaning agents and pigments as well as for the electrochemical manufacture of protective coatings on aluminium and in electroplating (RÖMPP-Redaktion and Schmidt 2008).

The OELV for oxalic acid is 1 mg/m³, measured in the inhalable particle fraction. The short-term exposure limit is classified in Peak Limitation Category I with an excursion factor of 1 (AGS 2022).

Malonic acid [141-82-2]



Synonym: Propanedioic acid

Malonic acid is a colourless and odourless, crystalline solid (molar mass 104.06 g/mol, melting point 132 to 135 °C, boiling point from 140 °C), which causes decomposition. It is readily soluble in water (1400 g/l at 20 °C) and reacts as an acid. The vapour pressure at 25 °C is in the range of $5 \cdot 10^{-5}$ Pa to $3 \cdot 10^{-3}$ Pa (Cai et al. 2015; Henk 2005; O'Meara et al. 2014, supplementary information; Soonsin et al. 2010). The esters and salts are known as malonates.

Malonic acid occurs naturally e.g. in the sap of sugar beet. It inhibits succinate dehydrogenase (RÖMPP-Redaktion and Schmidt 2008).

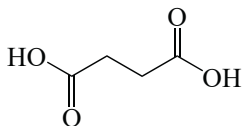
It can be synthetically produced through conversion of chloroacetic acid with sodium cyanide and subsequent hydrolysis. Furthermore, it is formed by the oxidation of malic acid (hydroxysuccinic acid) (RÖMPP-Redaktion and Schmidt 2008).

Malonic acid is an important reagent in organic synthesis, in particular for the production of barbituric acid and its derivatives. Furthermore, it serves as a starting material for the synthesis of diethyl malonate as well as for heterocyclic

compounds. Malonic acid derivatives are used as building-blocks in the Knoevenagel condensation or for the syntheses of malonic esters and barbituric acid and its derivatives (RÖMPP-Redaktion and Schmidt 2008).

There is currently no OELV for malonic acid and no maximum workplace concentration (MAK value). In the absence of a limit value, the same measurement range as for oxalic acid has been adopted and validated.

Succinic acid [110-15-6]



Synonyms: Butanedioic acid, amber acid, wormwood acid

Succinic acid, a dicarboxylic acid that is soluble in water (58 g/l at 20 °C), is a colourless and odourless, crystalline solid with a strongly acidic taste (molar mass 118.09 g/mol, melting point 185 to 190 °C, boiling point from 235 °C to form the anhydride). The vapour pressure is in the range of $6 \cdot 10^{-6}$ Pa to $4 \cdot 10^{-3}$ Pa at a temperature of 25 °C (Cai et al. 2015; Chattopadhyay and Ziemann 2005; Henk 2005; O'Meara et al. 2014; Soonsin et al. 2010). The esters and salts are known as succinates.

Succinic acid occurs in amber, in lignite, in many fruits and vegetables as well as in lichens and fungi. It is formed in the human organism as an intermediary product in the citric acid cycle. Succinic acid is a by-product of alcoholic fermentation (RÖMPP-Redaktion and Schmidt 2008).

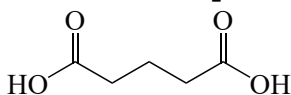
Succinic acid is synthetically produced by hydrogenation of maleic acid or maleic acid anhydride or by oxidation of butane-1,4-diol. It can be biotechnologically produced by fermentation of carbohydrates (RÖMPP-Redaktion and Schmidt 2008).

Succinic acid is used, for instance, in the production of polyester and alkyd resins, pigments and pharmaceuticals. Succinates esterified with polyols can be used as solvents and plasticisers for plastics. Succinates can be used as starting materials for the synthesis of e.g. malic acid, fumaric acid and butane-1,4-diol (RÖMPP-Redaktion and Schmidt 2008).

Furthermore, succinic acid is approved as a food additive (E363) and is used as a flavour enhancer and acidulant (RÖMPP-Redaktion and Schmidt 2008).

The OELV for succinic acid is 2 mg/m³, measured in the inhalable particle fraction. The short-term exposure limit is classified in Peak Limitation Category I with an excursion factor of 2 (AGS 2022). The MAK value is the same as the OELV (DFG 2021). Detailed information on the toxicity of succinic acid is found in the MAK Value Documentation (Hartwig and MAK Commission 2018 b).

Glutaric acid [110-94-1]



Synonyms: Pentanedioic acid, 1,5-pentanedioic acid, propane-1,3-dicarboxylic acid, 1,3-propanedicarboxylic acid

Glutaric acid is a colourless and odourless, crystalline solid (molar mass 132.12 g/mol, melting point 97.5 to 99 °C, boiling point 302 to 304 °C). It is sensitive to light and reacts as an acid. The solubility in water is 640 g/l (20 °C). The vapour pressure is in the range of $5 \cdot 10^{-5}$ Pa to $3 \cdot 10^{-3}$ Pa at a temperature of 25 °C (Cai et al. 2015; Chattopadhyay and Ziemann 2005; Henk 2005; O'Meara et al. 2014; Soonsin et al. 2010). The esters and salts are called glutarates.

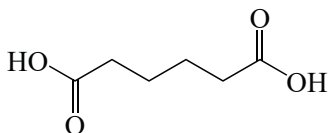
Glutaric acid can be found in e.g. the juice of turnips and in the rinsing water of untreated sheep's wool (RÖMPP-Redaktion and Schmidt 2008).

It can be produced e.g. by oxidation of cyclopentanone with nitric acid and vanadium(V) oxide (RÖMPP-Redaktion and Schmidt 2008).

Glutaric acid is an intermediary product in organic syntheses (RÖMPP-Redaktion and Schmidt 2008).

The OELV for glutaric acid is 2 mg/m³, measured in the inhalable particle fraction. The short-term exposure limit is classified in Peak Limitation Category I with an excursion factor of 2 (AGS 2022). The MAK value is the same as the OELV (DFG 2021). Detailed information on the toxicity of glutaric acid can be found in the MAK Value Documentation (Hartwig and MAK Commission 2020).

Adipic acid [124-04-9]



Synonyms: 1,6-Hexanedioic acid, 1,4-butanedicarboxylic acid, 1,4-dicarboxylic butane, E355, acifloctin

Adipic acid is a colourless and odourless crystalline solid with an acidic taste. The molar mass is 146.14 g/mol and the melting point is between 151 and 152 °C. At 15 g/l (20 °C) the solubility in water is low. The vapour pressure is in the range of $5 \cdot 10^{-6}$ Pa to $3 \cdot 10^{-4}$ Pa at 25 °C (Chattopadhyay and Ziemann 2005; Hartwig and MAK Commission 2018 a; Henk 2005; O'Meara et al. 2014). The esters and salts are known as adipates.

Adipic acid occurs naturally e.g. in sugar beet and beetroot (RÖMPP-Redaktion and Schmidt 2008).

The preferred synthetic production of adipic acid is by oxidative cleavage of cyclohexane (RÖMPP-Redaktion and Schmidt 2008).

It is a starting material for nylon production and a precursor for polyester polyols for polyurethane systems and thermoplastic polyurethanes. It is also used as a food additive (E355) (RÖMPP-Redaktion and Schmidt 2008).

The OELV for adipic acid is 2 mg/m³, measured in the inhalable particle fraction. The short-term exposure limit is classified in Peak Limitation Category I with an excursion factor of 2 (AGS 2022). The MAK value for adipic acid is the same as the OELV (DFG 2021). Detailed information on the toxicity of adipic acid is found in the MAK Value Documentation (Hartwig and MAK Commission 2018 a).

3 General principles

This analytical method permits the determination of the dicarboxylic acids oxalic acid, malonic acid, succinic acid, glutaric acid and adipic acid in the workplace air. The concentration range is one tenth to twice the currently valid OELV of

- 1 mg/m³ (inhalable particle fraction) for oxalic acid or
- 2 mg/m³ (inhalable particle fraction) for succinic acid, glutaric acid and adipic acid

It is also possible to monitor the peak limit with an excursion factor of 1 for oxalic acid as well as 2 for succinic acid, glutaric acid and adipic acid (AGS 2022; DIN 2021).

There is currently no OELV and no MAK value for malonic acid. Therefore, the concentration range for oxalic acid was adopted.

Sampling is carried out by a flow-regulated pump drawing a defined volume of air through an alkaline-impregnated glass fibre filter that is located in a GSP sampling system with an intake cone for a flow rate of 10 l/min. The dicarboxylic acids deposited on the filter in particulate form are measured after elution by means of ion chromatography and conductivity detection. The quantitative determination is carried out on the basis of external ten-point calibrations.

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 (e.g. from DEHA Haan & Wittmer GmbH, 71296 Heimsheim, Germany)
- Filter cassettes for the PGP sampling system with metal supporting sieve (e.g. from DEHA Haan & Wittmer GmbH, 71296 Heimsheim, Germany)
- Mass flow meter for 0 to 20 l/min (e.g. TSI 4146, from Driesen+Kern GmbH, 24576 Bad Bramstedt, Germany)
- Glass fibre filter, free from binding agents, diameter 37 mm (e.g. MN85/90 BF, Ref: 4060037, from Macherey-Nagel GmbH & Co. KG, 52355 Düren, Germany)

For sample preparation and analysis:

- Analytical balance (e.g. XPE20S Delta Range[®], from Mettler-Toledo, Columbus, Ohio, United States of America)
- Stainless steel spatula
- Weighing boats made of glass
- Volumetric flasks, 5 ml, 10 ml, 25 ml, 50 ml, 100 ml and 2000 ml (e.g. from Brand GmbH + Co. KG, 97877 Wertheim, Germany)
- Variable piston pipettes, volumes of 10 to 100 µl, 100 to 1000 µl and 500 µl to 2500 µl (e.g. Reference[®] 2, from Eppendorf AG, 22366 Hamburg, Germany)
- Stainless steel tweezers
- Amber glass bottles, 10 ml, with Teflon seals (e.g. Dichtscheibe G18, from CS-Chromatographie Service GmbH, 52379 Langerwehe, Germany)
- Dispenser, 10 ml (e.g. Dispensette[®], from BRAND GmbH & Co. KG, 97877 Wertheim, Germany)
- Laboratory shaker (e.g. KS 15 A Control Shaker, from Edmund Bühler GmbH, 72379 Hechingen, Germany)
- Disposable syringes made of polyethylene, 10 ml (e.g. BD Discardit II, from Becton Dickinson and Company, Warwick, Rhode Island, USA)
- Disposable hypodermic needles, 1.2 × 40 mm (e.g. BD Microlance 3, from Becton Dickinson and Company, Warwick, Rhode Island, USA)
- Disposable filters made of PTFE with Luer lock connection, diameter 25 mm, pore width 0.45 µm (e.g. Chromafil[®] Xtra H-PTFE-45/25, Ref: 729246, from Macherey-Nagel GmbH & Co. KG, 52355 Düren, Germany)
- Autosampler vials made of polyethylene, 2.5 ml, with perforated stoppers (e.g. Article No. 6.2743.040 and Article No. 6.2743.077, from Deutsche Metrohm GmbH & Co. KG, 70794 Filderstadt, Germany)
- Ion chromatograph with degasser, column oven, autosampler, chemical and CO₂ suppression and conductivity detector (e.g. 930 Compact IC Flex, from Deutsche Metrohm GmbH & Co. KG, 70794 Filderstadt, Germany)
- Metrosep A Supp 16-250/4.0 separation column with Metrosep A Supp 16 Guard/4.0 precolumn (e.g. Article No. 6.1031.430 and Article No. 6.1031.500, from Deutsche Metrohm GmbH & Co. KG, 70794 Filderstadt, Germany)

4.2 Chemicals

- Adipic acid, BioXtra, $\geq 99.5\%$ (HPLC) (e.g. Article No. 09582-250G, from Sigma-Aldrich CHEMIE GmbH, 89555 Steinheim, Germany)
- Succinic acid, p.a. ACS, $> 99\%$ (e.g. Article No. 2725.1, from Carl Roth GmbH + Co. KG, 76185 Karlsruhe, Germany)
- Glutaric acid, for synthesis (e.g. Article No. 8.00295.0250, from Merck KGaA, 64271 Darmstadt, Germany)
- Malonic acid, reagent grade, 99.5% (e.g. Article No. 31715, from Thermo-Fischer (Kandel) GmbH, 76870 Kandel, Germany)
- Oxalic acid dihydrate, p.a., $> 99.5\%$ (e.g. Article No. 1.00495.0500, from Merck KGaA, 64271 Darmstadt, Germany)
- Sodium carbonate, anhydrous, p.a., $\geq 99.9\%$ (e.g. Article No. 1.06392.1000, from Merck KGaA, 64271 Darmstadt, Germany)
- IC eluent concentrate (20 \times) for Metrosep A Supp 16 columns (sodium carbonate/sodium hydroxide concentrate with 150 mM Na₂CO₃ and 15 mM NaOH in water) (e.g. Article No.: 38302-2.5L, from Sigma-Aldrich CHEMIE GmbH, 89555 Steinheim, Germany)
- Sulphuric acid 2.5 mol/l (5N) in an aqueous solution, AVS TITRINORM volumetric solution, for suppressor regeneration (e.g. Article No. 30138293, from VWR International, 94126 Fontenay-sous-Bois, France)
- Water for chromatography, electric conductivity $< 0.055 \mu\text{Scm}^{-1}$ (at 25 °C)

4.3 Spiking solutions for calibration

Eluent: 7.5 mmol/l of sodium carbonate / 0.75 mmol/l of sodium hydroxide

100 ml of the IC eluent concentrate (20 \times) for the Metrosep A Supp 16 columns are added to a 2000-ml volumetric flask, into which approx. 500 ml of water (for chromatography) have been previously placed. The volumetric flask is then filled to the mark with water (for chromatography) and shaken.

Impregnation solution for impregnating the quartz fibre filters: $c(\text{Na}_2\text{CO}_3) = 1.0 \text{ mol/l}$

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

Dicarboxylic Acid Stock Solutions 1 for the calibration:

Stock Solutions 1 for the calibration are prepared as individual substance solutions. A defined mass of dicarboxylic acid with a known purity is weighed exactly into a 100-ml volumetric flask according to Table 1. The volumetric flask is then filled to the mark with water (for chromatography) and shaken. The concentrations of the dicarboxylic acid solutions can be found in Table 1 and are in a range of 18 mmol/l for malonic acid to 28 mmol/l for succinic acid.

Tab. 1 Preparation of Dicarboxylic Acid Stock Solutions 1 for the calibration

Dicarboxylic Acid	Purity [%]	Volume [ml]	Concentration	
			[mmol/l]	[g/l]
Oxalic acid $\times 2 \text{ H}_2\text{O}$	99.5	100	20	1.80 ^{a)}
Malonic acid	99.5	100	18	1.87
Succinic acid	99	100	28	3.31
Glutaric acid	99	100	25	3.30
Adipic acid	99.5	100	25	3.65

^{a)} on the basis of anhydrous oxalic acid

The stock solutions are stable for 3 months when stored protected from light at room temperature.

Dicarboxylic acid Stock Solutions 2 for the control standards:

Stock Solutions 2 for the control standards are prepared as individual substance solutions. A defined mass of dicarboxylic acid with a known purity is weighed exactly into a 50-ml volumetric flask according to Table 2. The volumetric flask is then filled to the mark with water (for chromatography) and shaken. The concentrations of the dicarboxylic acid solutions can be found in Table 2 and are in a range of 36 mmol/l for malonic acid to 56 mmol/l for succinic acid.

Tab. 2 Preparation of Dicarboxylic Acid Stock Solutions 2 for the control standards

Dicarboxylic Acid	Purity [%]	Mass [mg]	Volume [ml]	Concentration [mmol/l]	[g/l]
Oxalic acid × 2 H ₂ O	99.5	253.4	50	40	3.60 ^{a)}
Malonic acid	99.5	188.3	50	36	3.75
Succinic acid	99	334.0	50	56	6.61
Glutaric acid	99	333.6	50	50	6.61
Adipic acid	99.5	367.2	50	50	7.31

^{a)} on the basis of anhydrous oxalic acid

4.4 Calibration and control standards

Calibration standards:

Calibration standards are prepared in order to plot the calibration graphs. The calibration standards are prepared as individual substance solutions.

Ten calibration solutions are prepared from Stock Solution 1 as follows:

The volumes (100 µl to 1000 µl) of Stock Solution 1 (see Table 1) are added to 25-ml volumetric flasks, into each of which approx. 5 ml of eluent have been previously placed as listed in Table 3. The volumetric flasks are then filled to the mark with eluent and shaken. The concentrations of the calibration standards are shown in Table 3.

Tab. 3 Pipetting scheme for the preparation of the calibration standards

Calibration Standard	Stock Solution 1 [µl]	Oxalic acid [mg/l]	Malonic acid [mg/l]	Glutaric acid [mg/l]	Adipic acid [mg/l]
I	100	7.20	7.49	13.2	14.6
II	200	14.4	15.0	26.4	29.2
III	300	21.6	22.5	39.6	43.8
IV	400	28.8	30.0	52.9	58.5
V	500	36.0	37.5	66.1	73.1
VI	600	43.2	45.0	79.3	87.7
VII	700	50.4	52.5	92.5	102
VIII	800	57.6	59.9	106	117
IX	900	64.8	67.4	119	132
X	1000	72.0	74.9	132	146

At an air sample volume of 1.2 m³ and an eluent volume of 10 ml, the calibration standards cover approximately 6 to 60% of the respective OELV.

Reference standards:

One reference standard is prepared as an individual substance solution for each dicarboxylic acid.

600 µl of Stock Solution 2 are added to each individual 50-ml volumetric flasks, into which approx. 5 ml of eluent have been previously placed. The volumetric flasks are filled to the mark with eluent and shaken. The concentrations of the reference standards are listed in Table 4.

Tab. 4 Pipetting scheme for the preparation of the reference standards

Reference standard	Stock Solution 2 [µl]	Oxalic acid [mg/l]	Succinic acid [mg/l]	Glutaric acid [mg/l]	Adipic acid [mg/l]
Control	600	43.2	79.4	79.3	87.7

5 Sampling and sample preparation

5.1 Preparation of the sample carriers

The glass 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 the air for approx. six hours. The sample carriers are prepared by first laying down a supporting sieve and then the dried impregnated filter on top. Until sampling commences, the filter capsules remain sealed with the pertinent caps. The prepared sample carriers can be used for two weeks.

5.2 Sampling

The filter cassette containing a supporting sieve and an impregnated glass fibre filter is inserted into the GSP sampling head (Riediger 2001) and equipped with an intake cone for 10 l/min. A sampling period of 15 minutes can be selected for checking the peak limit or two hours for checking the shift mean value, which results in air sample volumes of 150 litres or 1200 litres. Sampling can be carried out as stationary or personal sampling. 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 2014). The filter cassette with the loaded filter is sealed with the caps designated for the purpose and transported to the laboratory.

One blank sample ('field blank') per sample series must be prepared. 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 filter remains in the sealed filter cassette up to one day after sampling. The filter is then carefully removed from the filter cassette using tweezers, transferred into a 10-ml amber glass bottle, immersed in 10 ml of IC eluent using a dispenser and briefly manually shaken. The sealed bottle is stored in the refrigerator at 2 to 8 °C until analysis.

For sample preparation, the amber glass bottle with the loaded filter is removed from the refrigerator and acclimatised to room temperature. This is followed by treatment in the laboratory shaker for 60 minutes at 300 rpm.

Approx. 4 ml of liquid are withdrawn from the suspension using a disposable syringe. The first half millilitre, filtered through a disposable filter (pore width 0.45 µm), is discarded. The subsequent filtrate is collected into an autosampler vial. The sample vial with the remaining liquid is sealed and stored in the refrigerator as a retention sample.

The blank sample ('field blank') is prepared in the same manner as the sample.

Additionally, in order to determine the reagent blank value, several millilitres of eluent are pipetted into an autosampler vial and analysed ('lab blank').

6 IC operating conditions

Apparatus:	Ion chromatograph with degasser, column oven, chemical and CO ₂ suppression
Detector:	Conductivity detector
Separation column:	Metrosep A Supp 16-250/4.0 and Metrosep A Supp 16 Guard/4.0 precolumn
Column temperature:	45 °C
Mobile phase:	7.5 mmol/l of sodium carbonate / 0.75 mmol/l of sodium hydroxide isocratic
Flow rate:	0.8 ml/min
Injection volume:	10 µl
Flow time:	50 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 dicarboxylic acid to be determined, the relevant calibration graph is used for evaluation. If the measured concentrations are above the calibration range, suitable dilutions must be prepared with the eluent and the analysis must be repeated. Furthermore, the prepared blank sample ('field blank') and the reagent blank value ('lab blank') must be analysed in the same manner as the analytical samples.

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 times for succinic acid, malonic acid, glutaric acid and oxalic acid are very close to one another under the chromatographic conditions specified in [Section 6](#). As it is impossible to achieve a baseline separation of the signals, a calibration of the individual substance was carried out for each dicarboxylic acid.

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 eluent volume, the dilution and the recovery into account the concentration of dicarboxylic acid in the workplace air is calculated according to [Equation 1](#). If a recovery of 100 ± 5% in the range of one tenth to twice the limit value is determined, then no correction in [Equation 1](#) should be carried out.

$$\rho = \frac{((C \times f_v) - C_{blank}) \times V \times 100}{V_{air} \times R} \quad (1)$$

where:

- ρ is the mass concentration of dicarboxylic acid in the air sample in mg/m³
- C is the concentration of dicarboxylic acid in the measured solution in mg/l

C_{blank}	is the concentration of the blank value (mean value) in mg/l
f_v	is the dilution factor
V	is the volume of the eluent in litres (0.01 litres in this case)
V_{air}	is the air sample volume in litres
R	is the recovery in %

10 Reliability of the method

The characteristics of the method were calculated as stipulated in DIN EN 482 (DIN 2021) and DIN 32645 (DIN 2008).

10.1 Repeatability

The repeatability was ascertained by analysing one dicarboxylic acid standard solution each in the intermediate concentration range of the calibration curve on six different days. The concentrations and the relative standard deviations thus determined are listed in Table 5.

Tab. 5 Repeatability

Dicarboxylic acid	Concentration [mg/l]	Relative standard deviation [%]
Oxalic acid	36.1	1.4
Malonic acid	45.6	0.68
Succinic acid	79.7	0.67
Glutaric acid	79.5	0.59
Adipic acid	87.8	0.53

10.2 Recovery and reproducibility

The recovery was determined by spiking six impregnated filters each with three different amounts of dicarboxylic acid using dicarboxylic acid solutions, whereby each filter was spiked with one dicarboxylic acid only.

Spiking Solution 1 was prepared for each dicarboxylic acid by weighing the respective dicarboxylic acid into a 100-ml volumetric flask, filling it to the mark with water (for chromatography) according to Table 6 and subsequent manual shaking. The concentrations of oxalic acid, malonic acid and succinic acid were 24 g/l, of glutaric acid 48 g/l and of adipic acid 12 g/l.

Tab. 6 Preparation of Spiking Solution 1 for spiking the filters

	Oxalic acid	Malonic acid	Succinic acid	Glutaric acid	Adipic acid
Spiking Solution 1					
Mass [mg]	3380 ^{a)}	2410	2420	4850	1210
Purity [%]	99.5	99.5	99	99	99.5
Volume [ml]	100	100	100	100	100
Concentration [mg/l]	24000	24000	24000	48000	12000

^{a)} weighed in as oxalic acid dihydrate

Spiking Solutions 2 and 3 were prepared by diluting Spiking Solution 1 with water (for chromatography) according to Table 7. The dicarboxylic acid concentrations of the spiking solutions are also listed there. The dilution factors are based on Spiking Solution 1.

Tab. 7 Preparation of Spiking Solution 2 and 3 for spiking the filters

	Oxalic acid	Malonic acid	Succinic acid	Glutaric acid	Adipic acid
Spiking Solution 2					
Dilution factor	2	2	10	2	5
Volume of Spiking Solution 1 [ml]	10	10	2	10	4
Volume [ml]	20	20	20	20	20
Concentration [mg/l]	12000	12000	2400	24000	2400
Spiking Solution 3					
Dilution factor	20	20	n/a	20	n/a
Volume of Spiking Solution 1 [ml]	1.0	1.0	n/a	1.0	n/a
Volume [ml]	20	20	n/a	20	n/a
Concentration [mg/l]	1200	1200	n/a	2400	n/a

The filters were spiked with concentrations corresponding to masses equivalent to 0.1 OELV, 1 OELV and 2 OELV for an air sample volume of 1.2 m³ as specified in Table 8.

In most cases, the filters were spiked with exactly 100 µl of spiking solution and then left to dry in the air. As the succinic acid solutions and the adipic acid solutions have low concentrations due to their solubility in water, a larger spiking volume was necessary. At spiking volumes of 200 µl and 400 µl, the filter was spiked with 100 µl each time and after drying another 100 µl was added accordingly, until the required spiking volume was reached.

Tab. 8 Pipetting scheme for the preparation of the spiked filters and the respective recovery

	Oxalic acid	Malonic acid	Succinic acid	Glutaric acid	Adipic acid
Concentration equivalent to 2 OELV^{a)}					
Spiking Solution	1	1	1	1	1
Volume [µl]	100	100	200	100	400
Content per filter [mg]	2.4	2.4	4.8	4.8	4.8
Recovery [%]	100.4	98.9	101.7	101.3	103.3
Concentration equivalent to 1 OELV^{a)}					
Spiking Solution	2	2	1	2	1
Volume [µl]	100	100	100	100	200
Content per filter [mg]	1.2	1.2	2.4	2.4	2.4
Recovery [%]	101.9	100.1	100.2	102.2	100.2
Concentration equivalent to 0.1 OELV^{a)}					
Spiking Solution	3	3	2	3	2
Volume [µl]	100	100	100	100	100
Content per filter [mg]	0.12	0.12	0.24	0.24	0.24
Recovery [%]	109.6	103.4	95.9	101.9	100.9

^{a)} for an eluent volume of 10 ml and an air sample volume of 1.2 m³

After drying, 1.2 m³ of clean air at a flow rate of 10 l/min, at 20 °C room temperature and 50% relative humidity were drawn through the spiked filters. The filter cassettes were then sealed with caps. The filters were prepared on the following day as described in [Section 5.3](#)

The preparation of the filters with the spiked intermediate mass (equivalent to 1 OELV) required a dilution with the IC eluent to double the volume, so that the concentration was within the calibration range. A dilution to five times the volume was necessary for the spiked high mass (equivalent to 2 OELV).

The analytical determination was carried out according to [Sections 6 and 7](#).

The recoveries are listed in [Table 8](#).

The mean recoveries are:

- Oxalic acid 104%
- Malonic acid 101%
- Succinic acid 99%
- Glutaric acid 102%
- Adipic acid 102%

Longer sampling periods:

Experiments with three filters spiked with the mass equivalent to 1 OELV according to [Table 8](#) and an air sample volume of 2.4 m³ equivalent to a sampling period of four hours at a flow rate of 10 l/min show similar recoveries (see [Table 9](#)).

Tab. 9 Recovery with double the air sample volume (2.4 m³)

	Oxalic acid	Malonic acid	Succinic acid	Glutaric acid	Adipic acid
Content per filter [mg]	1.2	1.2	2.4	2.4	2.4
Recovery [%]	101.9	103.1	100.8	101.9	101.9

Influence of the humidity:

The influence of the humidity was additionally checked at 35% and 80% relative humidity and an air temperature of 20 °C. There is no evidence that the recoveries of the dicarboxylic acids depend on the relative humidity in this range.

Correction of the analytical results:

The recovery and the relative humidity do not need to be taken into account for calculation of the analytical results.

10.3 Expanded uncertainty of the entire method

In order to determine the expanded uncertainty, six glass fibre filters were spiked with different masses of individual dicarboxylic acids as described in [Section 10.2](#), dried and subjected to all the steps of the sample preparation and analysis as described in [Sections 5.3, 6 and 7](#). The filters were therefore spiked with concentrations equivalent to 0.1 OELV, 1 OELV and 2 OELV at an air sample volume of 1.2 m³.

The expanded uncertainty was obtained by estimation of all relevant influencing parameters. It consists basically of two parts, the uncertainties for sampling and for analysis. In order to estimate the uncertainty for sampling, the uncertainty associated with the air sample volume and the sampling effectiveness for inhalable particles were determined according to Appendix C in DIN EN ISO 21832 ([DIN 2020](#)).

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

Table 10 summarises all the determined uncertainty contributions, whereby a differentiation is made between low, intermediate and high spiking of the filters at an air sample volume of 1.2 m³. Low, intermediate and high concentration are equivalent to the concentrations of 0.1 OELV, 1 OELV and 2 OELV. The uncertainty was calculated for sampling periods of 15 and 120 minutes.

Tab. 10 Determination of the uncertainty in %

Uncertainty	Sampling period 15 minutes	Sampling period 120 minutes
Oxalic acid		
<i>u</i> Sampling, transport, storage	9.7	9.3
<i>u</i> Recovery low concentration	9.8	9.8
<i>u</i> Recovery intermediate concentration	2.4	2.4
<i>u</i> Recovery high concentration	1.3	1.3
<i>u</i> Analytical variability	4.0	4.2
<i>U</i> Expanded low concentration	29	28
<i>U</i> Expanded intermediate concentration	22	21
<i>U</i> Expanded high concentration	21	21
Malonic acid		
<i>u</i> Sampling, transport, storage	9.6	9.2
<i>u</i> Recovery low concentration	3.8	3.8
<i>u</i> Recovery intermediate concentration	1.5	1.5
<i>u</i> Recovery high concentration	1.7	1.7
<i>u</i> Analytical variability	2.8	2.8
<i>U</i> Expanded low concentration	21	21
<i>U</i> Expanded intermediate concentration	20	20
<i>U</i> Expanded high concentration	20	20
Succinic acid		
<i>u</i> Sampling, transport, storage	9.6	9.2
<i>u</i> Recovery low concentration	4.4	4.4
<i>u</i> Recovery intermediate concentration	1.3	1.3
<i>u</i> Recovery high concentration	2.2	2.2
<i>u</i> Analytical variability	2.7	2.8
<i>U</i> Expanded low concentration	22	21
<i>U</i> Expanded intermediate concentration	20	20
<i>U</i> Expanded high concentration	21	20
Glutaric acid		
<i>u</i> Sampling, transport, storage	9.7	9.3
<i>u</i> Recovery low concentration	2.5	2.5
<i>u</i> Recovery intermediate concentration	2.7	2.7
<i>u</i> Recovery high concentration	1.8	1.8
<i>u</i> Analytical variability	2.7	2.7
<i>U</i> Expanded low concentration	21	20
<i>U</i> Expanded intermediate concentration	21	20
<i>U</i> Expanded high concentration	20	20

Tab. 10 (continued)

Uncertainty	Sampling period 15 minutes	Sampling period 120 minutes
Adipic acid		
<i>u</i> Sampling, transport, storage	9.8	9.4
<i>u</i> Recovery low concentration	1.8	1.8
<i>u</i> Recovery intermediate concentration	1.4	1.4
<i>u</i> Recovery high concentration	3.8	3.8
<i>u</i> Analytical variability	2.6	2.7
<i>U</i> Expanded low concentration	21	20
<i>U</i> Expanded intermediate concentration	20	20
<i>U</i> Expanded high concentration	22	21

With one exception, the expanded uncertainties for the analytical method described here were from 20% to 22% in the measurement range of one tenth up to double the limit value for a sampling period of 120 minutes. An expanded uncertainty of 29% was calculated for oxalic acid in the low concentration range (equivalent to 0.1 OELV).

10.4 Limits of quantification

The limits of quantification for the dicarboxylic acids were determined by means of a 10-point calibration in each case as stipulated in DIN 32645 (DIN 2008).

The 10-point calibration was obtained by weighing in the dicarboxylic acid and by multiple dilution steps as follows:

The quantities of dicarboxylic acid stated in Table 11 are each weighed into individual 100-ml volumetric flasks. These were filled to the mark with water (for chromatography) and shaken. This Solution 1 was diluted with water (for chromatography) to prepare Solution 2 according to Table 11.

Tab. 11 Preparation of the standard solutions to determine the limit of quantification

	Oxalic acid	Malonic acid	Succinic acid	Glutaric acid	Adipic acid
Solution 1					
Mass [mg]	254.1 ^{a)}	190.8	335.3	334.5	367.5
Purity [%]	99.5	99.5	99	99	99.5
Volume [ml]	100	100	100	100	100
Concentration [mg/l]	1810	1900	3320	3310	3660
Solution 2					
Dilution of Standard 1	approx. 1:75 ^{b)}	1:50	1:25	1:50	1:40
Concentration [mg/l]	23.8	38.0	133	66.2	91.4

^{a)} weighed in as oxalic acid dihydrate

^{b)} 660 µl of Solution 1 to 50 ml of water

Ten 10-ml volumetric flasks were prepared per dicarboxylic acid into which 5 ml of eluent have been previously placed. The volumes of Solution 2 shown in Table 11 were added according to Table 12. The flasks are then filled to the mark with eluent and shaken. Table 12 shows the concentrations of the ten calibration standards.

Tab. 12 Pipetting scheme for the preparation of the ten calibration standard solutions per dicarboxylic acid for determination of the limit of quantification

Calibration standard	Solution 2 [μl]	Oxalic acid [mg/l]	Malonic acid [mg/l]	Succinic acid [mg/l]	Glutaric acid [mg/l]	Adipic acid [mg/l]
I	10	0.024	0.038	0.133	0.066	0.091
II	20	0.048	0.076	0.266	0.132	0.183
III	30	0.072	0.114	0.398	0.199	0.274
IV	40	0.095	0.152	0.531	0.265	0.366
V	50	0.119	0.190	0.664	0.331	0.457
VI	60	0.143	0.228	0.797	0.397	0.548
VII	70	0.167	0.266	0.929	0.464	0.640
VIII	80	0.191	0.304	1.06	0.530	0.731
IX	90	0.215	0.342	1.20	0.596	0.823
X	100	0.238	0.380	1.33	0.662	0.914

The relative limits of quantification (LOQ) of the five dicarboxylic acids are listed in [Table 13](#). They are based on an eluent volume of 10 ml, an air sample volume of 1.2 m³ for the shift mean value or 0.15 m³ for the short-term exposure limit and a confidence interval of 95%.

Tab. 13 Limits of quantification (LOQ)

Dicarboxylic acid	LOQ absolute [mg]	LOQ relative Air sample volume of 0.15 m ³ [mg/m ³]	LOQ relative Air sample volume of 1.2 m ³ [mg/m ³]
Oxalic acid	0.00029	0.0020	0.0002
Malonic acid	0.00047	0.0032	0.0004
Succinic acid	0.00114	0.0076	0.0009
Glutaric acid	0.00060	0.0040	0.0005
Adipic acid	0.00078	0.0052	0.0007

10.5 Storage stability

15 filters per dicarboxylic acid were spiked with the equivalent of 0.1 OELV, 1 OELV and 2 OELV, as shown in [Table 8](#), in the same manner as for the recovery according to [Section 10.2](#)

After drying in the air, 1.2 m³ of clean air at a flow rate of 10 l/min were drawn through the filters and the filter cassettes were then sealed with caps. The following day, the filters were transferred into 10-ml amber glass bottles using tweezers, immersed in 10 ml of IC eluent using a dispenser and sealed. After being briefly shaken, the amber glass bottles were stored in the refrigerator at +5 °C. The bottles were removed from the refrigerator on the day of analysis, acclimatised and prepared in accordance with [Section 5.3](#)

Three filters per concentration were prepared and measured on the same day (without storage in the refrigerator), after one week, after two weeks, after three weeks and after four weeks in accordance with [Section 5.3](#)

The recoveries are listed in [Table 14](#).

Tab. 14 Recoveries for the storage stability experiments over four weeks

	Contents per filter [mg]	Equivalent to OELV ^{a)}	Recovery [%]
Oxalic acid	0.12	0.1	107–109
	1.2	1	102–105
	2.4	2	102–103
Malonic acid	0.12	0.1	102–105
	1.2	1	99–103
	2.4	2	100–101
Succinic acid	0.24	0.1	99–101
	2.4	1	100–101
	4.8	2	101–103
Glutaric acid	0.24	0.1	101–103
	2.4	1	101–103
	4.8	2	101–103
Adipic acid	0.24	0.1	100–103
	2.4	1	99–102
	4.8	2	100–102

^{a)} for an eluent volume of 10 ml and an air sample volume of 1.2 m³

The mean recoveries are:

- Oxalic acid 105%
- Malonic acid 102%
- Succinic acid 101%
- Glutaric acid 102%
- Adipic acid 101%

Storage stability of the loaded glass fibre filters for a period of 4 weeks is ensured.

10.6 Selectivity

Dicarboxylic acids and their salts (e.g. alkali salts) cannot be differentiated.

The anions fluoride, bromide, chloride and nitrite do not interfere with the analysis. In the case of high nitrate concentrations, there is a possibility that the analysis of succinic acid may be adversely affected due to overlapping peaks.

Substances, which co-elute with the individual dicarboxylic acids under the stated conditions, can yield a false positive signal. This applies e.g. to tartaric acid and tartrates, that have only a slightly shorter retention time than succinic acid. Separation is not possible under the selected chromatographic conditions.

If malonic acid is absent, oxalic acid, succinic acid, glutaric acid and adipic acid can be determined in one analysis (see [Figure 1](#)).

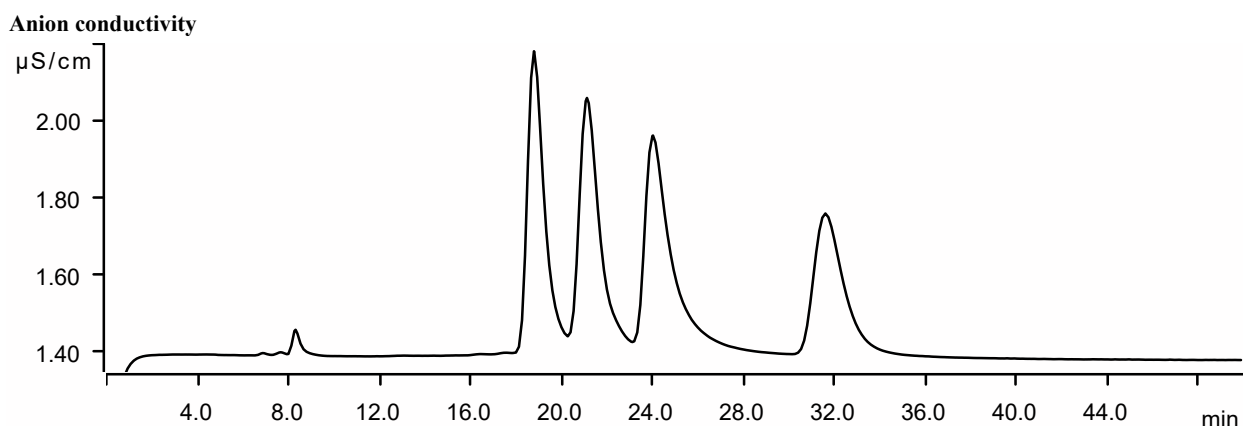


Fig. 1 IC chromatogram of the investigated dicarboxylic acids: succinic acid (18.8 min), glutaric acid (21.1 min), oxalic acid (24.0 min), adipic acid (31.6 min). Concentration per component approx. 12 to 24 mg/l

In the presence of other substances the following interferences may occur:

- Malic acid elutes before succinic acid and may possibly restrict the analysis of succinic acid.
- Sulphate and in particular phosphate can interfere in the analysis of oxalic acid in the conductivity detector. As sulphate and phosphate are not active in the UV range, it is possible to revert to UV detection of oxalic acid, if necessary.
- Sulphate elutes directly after glutaric acid and interferes in the analysis of glutaric acid in the conductivity detector.

Malonic acid can be determined beside oxalic acid and adipic acid (see [Figure 2](#)). The signals of succinic acid and glutaric acid partially overlap with the signal of malonic acid and greatly restrict the determination of malonic acid. Malic acid, sulphate and phosphate do not interfere in the analysis of malonic acid.

The dicarboxylic acids are optically active with low intensity, as are also malic acid and tartaric acid, and can be measured in the range of the calibration using the UV detector at a wavelength of 205 nm. This can be used as an additional qualitative identification characteristic. Oxalic acid is more sensitive than the other four dicarboxylic acids in the UV detector.

Blank values are taken into account due to the parallel sample work-up of the prepared field blanks; however, they could not be ascertained. No blank values were detected in the glass fibre filters and the eluent.

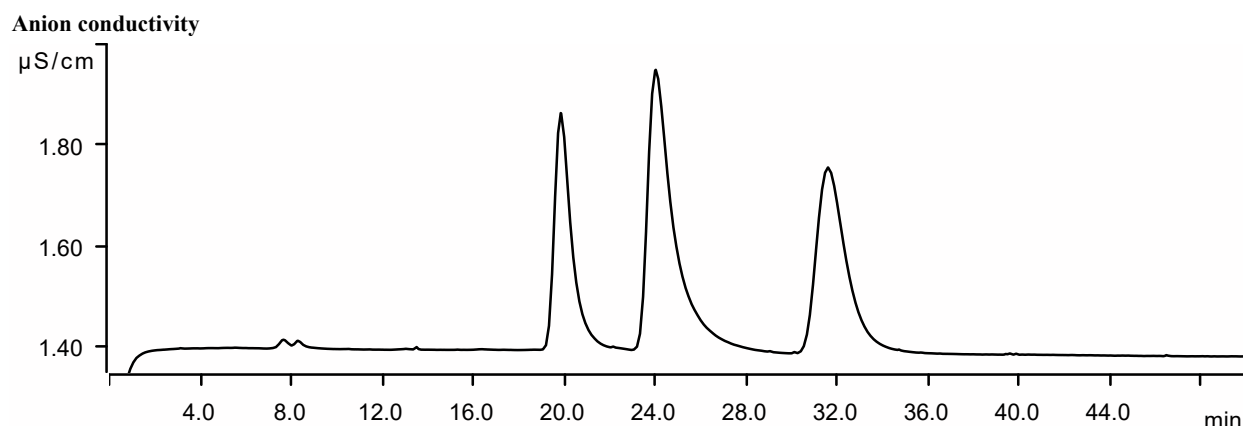


Fig. 2 IC chromatogram of the investigated dicarboxylic acids: malonic acid (19.9 min), oxalic acid (24.0 min), adipic acid (31.6 min). Concentration per component approx. 12 to 24 mg/l

11 Discussion

The analytical procedure described here permits the determination of oxalic acid, succinic acid, glutaric acid and adipic acid in the workplace air in a concentration range from a tenth to twice the currently valid OELV of 1 mg/m³ (inhalable particle fraction) for oxalic acid and 2 mg/m³ (inhalable particle fraction) for the other three dicarboxylic acids. Furthermore, the analytical method is suitable for monitoring compliance with the short-term exposure.

There is currently no OELV and no MAK value for malonic acid. Therefore, the concentration range for oxalic acid was adopted in this case.

The method is suitable for the determination of individual substances. If several dicarboxylic acids occur simultaneously in the workplace air, [Section 10.6](#) should be consulted.

The analytical method was validated for a flow rate of 10 l/min during sampling. Sampling with a flow rate of 3.5 l/min is also possible for the currently valid evaluation criteria, the sample preparation described here as well as the analytical method used. The method must then be validated for this flow rate.

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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