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Method for the determination of hexavalent chromium in workplace air using photometry

Air Monitoring Method

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Abstract

This analytical method is a validated measurement procedure for the determination of hexavalent chromium in workplace air averaged over the sampling period after personal or stationary sampling. Sampling is performed by drawing a defined volume of air through a quartz fibre filter located in a GSP sampling head using a suitable flow-regulated pump. The flow rate is set to 3.5 or 10 L/min with a recommended air sample volume of 420 or 1200 L. The collected hexavalent chromium oxidised 1,5-diphenylcarbazide to 1,5-diphenylcarbazone, which forms a red-violet coloured complex with the generated trivalent chromium. The filtered extract is measured photometrically at 540 nm after a waiting time of 30 minutes within a period of 15 minutes. The quantitative determination is based on a calibration function obtained by means of multi-point calibrations. The absolute limit of quantification (LOQ) is 0.1 µg for hexavalent chromium deposited on the filter. The mean recovery was 104% and the expanded uncertainty for hexavalent chromium was approx. 20%.

Joint Publication of the Analytical Subcommittee of the Chemistry Board of Experts of the Expert Committee Raw Materials and Chemical Industry of the German Social Accident Insurance and the working group "Air Analyses" of the Permanent Senate Commission of the Deutsche Forschungsgemeinschaft for the Investigation of Health Hazards of Chemical Compounds in the Work Area.

Keywords

DGUV Information 213-505; hexavalent chromium; Cr(VI); 1,5-diphenylcarbazide; 1,5-diphenylcarbazone; GSP sampling head; air analysis; analytical method; workplace measurement; hazardous substances; photometry

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Method for the determination of hexavalent chromium in workplace air using photometry

**German Social Accident Insurance
Expert Committee Raw Materials and Chemical Industry
Subcommittee Hazardous Substances**

Analytical Subcommittee of the Chemistry Board of Experts¹⁾

**Recognised analytical procedures for the determination of carcinogens,
mutagens or substances toxic to reproduction**

Order number: DGUV Information 213-505, Method 04

Issued: March 2017

This method has been tested and recommended for the determination of hexavalent chromium in the air at workplaces by the German Social Accident Insurance.

Both personal and stationary sampling can be performed for risk assessment at work.

Sampling is carried out with a pump and collection on a quartz fibre filter. Analysis is performed by photometry after addition of diphenylcarbazide to the sample solution and a reaction time of 30 minutes.

Chemical names: Chromium(VI); Hexavalent chromium; Chromium(6+)ion
CAS number: 18540-29-9
Symbol: Cr⁺⁶
Atomic mass: 51.996 g/mol

Summary

This analytical method describes the determination of the mean concentration of hexavalent chromium in workplace air averaged over the sampling period after personal or stationary sampling.

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Principle:	A pump draws a defined volume of air through a quartz fibre filter. The hexavalent chromium compounds retained on the filter in the form of dusts and aerosols are determined using the diphenylcarbazide method [1].
Limit of quantification:	<p>Absolute: 0.1 µg of Cr(VI) for a measurement solution of 50 mL and a cuvette layer thickness of 50 mm.</p> <p>Relative: 0.24 µg/m³ of Cr(VI) for an air sample volume of 0.42 m³, an extraction solution of 10 mL, a measurement solution of 50 mL and a cuvette layer thickness of 50 mm.</p> <p>0.48 µg/m³ of Cr(VI) for an air sample volume of 0.42 m³, an extraction solution of 10 mL, a filtrate solution of 5 mL, a measurement solution of 50 mL and a cuvette layer thickness of 50 mm, aliquot factor 2.</p> <p>0.083 µg/m³ of Cr(VI) for an air sample volume of 1.2 m³ an extraction solution of 10 mL, a measurement solution of 50 mL and a cuvette layer thickness of 50 mm.</p> <p>0.17 µg/m³ of Cr(VI) for an air sample volume of 1.2 m³, an extraction solution of 10 mL and a filtrate solution of 5 mL, a measurement solution of 50 mL and a cuvette layer thickness of 50 mm, aliquot factor 2.</p>
Selectivity:	Interference might be caused from reducing agents present in the air. Oxygen can lead to oxidation of the collected trivalent chromium during the alkaline preparation. The presence of molybdenum(VI), copper(II), mercury(I/II), iron(III) and vanadium(V) compounds can also lead to interference.
Advantages:	Personal sampling is possible, does not require expensive or complicated apparatus.
Disadvantages:	No indication of concentration peaks.
Apparatus:	Pump, flow meter, quartz fibre filter, photometer

Detailed description of the method

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1 Equipment, chemicals and solutions

1.1 Equipment

For sampling:

- Pump, suitable for a flow rate of 3.5 L/min, e.g. SG4000, from GSA Gesellschaft für Schadstoffanalytik GmbH, 40880 Ratingen, Germany
- Pump, suitable for a flow rate of 10.0 L/min, e.g. SG10-2, from GSA
- GSP sampling head with intake cone adjusted to a flow rate of 3.5 L/min or 10 L/min, from GSA
- Quartz fibre filter, Ø 37 mm (a check for blank values is necessary), e.g. Whatman QM-A 1851-037 supplied by Sigma-Aldrich, 82024 Taufkirchen, Germany
- Filter holder made of polyoxymethylene (POM), from Kern, 56276 Großmaischeid, Germany
- Flow meter, e.g. Gilibrator II from Gilian supplied by DEHA Haan & Wittmer, 71296 Heimsheim, Germany

For sample preparation and analytical determination:

- Double beam UV/Vis spectrophotometer, e.g. UV2-200, from Unicam Chromatography, 34132 Kassel, Germany
- Quartz cuvettes, layer thickness 50 mm

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- Analytical balance
- Volumetric flasks, 50, 100 and 1000 mL
- Variable piston pipettes, dosing ranges 20–100 µL, 200–1000 µL and 1–5 mL
- Measuring cylinders, 100 mL and 250 mL
- Reaction vessels made of polypropylene (PP) with screw caps, nominal volume of 15 mL
- Funnel made of polypropylene (PP), Ø 50 mm
- Folded paper filters (without blank values and tested for product strength)
- Ceramic tweezers
- Ceramic spatula
- Dispenser, 1–10 mL, 10–50 mL with a 1 L glass bottle
- Ultrapure water unit, e.g. from Wilhelm Werner, 51381 Leverkusen, Germany

1.2 Chemicals

- Potassium dichromate, p.a., 99.9%, e.g. from Merck, 64293 Darmstadt, Germany
- Sodium hydroxide, p.a., 99%, e.g. from Merck
- Sodium carbonate, p.a., 99.9%, e.g. from Merck
- Sulphuric acid, p.a., 95–97%, e.g. from Merck
- Phosphoric acid, p.a., 85%, e.g. from Merck
- 1,5-Diphenylcarbazide, p.a., e.g. from Merck
- Acetone, p.a., e.g. from Merck
- Acetic acid, 100% (glacial acetic acid), e.g. from Merck
- Ultrapure water (Conductivity $\leq 0.1 \mu\text{S}/\text{cm}$ at 25 °C)

1.3 Solutions

Sodium hydroxide:	1.0 mol/L of sodium hydroxide in water Approx. 40 g of sodium hydroxide are weighed into a 1000 mL volumetric flask. The volumetric flask is then filled to the mark with ultrapure water and shaken.
Extraction solution:	0.5 mol/L of sodium hydroxide and 0.28 mol/L of sodium carbonate in water Approx. 20 g of sodium hydroxide and approx. 30 g of sodium carbonate are weighed into a 1000 mL volumetric flask. The volumetric flask is then filled to the mark with ultrapure water and shaken.
Acid solution:	1.4 mol/L of sulphuric acid and 1.4 mol/L of phosphoric acid in water Approx. 500 mL of ultrapure water are placed into a 1000 mL volumetric flask. Then 80 mL of sulphuric acid and 100 mL of phosphoric acid are carefully added and the volumetric flask is filled to the mark with ultrapure water and shaken.

Diphenylcarbazide solution:	Solution of 10.0 g/L of 1,5-diphenylcarbazide in acetone/glacial acetic acid 0.5 g of 1,5-diphenylcarbazide are weighed into a 50 mL volumetric flask and 50 μ L of glacial acetic acid are added. The volumetric flask is then filled to the mark with acetone and shaken.
Chromate stock solution:	Solution of approx. 500 mg/L of hexavalent chromium in 1 molar sodium hydroxide solution The potassium dichromate is dried overnight in the drying cabinet at 105 °C and then stored in a desiccator containing silica gel orange. Approx. 141 mg of potassium dichromate are weighed exactly to the nearest 0.1 mg into a 100 mL volumetric flask. The volumetric flask is then filled to the mark with sodium hydroxide and shaken. This stock solution is stable for at least 6 months, if stored in a cool place.
Chromate standard solution 1:	Solution of 10 mg/L of hexavalent chromium in 1 molar sodium hydroxide solution Using a pipette, 1 mL of the chromate stock solution is dosed into a 50 mL volumetric flask, into which approx. 10 mL of sodium hydroxide solution have been previously placed. The volumetric flask is then filled with sodium hydroxide and shaken. This solution is stable for at least 3 months, if stored in a cool place.
Chromate standard solution 2:	Solution of 1 mg/L of hexavalent chromium in 1 molar sodium hydroxide solution Using a pipette, 5 mL of the chromate standard solution 1 are dosed into a 50 mL volumetric flask, into which approx. 10 mL of the sodium hydroxide solution have been previously placed. The volumetric flask is then filled to the mark with sodium hydroxide and shaken. This solution must be freshly prepared every working day.

The calibration solutions listed in Table 1 and Table 2 are prepared from chromate standard solutions 1 and 2.

For this purpose approx. 10 mL of the extraction solution are placed into 50 mL volumetric flasks. The volumes of chromate standard solution 1 and chromate standard solution 2 listed in Table 1 and Table 2 are then added and 6 mL of the acid solution are then carefully pipetted into the respective volumetric flasks. The volumetric flasks are gently shaken in order to completely expel the generated carbon dioxide. Then 2 mL of the diphenylcarbazide solution are added, and the volumetric flask is filled to the mark with ultrapure water and shaken. After a standing time of 30 minutes, the absorbances of the solutions are measured within a period of 15 minutes.

The calibration solutions must be freshly prepared before each new calibration.

Two solutions, which have been prepared independently from the calibration solutions described above, are used to check the calibration solutions and each measurement series (see Table 3). In each case, the concentrations are in the middle calibration range of the individual calibrations. The control solutions must be freshly prepared before each measurement.

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Table 1: Concentrations of the calibration solutions in the working range of 0.1 µg to 1.0 µg Cr(VI) absolute for a measurement solution of 50 mL

Addition of standard solution 2	Concentration of Cr(VI)	Concentration of Cr(VI)	Concentration of Cr(VI)
[µL]	[µg/50 mL]	[µg/m³]*	[µg/m³]**
100	0.1	0.48	0.17
200	0.2	0.95	0.33
300	0.3	1.43	0.50
400	0.4	1.90	0.67
500	0.5	2.38	0.83
600	0.6	2.86	1.00
700	0.7	3.33	1.17
800	0.8	3.81	1.33
900	0.9	4.28	1.50
1000	1.0	4.76	1.67

* based on an air sample volume of 420 L and an aliquot factor of 2

** based on an air sample volume of 1200 L and an aliquot factor of 2

2 Sampling

Sampling can be carried out as stationary or personal sampling. The filter holder containing the filter (from a batch checked for blank values) is inserted into the GSP sampling system immediately before sampling commences. A flow rate of 3.5 or 10 L/min is then set. This is equivalent to an air sample volume of 420 L or 1200 L respectively for a sampling period of 2 hours. After sampling, the flow rate must be checked for constancy. If the deviation from the adjusted flow rate is greater than $\pm 5\%$, it is advisable to discard the measurement (*see* DGUV Information 213-500 "General Part", Section 3 [2]).

Immediately after sampling the filter holder with the loaded filter is removed from the GSP sampling system and sealed with the caps in preparation for transport. The important parameters for the determination of the concentration in air are documented in a sampling record.

3 Analytical determination

3.1 Sample preparation

3.1.1 Complete transfer of the extract

The loaded filters are transferred into 15 mL reaction vessels made of polypropylene, covered with 10 mL of the extraction solution and allowed to stand for approx.

Table 2: Concentrations of the calibration solutions in the working range of 1.0 µg to 10.0 µg Cr(VI) absolute for a measurement solution of 50 mL

Addition of standard solution 1	Concentration of Cr(VI)	Concentration of Cr(VI)	Concentration of Cr(VI)
[µL]	[µg/50 mL]	[µg/m ³]*	[µg/m ³]**
100	1.0	4.8	1.7
200	2.0	9.5	3.3
300	3.0	14.3	5.0
400	4.0	19.0	6.7
500	5.0	23.8	8.3
600	6.0	28.6	10.0
700	7.0	33.3	11.7
800	8.0	38.1	13.3
900	9.0	42.8	15.0
1000	10.0	47.6	16.7

* based on an air sample volume of 420 L and an aliquot factor of 2

** based on an air sample volume of 1200 L and an aliquot factor of 2

Table 3: Concentration of the control solutions

Control solution	Addition of standard solution 1	Concentration of Cr(VI)
No.	[µL]	[µg/50 mL]
1	50	0.5
2	500	5.0

18 hours. The respective extract is then filtered through a folded paper filter into a 50 mL volumetric flask. For the purpose of quantitative transfer, the sample vessel and filter are rinsed with at least 20 mL of ultrapure water. Subsequently, 6 mL of the sulphuric acid solution and 2 mL of the diphenylcarbazide solution are added to the filtered sample solution and the volumetric flask is filled to the mark with ultrapure water. After a waiting time of 30 minutes, the absorbances of the solutions are measured within a period of 15 minutes [3].

3.1.2 Use of an aliquot of the sample

The loaded filters are transferred into 15 mL reaction vessels made of polypropylene, covered with 10 mL of the extraction solution and allowed to stand for approx. 18 hours. The respective extract is then filtered through folded paper filter and 5 mL of the filtrate is pipetted into a 50 mL volumetric flask. 5 mL of the extraction solution and 6 mL of the acid solution are also added to the volumetric flask and

this is gently shaken in order to expel the resulting carbon dioxide. Then 2 mL of the diphenylcarbazine solution are added and the volumetric flask is filled to the mark with ultrapure water and shaken. After a waiting time of 30 minutes, the absorbances of the solutions are measured within a period of 15 minutes.

The blank value is determined by preparing and analysing a non-spiked filter in the same manner as the analysis samples.

3.2 Photometric working conditions

UV/Vis-Photometer:	Double Beam UV/Vis Spectrophotometer, e.g. UV2-200, from Unicam Chromatography
Cuvette:	Quartz cuvettes, layer thickness 50 mm
Temperature:	Room temperature
Measured wavelength:	540 nm
Slit width, monochromator:	2 nm

4 Evaluation

4.1 Calibration

The calibration solutions described in Section 1.3 are each filled into the quartz cuvette and the respective absorbance is determined using a photometer with respect to ultrapure water in the reference cuvette.

The calibration function is obtained by plotting the absorbances versus the corresponding concentrations of hexavalent chromium.

4.2 Calculation of the analytical result

The mass of hexavalent chromium is determined from the absorbance of the measurement solution with the help of the calibration function. The concentration c_m of hexavalent chromium in the air sample in mg/m^3 is calculated according to Equation (1) as follows:

$$c_m = \frac{m \cdot F}{V \cdot \eta} \tag{1}$$

where:

- c_m is the mass concentration of hexavalent chromium in the air sample in mg/m^3
- m is the mass of hexavalent chromium in the measurement solution in mg determined using the calibration curve and corrected for the blank value
- F is the aliquot factor (in the case of complete transfer of extraction solution 1)
- V is the air sample volume in m^3
- η is the recovery

If the concentration of hexavalent chromium is outside the calibration range, then the prepared measurement solution is diluted with ultrapure water to a value within the calibration range and the measurement is repeated.

5 Reliability of the method

5.1 Precision and recovery

The precision and recovery are determined using spiked filters.

The volumes of chromate standard solution 1 used and the equivalent concentrations are listed in Table 4. The filters were dried overnight at room temperature after being spiked with chromate standard solution 1. Spiking was carried out in two steps, separated by a drying period, to give the highest concentration.

On the following day after drying, the variation coefficients were determined by preparing 10 spiked filters per concentration according to Section 3.1.2. The results are shown in Table 4.

For the purpose of determining the recovery, 10 spiked and dried filters per concentration were transferred into the sampling system. 420 L or 1200 L of air were then drawn through the filters. The samples were prepared as described in Section 3.1.2.

The recoveries of hexavalent chromium were between 0.98 and 1.08 for all experiments. The mean recovery was 1.04.

In recovery tests of chromates that are considered poorly soluble or insoluble, such as barium, lead, strontium and zinc chromates, approx. 1 mg of the respective chromate was applied to 10 weighed filters each in a stream of air of approx. 3.5 L/min. The following recoveries were determined for filters prepared as described in Section 3.1:

Barium chromate:	0.67
Lead chromate:	0.84
Strontium chromate:	0.91
Zinc chromate:	0.79

If it is assumed that these chromates are present during sampling, then the respective recoveries must be taken into consideration when calculating the results.

Table 4: Variation coefficients for three different concentrations and n = 10 determinations in each case

Dosage volume of standard solution	Concentration of Cr(VI)	Concentration of Cr(VI)	Variation coefficient
[μL]	[$\mu\text{g}/\text{filter}$]	[$\mu\text{g}/\text{m}^3$]*	[%]
30	0.3	0.25	3.3
1000	10.0	8.3	1.0
2 × 1000	20.0	16.7	0.37

* based on an air sample volume of 1200 L; an aliquot factor of 2

5.2 Limit of quantification

The limit of quantification was determined on the basis of the equidistant 10-point calibration over a range of one order of magnitude in the lower working range according to DIN 32645 [4].

The absolute limit of quantification of the procedure is 0.1 µg for hexavalent chromium deposited onto the filter. Using the different sampling systems and the preparation options the following relative limits of quantification are obtained at a sampling period of two hours, a measurement solution of 50 mL in each case and using a 50 mm quartz cuvette:

- 0.24 µg/m³ for an air sample volume of 420 L (preparation according to Section 3.1.1)
- 0.48 µg/m³ for an air sample volume of 420 L (preparation according to Section 3.1.2)
- 0.083 µg/m³ for an air sample volume of 1200 L (preparation according to Section 3.1.1)
- 0.17 µg/m³ for an air sample volume of 1200 L (preparation according to Section 3.1.2)

The limit of quantification can be further lowered at workplaces with only a small amount of dust by increasing the sampling period. The sampling period should, however, only exceed four hours in exceptional cases.

5.3 Storage stability

It is advisable to prepare the loaded filters within 24 hours. If this is not possible, then the storage period can be extended to 5 days by cooling to 4 °C.

Loaded filters can be stored in the extraction solution for up to 8 days at room temperature, if they are placed in 10 mL of the extraction solution immediately after sampling.

5.4 Selectivity

Interference can be caused by reducing agents (e.g. low metal oxides) present in the air. The presence of oxygen can lead to the oxidation of trivalent chromium during the alkaline preparation.

Cross-sensitivities due to the presence of iron(III), vanadium(V), mercury(I), mercury(II) and molybdenum(VI) compounds as well as nitrates have also been observed. Overestimation of the Cr(IV) concentration may occur in the presence of vanadium(V) compounds, but only when its concentration exceeds 10 times that of the hexavalent chromium present and, in the case of molybdenum(VI) compounds as well as mercury(I) and mercury(II) compounds, only above a concentration of these substances of 200 mg/L [1].

Interference due to iron(III) compounds are significantly reduced by the addition of phosphoric acid.

Lowered values due to mercury(I) and mercury(II) compounds are prevented by the addition of sodium chloride. Interference caused by nitrates are prevented by addition of sodium dihydrogen phosphate (pH = 2). These substances are added before the dosing of the diphenylcarbazide solution to the sample solutions.

5.5 Uncertainty

According to EN 13890 [5] the estimation of the uncertainty with regard to sampling of inhalable particles results in an error due to the sampling system of 8.5%, taking reductions for the calibration of the of the test system of the sampling device (0.5%), the estimation of the aggregated concentration (4.0%) and the systematic measurement deviation from the sampling convention (7.5%) into account. Taking a sampling period of 120 minutes into consideration, the total error is 9.2% for sampling.

An error of 2.9% for the transport of the samples is calculated in accordance with EN 13205-6 [6].

Taking the variation coefficients for the analysis at three different concentrations and ten determinations in each case into account, the combined and expanded uncertainties listed in Table 5 are obtained.

Table 5: Combined and expanded uncertainties

Concentration of Cr(VI) [$\mu\text{g}/\text{m}^3$]	Combined uncertainty [%]	Expanded uncertainty [%]
0.27	10.2	20.4
8.3	9.7	19.4
16.7	9.7	19.4

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