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Method for the determination of cobalt and its compounds in workplace air using atomic absorption spectrometry with the graphite furnace technique (GFAAS) after high-pressure microwave digestion

Air Monitoring Method

Keywords

cobalt, air analysis, workplace measurement, hazardous substances, air sampling, measurement method, graphite furnace technique, atomic absorption spectroscopy, respirable fraction

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Abstract

This analytical method is a validated measurement procedure for the determination of cobalt and its compounds in workplace air averaged over a 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 FSP sampling head using a suitable flow-regulated pump. The flow rate is set to 10 l/min with a recommended air sample volume of 1200 l which corresponds to a sampling period of 2 h. The collected respirable dust fraction is treated with a mixture of sulphuric acid (65%) and hydrochloric acid (30%) and analysed using atomic absorption spectrometry with the graphite

furnace technique (GFAAS). The quantitative determination is based on a calibration function obtains by means of a 10-point calibration. For the respirable dust of cobalt and its compound the limit of quantification (LOQ) is 0.058 µg/m³ based on an air sample volume of 1200 l.

This method has been tested and recommended for the determination of cobalt and its compounds in the air at workplaces by the German Social Accident Insurance. The following table lists cobalt as well as giving examples of several relevant compounds:

Substance	CAS number
Metallic cobalt	7440-48-4
Cobalt dichloride	7646-79-9
Cobalt sulphate	10124-43-3

Both personal and stationary sampling can be employed for the assessment of workplaces.

1 Summary

This analytical method describes the determination of the mean concentration of cobalt and its compounds at the workplace over the sampling period using personal or stationary sampling.

Measurement Principle: A pump draws a defined volume of air through a quartz fibre filter. The respirable fraction deposited on the filter is analysed to determine cobalt using an atomic absorption spectrometer with a graphite furnace after high-pressure microwave digestion.

Limit of quantification (calculated as Co):

Absolute: 56 pg of cobalt
per sample carrier: 0.07 µg of cobalt

Relative: 0.058 µg/m³ for an air sample volume of 1.2 m³ (2 h of sampling at an intake rate of 10 l/min as well as a measurement solution of 25 ml and an injection solution of 20 µl)

Measurement range: 0.058 to 95 µg/m³, validated in the range of 0.058 to 0.83 µg/m³ based on an air sample volume of 1.2 m³ and 0.17 to 95 µg/m³, based on an air sample volume of 420 l.

Selectivity: Interference by other components was not observed. Possible matrix effects can be corrected by calibration with the sample in accordance with the standard addition method. It is not possible to differentiate between the individual cobalt compounds.

Advantages: It is possible to conduct personal sampling with great sensitivity and determine other analytes in the sample solution with the atomic spectrometric parameters adapted accordingly.

Disadvantages: No indication of concentration peaks, requires great technological resources.

Apparatus:

Sampling system:
consisting of pump and sampling head, filter holder with quartz fibre filter and a support screen
Flow meter
High-pressure microwave digestion system
Atomic absorption spectrometer with graphite furnace technique.

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, Gesellschaft für Schadstoffanalytik mbH, 40880 Ratingen, Germany
- FSP sampling head, suitable for a flow rate of 10 l/min
- Support screen and filter holder made of PTFE
- Quartz fibre filter, diameter 37 mm, e.g. QM-A from Whatman, supplied by VWR, 64295 Darmstadt, Germany
- Flow meter e.g. rotameter flow meter, influx 1 to 13 litres, from DEHA Haan & Wittmer, 71296 Heimsheim, Germany

For sample preparation and analytical determination:

- Atomic absorption spectrometer with graphite furnace technique, autosampler and cobalt hollow cathode lamp
- High-pressure microwave digestion system, e.g. MarsXpress from CEM, 47475 Kamp-Lintford, Germany, with digestion vessels and seals made of PTFE
- Analytical balance
- Plastic tweezers
- Variable piston pipettes in the volumetric measurement range of 100 to 50 000 µl, e.g. Multipette Pro from Eppendorf, 22339 Hamburg, Germany
- Volumetric flasks 10, 25 ml
- Glass funnel, e.g. diameter of 55 mm
- Wide-necked flasks, 50 ml made of PE
- Disposable syringes, 5 ml made of PE
- Syringe filter made of regenerated cellulose, pore width 0.45 µm, diameter 30 mm, e.g. from Roth, 76185 Karlsruhe, Germany
- Autosampler vials made of PE or PTFE

2.2 Chemicals

- Nitric acid, 65%, with a low metal content, e.g. Suprapur, from Merck, 64293, Darmstadt, Germany
- Hydrochloric acid, 30%, with a low metal content, e.g. Suprapur, from Merck
- Magnesium nitrate hexahydrate, with a low metal content, e.g. Suprapur, from Merck
- Water for trace analysis, with a low metal content, e.g. Tracepur, from Merck
- Cobalt standard solution for AAS with a certified cobalt content of 1000 mg/l in dilute nitric acid, traceable to a standard reference material, e.g. from Merck (Order No. 05205)
- Multi-element standard solution 4 for ICP, with a certified cobalt content of 10 mg/l in dilute nitric acid, traceable to a standard reference material, e.g. from Merck (Order No. 51844)
- Argon 5.0 (purity at least 99.999%).

2.3 Solutions

Note: All glassware must be pre-washed with dilute nitric acid and then rinsed with water for trace analysis, in order to ensure that all vessels are free of any traces of metal.

Modification solution: Solution of 3 g of magnesium nitrate/l of water

Water for trace analysis is placed into a 25 ml volumetric flask. Approx. 130 mg of magnesium nitrate hexahydrate are weighed out and transferred into the 25 ml volumetric flask. The volumetric flask is filled to the mark and shaken.

Blank value solution: Solution of 0.65% nitric acid in water

0.1 ml of nitric acid (65%) is pipetted into a 10 ml volumetric flask, into which approx. 5 ml of water for trace analysis has been previously placed. The volumetric flask is filled to the mark and shaken.

Calibration solution 1: Solution of 4 mg of cobalt/l of water

Water is placed into a 25 ml volumetric flask. 100 µl of the cobalt standard (1000 mg of Co/l) are pipetted into the volumetric flask, the flask is then filled to the mark and shaken.

Calibration solution 2: Solution of 40 µg of cobalt/l of water

In order to stabilise the dilution, 0.25 ml of nitric acid (65%) is pipetted into a 25 ml volumetric flask, into which water has been previously placed. Then 250 µl of calibration solution 1 are pipetted into the volumetric flask, which is then filled to the mark with water and shaken. This solution is stable for a period of at least three weeks.

Control solution: Solution of 20 µg of cobalt/l of water

In order to stabilise the dilution, 0.25 ml of nitric acid (65%) is pipetted into a 25 ml volumetric flask, into which water has been previously placed. Then 50 µl of multi-element standard solution 4 (10 mg of Co/l) are pipetted into the volumetric flask, which is then filled to the mark with water and shaken. This solution is stable for a period of at least three weeks.

Cobalt solution 1: Solution of 10 mg of cobalt/l of water

Water is placed into a 10 ml volumetric flask. 100 µl of the cobalt standard (1000 mg of Co/l) are pipetted into the volumetric flask, the flask is then filled to the mark with water and shaken.

3 Sampling

The filter batch used must to be checked for its suitability before carrying out the measurements (see Section 5.2).

Sampling can be carried out as stationary or personal sampling. The caps of the filter transport capsules are removed and a quartz fibre filter (Ø 37 mm) with a support screen is placed into the FSP sampling system. The flow rate is then adjusted to 10 l/min. This is equivalent to an air sample volume of 1200 l 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 ± 5%, it is advisable to discard the measurement (see DGUV Information 213-500 “General Part”, *Section 3* (DGUV 2015)).

A blank sample (field blank value) is included in the measurement series in addition to the samples. This serves as a transport check and differs from the analytical sample in that no sample air is drawn through the filter.

The loaded filters are then removed from the sampling system and sealed with the caps of the transport capsules.

4 Analytical determination

4.1 Sample preparation

The loaded filters and the field blank are each transferred into a separate digestion vessel using plastic tweezers and 6 ml of nitric acid (65%) and 3 ml of hydrochloric acid (30%) are added to each vessel. The vessels are covered with the lids and sealed with the screw-caps. These are then evenly distributed on the sample turntable of the microwave digestion device and the dust deposited on the filters is digested.

Digestion is carried out by increasing the microwave power to 800 W within 10 minutes and maintaining it at this level for 10 minutes. The maximum permissible control temperature is around 210 °C. After the sample has been digested, it is left to cool in the microwave device.

A pre-washed glass funnel is used to quantitatively transfer the entire digestion solution into a 25 ml volumetric flask using water with a low metal content. The flask is filled to the mark and shaken. The digestion solution is transferred into a wide-necked flask made of PE for storage. If precipitation occurs or there are non-soluble components, the sample solution is filtered with a disposable syringe fitted with a syringe filter. Part of the sample solution is transferred into an autosampler vial immediately before analysis.

If the cobalt concentration of the field blank value is greater than that of the batch blank value and the limit of quantification, then an additional batch blank (a non-loaded filter from the same batch in the laboratory) must be prepared and analysed.

The prepared samples, field blank and batch blank are injected into the AAS by means of the autosampler and analysed.

4.2 Operating conditions

AAS parameters: AAS graphite furnace with autosampler (Perkin Elmer PinAAcle 900Z with Zeemann background compensation and transverse heated graphite tube including a L'vov platform)
Cobalt hollow cathode lamp, warm up period is at least 15 minutes

Wavelength: 242.49 nm

Slit width: 0.2 nm

Injection: 20 µl of sample solution + 5 µl of modification solution + 5 µl of blank value solution

Tab. 1 Furnace program

Step	Temperature [°C]	Heating period [s]	Dwell time [sec]	Gas flow [ml/min]
Drying 1	120	1	30	250
Drying 2	140	20	40	250
Pyrolysis	1350	10	20	250
Atomisation	2350	0	3	0
Heating	2400	1	3	250

The prepared samples, the field blank, a reagent blank, consisting of nitric acid and hydrochloric acid at a ratio of 2:1, the blank value solution and if necessary a batch blank is analysed by means of AAS. For this purpose the autosampler injects 20 µl of the solution to be investigated, 5 µl of blank value solution and 5 µl of modification solution into the AAS and analysis is performed under the stipulated AAS operating conditions. Each sample is analysed in duplicate and the average value is taken to calculate the result.

If the cobalt concentrations are higher than the values in the calibration function, the sample solutions are diluted with water and nitric acid (65%), so that there is an acid level of 0.65% in the dilution for stabilisation purposes.

The blank value is checked regularly by measuring the blank value solution at least every eighth sample and the signal (area) is adjusted to zero.

The control solution is analysed before analysis of the sample solution and at the end of the sequence. It must fall within defined limits (e.g. ± 10%), otherwise the calibration must be checked and the analysis of the sample solution must be repeated. Thus, the stability of the analysis devices and the accuracy of the results is checked. In the case of larger series of samples it is advisable to carry out further measurements of the control solution.

A possible matrix influence must be checked at least once per sample series. For this purpose, at least one sample is analysed anew and 5 µl of blank value solution is replaced by 5 µl of calibration solution 2. The recovery rate is calculated from the cobalt concentration of the spiked sample after deduction of the cobalt concentration of the original sample based on the spiked cobalt concentration. The recovery rate must be between 90 and 110%,

otherwise it must be assumed that a matrix effect has influenced the result. It is important to ensure that the spiked sample is within the calibration function.

Example: A cobalt concentration of the sample solution of 19 µg/l, a cobalt concentration of the spiked sample of 30 µg/l and a spiked cobalt concentration of 10 µg/l results in a recovery rate of 103% (=30/(19+10)×100).

If the matrix of the sample has a significant influence, it is necessary to evaluate the sample solution using the standard addition method.

5 Evaluation

5.1 Calibration

External calibration

The calibration must be performed every working day. Calibration solution 2 with a concentration of 40 µg/l of cobalt is diluted by the autosampler to obtain the ten calibration standards as shown in the following schematic representation (see Table 2). Additionally, 5 µl of modification solution is also pipetted by the autosampler in the same manner as the samples. The standards are analysed under the AAS operating conditions described in Section 4.2 and a linear calibration function is obtained.

The control solution is analysed in order to check the calibration.

Tab. 2 Calibration standards external calibration

	Concentration [µg Co/l]	Blank value solution [µl]	Calibration solution 2 [µl]
Cobalt blank value	0	25	0
Calibration standard 1	4	23	2
Calibration standard 2	8	21	4
Calibration standard 3	12	19	6
Calibration standard 4	16	17	8
Calibration standard 5	20	15	10
Calibration standard 6	24	13	12
Calibration standard 7	28	11	14
Calibration standard 8	32	9	16
Calibration standard 9	36	7	18
Calibration standard 10	40	5	20

Standard addition calibration

If a matrix effect is detected, then a standard addition calibration must be performed for the sample. The cobalt concentration of the sample should be between 15 and 40 µg/l. The calibration standards are obtained by addition of different concentrations of calibration solution 2 to the sample (see Table 3). All the standards and solutions are analysed in duplicate.

Otherwise the AAS operating conditions remain unaltered.

The value at which the calibration function intercepts the x-axis is equivalent to the cobalt concentration of the sample solution.

Tab. 3 Standard addition calibration

	Concentration [µg Co/l]	Blank value solution [µl]	Sample solution [µl]	Calibration solution 2 [µl]
Cobalt blank value	0	25	0	0
Sample		15	10	0
Calibration standard 1	16	11	10	4
Calibration standard 2	28	8	10	7
Calibration standard 3	40	5	10	10

5.2 Calculation of the analytical result

The cobalt concentration c [µg/m³] in the sample air is obtained as follows:

$$c = \frac{((c_{LSG} \times F) - c_{FB}) \times V_{LSG}}{1000 \times \eta \times V}$$

where:

c is the mass concentration of cobalt in the air sample in µg/m³

c_{LSG} is the concentration of cobalt in the measurement solution in µg/l obtained from the calibration function

c_{FB} is the concentration of cobalt in the measurement solution of the field blank value in µg/l obtained from the calibration function

F is the dilution factor

V_{LSG} is the volume of the prepared sample solution in ml (=25)

V is the air sample volume in m³ (=1.2)

η is the recovery (=1)

6 Reliability of the method

The characteristics of the method were calculated as stipulated in DIN EN 13890 (DIN 2010), DIN EN 482 (DIN 2015) and DIN 32645 (DIN 2008).

6.1 Repeatability

The repeatability was determined by analysing one standard solution and one prepared sample on six different days. Calibration was carried out every working day for quantification. The cobalt concentration of the solutions was 24 µg/l. The repeatability of the standard solution was 1.4% and 2.6% for the sample.

6.2 Reproducibility and recovery

As a result of the different chemical compositions and the different physical properties of the aerosols in different work areas, it is impossible to provide generally valid data on the recovery rates for the entire procedure.

The recovery was determined by spiking 12 quartz fibre filters each with 60 µl of cobalt solution 1 (10 mg/l). Six filters were prepared and analysed as described in Sections 4.1 and 4.2. Laboratory air was drawn through the other six filters over a time period of 2 hours at a flow rate of 10 l/min. These samples were also subsequently prepared and analysed.

In order to obtain reference values, a pipette was used to add 60 µl of cobalt solution 1 (10 mg/l) each into six 25 ml volumetric flasks, into which water and 0.25 ml of 65% nitric acid had been previously placed. The volumetric flasks were filled to the mark with water, shaken and analysed according to Section 4.2.

The cobalt concentrations of the spiked filters were based on the reference values and on this basis the analytical recovery (transfer) and the recovery with air sampling were determined. The analytical recoveries as well as the recovery with sampling determined in this manner were not statistically significantly different from the reference values and are taken into account as $\eta=1$ for calculating the result.

The spiked amount of cobalt was equivalent to the concentration in the air sample listed in Table 4 for an air sample volume of 1.2 m^3. The reproducibility, expressed as relative standard deviation, was determined from the six spiked filters that underwent sampling and was approx. 0.8%.

Tab. 4 Characteristics of the validation

Spiked volume per filter	Spiked mass per filter	Concentration in the air sample	Transfer	Recovery	Relative standard deviation
[µl]	[µg]	[µg/m^3]			[%]
60	0.6	0.5	0.99	1.01	0.8

Recovery experiments with a spiked mass of 40 µl of the cobalt standard (1 g/l) and an air sample volume of 420 l resulted in a statistically significant recovery of 0.95 and a reproducibility of 3.3% (Table 5).

Tab. 5 Characteristics of the validation at higher concentrations

Spiked volume per filter	Spiked mass per filter	Concentration in the air sample	Transfer	Recovery	Relative standard deviation
[µl]	[µg]	[µg/m^3]			[%]
40	40	95	0.92	0.95	3.3

The recovery is defined as 100% on the basis of the sample preparation described above according to DIN EN 13890 (DIN 2010) (restricted to those metals and compounds that are soluble in the stated system) (AGS 2016; DIN 2010).

6.3 Limit of quantification

As no cobalt blank values were detectable on the filters, the absolute limit of quantification was determined using a calibration function in the lower concentration range as stipulated in DIN 32645 (DIN 2008). For this purpose, calibration solution 2 with a concentration of 40 µg/l was diluted by the autosampler in the same manner as the ten calibration standards as shown below (Table 6). Additionally, 5 µl of modification solution were also added by the autosampler in the same manner as the samples. The standards and the blank value were analysed according to the AAS operating conditions described in Section 4.2 and a linear calibration function was obtained.

Tab. 6 Calibration standards for external calibration for the determination of the limit of quantification

	Concentration [µg Co/l]	Blank value solution [µl]	Cobalt solution 2 [µl]
Cobalt blank value	0	25	0
Calibration standard 1	2	24	1
Calibration standard 2	4	23	2
Calibration standard 3	6	22	3
Calibration standard 4	8	21	4
Calibration standard 5	10	20	5
Calibration standard 6	12	19	6
Calibration standard 7	14	18	7
Calibration standard 8	16	17	8
Calibration standard 9	18	16	9
Calibration standard 10	20	15	10

The absolute limit of quantification is 2.8 µg/l or 56 pg of cobalt per injection for a statistical certainty of 95% and a relative uncertainty of the result of 33% or k = 3.

A relative limit of quantification of 0.058 µg/m³ of cobalt or 0.07 µg of cobalt per sample carrier is obtained for an air sample volume of 1.2 m³, a sample solution volume of 25 ml and an injection volume of 20 µl.

6.4 Storage stability

Cobalt deposited on a filter can be stored at room temperature without losses for at least 22 days.

6.5 Selectivity

High concentrations of other elements and compounds can lead to interferences. In the case of the principally unknown samples, the measurement results must be checked for possible interferences and if necessary a suitable dilution step must be chosen in order to achieve a valid result. Non-spectral interferences and matrix effects can be reduced by means of a suitable dilution.

To date, interferences by other components, in particular at high concentrations, were not observed. However, these cannot be ruled out. Possible matrix effects can be corrected by calibration with the sample solution in accordance with the standard addition method. It is not possible to differentiate between the individual cobalt compounds.

6.6 Uncertainty

The expanded uncertainty was obtained by estimation of all the relevant influencing parameters (bottom-up method). The uncertainty of the result consists principally of the uncertainty contributions of sampling u_S and the analytical preparation (preparation volume u_V, recovery and storage losses u_R, concentration-dependent influences of the measurement values u_{MV} attributable to the scatter of the calibration function, uncertainty of the calibration solution and the laboratory's own reproducibility).

The combination of all uncertainty contributions results in the concentration-dependent combined uncertainty U_{comb}. The corresponding expanded uncertainty that represents the concentration-dependent uncertainty of the entire procedure is obtained by multiplication with a probability factor (e.g. k = 2 for 95% confidence level).

The estimation of the sampling-dependent components of the uncertainty is based on the following factors according to Annex B of DIN EN 13890 (DIN 2010) for sampling respirable dusts:

Calibration of the test system:	1%
Estimation of the aggregated concentration:	1%
Systematic deviation from the sampling convention:	8%
Deviation from the nominal flow of the cyclone:	3%
Individual variability of the sampling device:	7%

This results in a combined uncertainty for the sampling system of 11.1%.

A total uncertainty for sampling respirable dusts u_S of 11.7% is obtained taking a sampling period of 120 minutes into account.

The individual uncertainty contributions and the resulting combined uncertainties are summarised in Table 7.

Tab. 7 Uncertainty contributions in %

u_S	u_V	u_R	u_{MV} [a)]				u_{comb}			
			C_1	C_2	C_3	C_4	C_1	C_2	C_3	C_4
11.7	0.1	1.2	16.2	6.9	4.5	3.9	20.0	13.7	12.6	12.4

[a)] The concentrations C_1 to C_4 are shown in Table 8.

Table 8 lists the concentration-dependent expanded uncertainties U_{exp} with the corresponding cobalt concentrations of the sample solution and the relevant cobalt concentrations in the air at an air sample volume of 1.2 m³. Only the uncertainties for the calibration range were taken into account, as at higher cobalt concentrations in the air the samples in the calibrated range are diluted.

Tab. 8 Expanded concentration-dependent uncertainties

	C_1	C_2	C_3	C_4
Expanded uncertainty [%]	40	27	25	25
Cobalt concentration in the sample solution [µg/l]	4.8	12	24	40
Cobalt concentration in the air [µg/m³]	0.1	0.25	0.5	0.83

6.7 Comparison with conventional digestion

In the context of a round-robin experiment in 2017 (Pitzke et al. 2018) – organised by the IFA and carried out by members of the "Air Analyses" working group of the DFG Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area – the high-pressure microwave digestion described here was compared to open conventional digestion. For this purpose, two ground dusts were prepared and analysed. The cobalt concentrations of the dusts for open conventional digestion were 3.7% and 0.015% on average with relative standard deviations of 4.9% and 22.1% for 5 to 6 participating laboratories. Concentrations of 3.7% and 0.016% were determined with the analytical method described here. Comparability between the high-pressure microwave digestion described here and the open conventional digestion is thus given.

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