Hexavalent chromium – Determination of particular hexavalent chromium in workplace air using ion chromatography with post-column derivatisation (IC-UV/VIS)

Air Monitoring Method

K. Timm[1]

T. H. Brock[2,*]

R. Hebisch[3,*]

A. Hartwig[4,*]

MAK Commission[5,*]

1 Method development, German Social Accident Insurance Institution of the raw materials and chemical industry, Department Hazardous Substances, Biological Agents and Analytical Chemistry, Laboratory Leuna, Rudolf-Breitscheid-Straße 18E, 06237 Leuna, Germany

2 Head of the working group "Analytics", German Social Accident Insurance, Institution for the raw materials and chemical industry, Prevention – Department of Hazardous Substances, Biological Agents and Analytical Chemistry, Kurfürsten-Anlage 62, 69115 Heidelberg, Germany

3 Head of the working group "Air Analyses" of the Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Federal Institute for Occupational Safety and Health (BAuA), Friedrich-Henkel-Weg 1–25, 44149 Dortmund, Germany

4 Chair of the Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Institute of Applied Biosciences, Department of Food Chemistry and Toxicology, Karlsruhe Institute of Technology (KIT), Adenauerring 20a, Building 50.41, 76131 Karlsruhe, Germany

5 Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Kennedyallee 40, 53175 Bonn, Germany

* email: T. H. Brock (analytik@bgrci.de), R. Hebisch (luftanalysen-dfg@baua.bund.de), A. Hartwig (andrea.hartwig@kit.edu), MAK Commission (arbeitsstoffkommission@dfg.de)

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Please direct correspondence to Berufsgenossenschaft Rohstoffe und chemische Industrie, Prävention, P.O. Box 101480, 69004 Heidelberg, Germany; analytik@bgrci.de

Abstract

This analytical method is a validated measurement procedure for the determination of particular hexavalent chromium e.g. potassium chromate [7789-00-6], potassium dichromate [7778-50-9], chromium(VI) oxide [1333-82-0] and lead(II) chromate [7758-97-6] after personal or stationary sampling. Sampling is performed by drawing a defined volume of air through a polymer membrane filter using a suitable flow-regulated pump. After liquid desorption, the hexavalent chromium retained on the filter is analysed using ion chromatography with UV/VIS detection after post-column derivatisation. The relative limit of quantification (LOQ) is 0.10 µg Cr(VI)/m³ for an air sample volume of 1.2 m³. The mean recovery for soluble hexavalent chromium was 98 to 103%. The concentration-dependent expanded uncertainty was 22%. This analytical method has been

accredited by the accident insurance companies for the detection in workplace air of substances that are carcinogenic, mutagenic or toxic to reproduction. This method has been tested and recommended for the determination of hexavalent chromium in work areas by the German Social Accident Insurance (DGUV). Both personal and stationary sampling can be performed for measurements in order to evaluate work areas.

Name	CAS No.	Molar mass [g/mol]	Formula	Substance-specific concentration values [μg Cr(VI)/m^3]
Chromium(VI) compounds	–	–	–	TC 1 (I) EF 8 (AGS 2021, 2023)
Potassium chromate	7789-00-6	194.19	K$_2$CrO$_4$	See above
Potassium dichromate	7778-50-9	294.19	K$_2$Cr$_2$O$_7$	See above
Chromium(VI) oxide	1333-82-0	99.99	CrO$_3$	See above
Lead(II) chromate	7758-97-6	323.18	PbCrO$_4$	See above

EF: excursion factor; I: inhalable fraction; TC: tolerable concentration

1 Summary

This method involves the use of personal or stationary sampling procedures to determine the mean concentration of chromium(VI) in the work area over the sampling period.

Measurement principle: A pump is used to draw a defined volume of air through a polymer membrane filter made of polytetrafluoroethylene (PTFE). The chromium(VI) compounds deposited on the filter in the form of dust and aerosol particles are first extracted with an aqueous solution of ammonium sulphate and ammonia and then determined using ion chromatography with post-column derivatisation and UV/VIS detection (DAD).

Limit of quantification:
Absolute: 0.12 μg of Cr(VI)
0.0023 μg of Cr(VI) per sample carrier
Relative: 0.10 μg/m^3 of Cr(VI) for an air sample volume of 1.2 m^3 and 10 ml of measurement solution

Measurement range: Validated in the range of 0.10 to 2.0 μg/m^3 based on an air sample volume of 1.2 m^3.

Selectivity: An extraction solution is used for stabilisation to minimize interference from reducing and oxidising agents in the air that occurs particularly in the presence of chromium in lower oxidation states.
Chromatographic separation is used to eliminate the effects of other metal ions that react with diphenylcarbazide.

Advantages: Personal sampling procedures can be used to obtain measurements with high sensitivity.

Disadvantages: Does not show concentration peaks; requires the use of relatively expensive and complex apparatus

Apparatus: Sampling devices:
Pump and sampling head, filter holder with polymer membrane filter and supporting sieve
Volumetric flow meter
IC system equipped for post-column derivatisation

2 Equipment and chemicals

2.1 Equipment

For sampling:

- Pump, suitable for a flow rate of 10.0 l/min (e.g. SG10-2, from GSA Messgerätebau GmbH, 40880 Ratingen, Germany)
- GSP 10 sampling head, suitable for a flow rate of 10 l/min (e.g. from GSA Messgerätebau GmbH, 40880 Ratingen, Germany)
- Polymer membrane filters (polytetrafluoroethylene (PTFE) on non-woven polypropylene (PP) support web), diameter 37 mm, from a suitable batch (see Section 5.3) (e.g. Whatman TE38, from Whatman GmbH, 37586 Dassel, Germany)
- Filter capsule made of polyoxymethylene (POM) (e.g. from Kern GmbH, 56272 Großmaischeid, Germany)
- Supporting sieve made of polypropylen (PP) (e.g. support pad, 225-2902, diameter 37 mm, from SKC Inc., Eighty Four, PA, USA)
- Vials made of polyethylene terephthalate (PET), 10 ml, with screw caps (e.g. from Carl Roth GmbH + Co. KG, 76185 Karlsruhe, Germany) filled with 10 ml of extraction solution 2
- Volumetric flow meter (e.g. Gilibrator II, from Gilian, supplied i.a. by DEHA Haan & Wittmer GmbH, 71296 Heimsheim, Germany)

For the sample preparation and the analytical determination:

- Disposable plastic tweezers (e.g. ratiomed, Order No. 126300, from Megro GmbH, 46485 Wesel, Germany)
- Volumetric flasks made of PP, 10, 25, 50, 100 and 1000 ml
- Autosampler vials made of polystyrene, sealable, 10 ml (e.g. from Thermo Fisher Scientific GmbH, 63303 Dreieich, Germany)
- Syringe filters, PTFE, pore width 0.45 μm, diameter 13 mm (e.g. PERFECT-FLOW, WIC 79145, from Wicom Germany GmbH, 64646 Heppenheim, Germany)
- Variable piston pipettes to cover a volume range of 10 μl to 10 ml (e.g. Acura manual 825, 10–100 μl, from Socorex Isba S.A., CH-1024 Ecublens, Switzerland, Research plus, variable, 100–1000 μl, from Eppendorf SE, 22339 Hamburg, Germany, Transferpette S, 1–10 ml, from Brand GmbH + Co. KG, 97877 Wertheim, Germany)
- Ultrapure water unit with reverse osmosis device and ultrapure water system for the preparation of ultrapure water ($\rho \geq 18.2 \ M\Omega \times cm$ at 25 °C)
- Vacuum degasser
- Ultrasonic bath with cooling system (e.g. Sonocool SC255, from Bandelin electronic GmbH & Co. KG, 12207 Berlin, Germany)
- Microbalance (repeatability < 0.01 mg) (e.g. Quantos QB5, from Mettler-Toledo GmbH, 35396 Gießen, Germany)
- Ion chromatography system with column oven
- Autosampler
- Separation column equipped with a pre-column, configured for anions
- Pump for post-column derivatisation, free of metals and low pulsation levels
- Reaction loop
- Variable wavelength detector or diode-array detector

2.2   Chemicals

- Chromate, ion chromatography standard solution, 1000 µg/ml CrO_4^{2-} (e.g. Specpure, Order No. 42564, from Thermo Fisher Scientific, 76057 Karlsruhe, Germany), referred to in the following as standard solution 1

- Chromium(VI) standard for ICP, 1000 mg/l Cr(VI) (e.g. Supelco TraceCERT, Order No. 19036, from Merck KGaA, 64293 Darmstadt, Germany), referred to in the following as standard solution 2

- Ultrapure water, specific resistance $\rho \geq 18.2$ MΩ × cm at 25 °C, degassed with argon or nitrogen

- Ammonium sulphate, for analysis (e.g. Fisher Chemical, Order No. A/6480/60, from Fisher Scientific GmbH, 58239 Schwerte, Germany)

- Ammonia solution 20–22%, for ultra-trace elemental analysis (e.g. Fisher Chemical Optima A/470, from Fisher Scientific GmbH, 58239 Schwerte, Germany)

- Sulphuric acid 93–98%, for trace metal analysis (e.g. Fisher Chemical Trace Metal, Order No. A/510, from Fisher Scientific GmbH, 58239 Schwerte, Germany)

- Methanol, HPLC gradient grade (e.g. Fisher Chemical M/4058, from Fisher Scientific GmbH, 58239 Schwerte, Germany)

- 1,5-Diphenylcarbazide, for analysis (e.g. Supelco, Order No. 103091, from Merck KGaA, 64293 Darmstadt, Germany)

- Nitric acid 65%, trace metal grade (e.g. Supelco Suprapur, Order No. 100441, from Merck KGaA, 64293 Darmstadt, Germany)

- Oxalic acid dihydrate, for analysis (e.g. Supelco EMSURE 100441, from Merck KGaA, 64293 Darmstadt, Germany)

- Inert gas, e.g. Argon 5.0

2.3   Solutions

The eluent, solutions and samples are prepared using only degassed ultrapure water (see Section 2.2); this has proven to be an effective method for reducing chromatographic baseline noise.

Eluent: solution of 250 mmol ammonium sulphate/l and 100 mmol ammonia/l in water
33 g of ammonium sulphate are weighed out into a 1000-ml volumetric flask. 9.0 ml of ammonia solution are added while filling up the flask with ultrapure water.

Extraction solution 1: solution of 250 mmol ammonium sulphate/l and 1500 mmol ammonia/l in water
33 g of ammonium sulphate are weighed out into a 1000-ml volumetric flask. 135 ml of ammonia solution are added while filling up the flask with degassed ultrapure water.

This solution can be stored for up to three months.

Extraction solution 2: solution of 25 mmol ammonium sulphate/l and 150 mmol ammonia/l in water
A flask with 1 ml of extraction solution 1 is filled to 10 ml with degassed ultrapure water.

This solution can be used for two weeks.

Derivatisation reagent: solution of 2 mmol diphenylcarbazide/l in a water/methanol mixture
28 ml of sulphuric acid are placed into a flask containing about 500 ml of ultrapure water degassed with argon. 0.5 g of diphenylcarbazide are dissolved in 200 ml of methanol. After the sulphuric acid solution has cooled, the diphenyl-carbazide solution is added and the flask is filled to 1000 ml with degassed ultrapure water. Before use, the solution is vacuum-filtrated through a membrane filter.

The solution can be used for two weeks.

Rinsing solution 1: solution of 3 mol nitric acid/l in water
209 ml of nitric acid 65% are placed into a 1000-ml volumetric flask, which is then filled up with ultrapure water.

Rinsing solution 2: solution of 0.2 mol oxalic acid/l in water
About 25 g of oxalic acid dihydrate are dissolved in ultrapure water in a volumetric flask (1000 ml).

Stock solution: solution 1000 µg Cr(VI)/l in water
223 µl of chromate standard solution 1 are measured out into a 100-ml volumetric flask and the flask filled with ultrapure water.

This solution can be used for up to four weeks.

Spiking solution 1: solution of 100 µg Cr(VI)/ml in water
2230 µl of chromate standard solution 1 are measured out into a volumetric flask (10 ml) and the flask filled with ultrapure water.

This solution can be used for up to four weeks.

Spiking solution 2: solution of 10 µg Cr(VI)/ml in water
1000 µl of spiking solution 1 are measured out into a 10-ml volumetric flask and the flask filled with ultrapure water.

This solution can be used for up to four weeks.

Control sample: solution of 10 µg Cr(VI)/l in water
1000 µl of standard solution 2 are measured out into a 100-ml volumetric flask and the flask filled with ultrapure water. 25 µl of this solution are removed and placed into a second 25-ml volumetric flask, which is then filled with extraction solution 2.

This solution must be freshly prepared each working day.

The calibration solutions listed in Table 1 are prepared using the 1000 µg Cr(VI)/l stock solution as the base material. The specified volumes are measured out into 50-ml volumetric flasks, which are then filled with extraction solution 2.

Tab. 1   Concentrations of the calibration solutions

Volume stock solution [µl]	Concentration in the calibration solution [µg/l]	Concentration in air[a] [µg/m³]
250	5.0	0.042
500	10.0	0.083
750	15.0	0.125
1000	20.0	0.167
1250	25.0	0.208
1500	30.0	0.250
1750	35.0	0.292
2000	40.0	0.333
2250	45.0	0.375
2500	50.0	0.417

[a] based on an air sample volume of 1200 l and 10 ml of extraction solution 2

3   Sampling

Samples are taken by stationary or personal sampling. Directly before sampling, a filter holder containing a filter from a tested batch (see Section 5.3) is inserted into the sampling head of the GSP 10 sampling system. The volumetric flow rate is set at 10 l/min. The definition of the inhalable dust is fulfilled at this volumetric flow rate (DIN 1993). At a sampling period of two hours, this corresponds to an air sample volume of 1200 l.

After sampling, the flow rate must be tested for constancy. If the deviation from the adjusted flow rate is ± 5%, it is advisable to discard the measurement (see DGUV Information 213-500 "General Part", Section 3, DGUV 2015).

The filter holder with the loaded filter is removed from the GSP 10 sampling system immediately after sampling. Nitrile gloves must be worn during removal. Plastic tweezers are used to fold the filter twice and transfer it into a PET vial filled with 10 ml of extraction solution 2. When folding the filter, it is advisable to hold the filter with a second pair of tweezers. Care must be taken to ensure that the filter is completely immersed in the solution. The vial is sealed and briefly shaken.

The temperature of the samples must not exceed 40 °C during transport.

At least one additional filter is processed as a 'field blank' during sampling. This blank sample is prepared and analysed in the same manner as the other samples.

4 Analytical determination

4.1 Sample preparation

The PET vials with the loaded filters are sonicated in an ultrasonic bath at the highest setting for 30 minutes under controlled temperature conditions. The temperature in the bath must not exceed 25 °C. The vials with the samples are then removed from the ultrasonic bath. After a rest period of 30 minutes at room temperature in the laboratory, the sample extracts are transferred by syringe filter to autosampler vials, which are then sealed.

The field blank is prepared as described above for each series of samples as a control.

4.2 Operating conditions for chromatography

An autosampler is used to deliver the sample solutions into the sample loop of an ion chromatographic system. Measurements are taken in pairs for each sample solution.

To increase the detection sensitivity, after chromatographic separation the eluate is injected into a reaction loop with a diphenylcarbazide solution (Thermo Fisher Scientific Inc. 2014) that forms coloured complexes with chromium(VI) ions.

The characteristics of the method stated in Section 5 were obtained under the following operating conditions:

Ion chromatography system:	ICS 1100 RFIC with column oven, from Thermo Scientific, 38116 Braunschweig, Germany
Autosampler:	AS-AP, from Thermo Scientific
Eluent:	ammoniacal ammonium sulphate solution, see Section 2.3
Degasser:	1260 Infinity, from Agilent, 76337 Waldbronn, Germany
Column:	IonPac AS7, length 250 mm, inner diameter 2 mm, particle size 10 µm with pre-column IonPac AG7, length 50 mm, inner diameter 2 mm, particle size 10 µm, from Thermo Scientific
Column thermostat:	40 °C
Column flow rate:	0.36 ml/min
Reaction loop:	125 µl, 40 °C
Post-column derivatisation:	Diphenylcarbazide solution, see Section 2.3, flow rate 0.12 ml/min
Detector:	DAD UltiMate 3000, from Thermo Fisher Scientific

Detection: VIS (543 nm)

If the measured chromium(VI) concentration lies above the calibration range, the sample extract is diluted with ultrapure water to a concentration that lies within the calibration range.

To avoid carryover, the solutions listed below are injected into the system after about 50 samples or if the amount of analyte in the preceding samples exceeds the measurement range:

1.   Extraction solution 2
2.   Nitric acid 3 mol/l
3.   Extraction solution 2
4.   Oxalic acid 0.2 mol/l
5.   Extraction solution 2
6.   Extraction solution 2

5   Evaluation

5.1   Calibration

The calibration solutions listed in Table 1 are each injected three times and the mean of the peak areas used for calculation. The characteristics of the method are determined by means of a 10-point calibration. A 5-point calibration is sufficient for routine operations. A calibration function is constructed by plotting the mean peak areas against the corresponding concentration of chromium(VI). The calibration function is linear under the given conditions.

At the beginning of each sample sequence, a control sample is analysed to check for accuracy (see Section 2.3).

5.2   Calculation of the analytical result

The calibration curve is used to determine the chromium(VI) concentration in the solution based on the mean peak areas of the sample solutions. The mass concentration of chromium(VI) in the air sample is calculated according to the Equation 1:

$$c_m = \frac{c_{sol} \times V_{sol}}{1000 \times \eta \times V_{air}} \tag{1}$$

where:

c_m is the mass concentration of chromium(VI) in the air sample in µg/m³

c_{sol} is the chromium(VI) concentration in the measurement solution in µg/l that was obtained from the calibration curve

V_{sol} is the volume of the sample extract in ml

V_{air} is the air sample volume in m³

η is the recovery ($\eta = 1$ for readily soluble chromium(VI) compounds)

5.3 Analysis of the material used for the sample carrier

In general, PTFE filters have negligible blank values for chromium. Some batches, however, contain other matrix components that may lead to redox reactions. These reactions are unwanted because they have marked effects on measurement results, particularly in the low concentration range.

Before a batch of filters is used for sampling, a random sample of at least six empty filters is taken from the batch and analysed to ensure that there are no blank values.

To test for possible interference from other matrix components, a random sample of twelve filters is each spiked with 24 µl of spiking solution 2 (see Section 2.3). The filters are spiked with a load equivalent to that expected at a concentration in air of 0.2 µg/m³ and a sampling period of two hours.

As the filter material has poor wettability, care needs to be taken that all filters are spiked in a similar manner towards the centre of the filter surface. After the samples have dried at room temperature, they are placed into PET vials as described above and covered with 10 ml of extraction solution 2. The first series of six filters (series 1) is analysed after 24 hours, the remaining filters (series 2) are analysed after the standard storage period; maximally, however, after seven days.

The mean concentration values determined for chromium(VI) in both series of samples are compared with the nominal value of 0.2 µg/m³. If the mean of the values measured for series 1 lies below 90% of the nominal value, the batch of filters is not suitable for taking measurements according to the described method.

If the mean of the values measured for series 2 is at least 90% of the average amount of analyte determined in series 1, the batch of filters is suitable for taking measurements if the samples are analysed within the specified period. Otherwise, the samples must be analysed within 24 hours of sampling.

Filters with analysed field blank values must not contain detectable amounts of analyte. If these are detected, it is assumed that the measurement results were distorted by interference during the transport and storage of the samples.

6 Reliability of the method

The characteristics of the method were calculated as stipulated in DIN EN 482 (DIN 2021), DIN EN ISO 21832 (DIN 2020), and DIN 32645 (DIN 2008).

6.1 Precision and recovery

Recovery and precision were evaluated taking the sample preparation into account. Six filters each were spiked to different concentration levels with chromium(VI) spiking solutions (see Section 2.3). Each filter was analysed three times after simulated sampling (the filter was placed into the holder and the specified volume of air was drawn through the filter). The measured values are shown in Table 2.

Tab. 2 Recovery and coefficient of variation

Spiking	Concentration in air[a] [µg/m³]	Recovery [%]	Coefficient of variation [%]
12 µl spiking sollution 2	0.1	98.3	1.43
12 µl spiking solution 1	1.0	103.2	0.74
24 µl spiking solution 1	2.0	102.1	0.52

[a] based on an air sample volume of 1200 l and 10 ml of extraction solution 2

If poorly soluble chromates are found in the work area, it is assumed that these substances will not be recovered completely under the conditions described in Section 4.1. In this case, the calculation must take the experimentally determined recovery into account (see Table 3).

The recovery was determined as specified in DIN EN ISO 21832 (Appendix B) (DIN 2020) by weighing out about 1 mg each of several poorly soluble chromates into 50-ml PP vials (six vials each) using a microbalance, and covering the substances with 10 ml of extraction solution. After treatment in an ultrasonic bath and sedimentation, aliquots of the supernatant were diluted and analysed as described.

Tab. 3   Recovery of selected chromium(VI) compounds

Compound	Recovery [%]	Coefficient of variation [%]
Barium chromate	68	0.49
Lead chromate	21	0.73
Strontium chromate	51	0.06
Zinc chromate	60	0.30

6.2   Limit of quantification

The limit of quantification for the basic analytical method, not including sampling, was calculated to be 1.3 µg/l (equivalent to 0.01 µg/m^3 at 1200 l air volume and 10 ml of sample extract). The calculation was carried out according to DIN 32645 (DIN 2008) based on an equidistant 10-point calibration in the range of 5.0 to 50.0 µg Cr(VI)/l.

However, the material of the sample carrier has a marked effect on the limit of quantification for the entire measurement method (see Section 5.3). For this reason, it is necessary to determine the lowest analyte concentration at which sufficient stability of the loaded sample carriers can be maintained during storage (see Section 5.3). A value of 0.12 µg Cr(VI) per sample carrier was established as a feasible limit of quantification for a robust method. This results in a relative limit of quantification of 0.10 µg Cr(VI)/m^3 for an air sample volume of 1.2 m^3.

6.3   Storage stability

To minimise the effects of external sources of interference and to largely rule out reactions of the analyte with matrix components on the sample carrier that would distort the results, the time period between sampling and the analysis of the filters must be kept as short as possible.

The values in Table 4 represent the percentage of recovery from sample carriers spiked with the standard solution after a storage period of seven days under controlled laboratory conditions.

Tab. 4   Recovery after storage of loaded sample carriers for 7 days at room temperature (23 °C)

Concentration of chromium(VI) in air[a] [µg/m^3]	Recovery [%]	Coefficient of variation [%]
0.1	98.6	0.89
0.2	99.4	0.64
2.0	96.3	1.26

[a] based on an air sample volume of 1200 l and 10 ml of extraction solution 2

Carriers spiked with chromium(VI) in amounts of 0.24 µg and above did not show negative effects after one week even after being stored at higher temperatures (see Table 5).

Tab. 5 Recovery after storage of loaded sample carriers for 7 days at higher temperature (40 °C)

Concentration of chromium(VI) in air[a) [µg/m³]	Recovery [%]	Coefficient of variation [%]
0.2	99.0	1.33
2.0	100	0.98

[a) based on an air sample volume of 1200 l and 10 ml of extraction solution 2

6.4 Selectivity

If the samples contain chromium compounds with lower oxidation states, these may be oxidised under certain conditions, resulting in excessively high readings. Reducing agents in the air, on the other hand, may lead to false low readings.

Interference by other metal compounds (e.g. vanadium, iron) is eliminated by chromatographic separation.

Dissolved gas in the eluent increases the baseline noise, thereby reducing the sensitivity of the measurement. For this reason, all eluents must be degassed.

Chromium(VI) may accumulate in the pre-column and separation column over time. This is prevented by flushing the system after each measurement cycle (see Section 4.2). The condition of the system is monitored by injecting extraction solution.

6.5 Uncertainty

The expanded uncertainty was determined by estimating all relevant influencing parameters (bottom-up method). The main sources of uncertainty in the measurement results are uncertainties in the sampling procedure and in the analytical procedure.

The uncertainty was calculated taking into account the uncertainties associated with the air sample volume and the sampling effectiveness for inhalable dusts as well as for the entire analytical procedure, including digestion, dilution, calibration, equipment drift, recovery, precision and storage. The uncertainties contributed by the sampling procedure were estimated based on Appendix C of DIN EN ISO 21832 (DIN 2020).

The combined, concentration-dependent uncertainties for the entire method were calculated by combining the contributions from all sources of uncertainty. The expanded uncertainties for the entire method were obtained by multiplying the values with the expansion factor k = 2.

For the described method, the expanded uncertainties were calculated to be 22% for chromium(VI) concentrations in air of 0.1 µg/m³ and 2.0 µg/m³.

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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