Selenium – Method for the determination of selenium and its inorganic compounds in workplace air using atomic absorption spectroscopy (AAS)

Air Monitoring Method – Translation of the German version from 2019

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Abstract

This analytical method is a validated measurement procedure for the determination of selenium [7782-49-2] and its inorganic compounds in workplace air in a concentration range of one tenth up to twice the currently valid OEL of $0.05 \, \text{mg/m}^3$ in the inhalable fraction (I). Sampling is performed by drawing a defined volume of air through a membrane filter using a suitable pump. The flow rate is set to $1.4 \, \text{l/min}$ and sampling is performed over 2 h. The dust deposited on the membrane filter is treated with acid and then analysed using atomic absorption spectrometry with the graphite furnace technique (GFAAS). The quantitative determination is based on a calibration function, whereat the selenium concentration of the reference standard is plotted against the peak area calculated with an integration program. The limit of quantification is $0.0004 \, \text{mg/m}^3$ based on an air sample volume of 168 l. The mean recovery was 113.9% and the expanded uncertainty for a validation range of $0.01 \, \text{mg/m}^3$ to $1.0 \, \text{mg/m}^3$ was between 22% and 27%.

Method number	1
Application	Air analysis
Analytical principle	Atomic absorption spectroscopy (AAS)

1   Characteristics of the method

Precision:	Standard deviation (rel.):	s = 1.4 to 4.7%
	Expanded uncertainty:	U = 22 to 27%
	in the concentration range from 0.01 to 1.0 mg/m³ and for n = 6 determinations	
Limit of quantification:	0.0004 mg/m³ for an air sample volume of 168 l and a sampling period of 2 h	
Recovery:	η = 1.14 (113.9%)	
Sampling recommendations:	Sampling period:	2 h
	Air sample volume:	168 l
	Flow rate:	1.4 l/min

2   Description of the substances

Selenium [7782-49-2]:
Selenium is a grey, shiny metalloid element with the symbol Se (atomic mass 78.96 u, melting point 221 ℃, boiling point 685 ℃, density 4.819 g/cm³). Selenium is industrially produced by roasting of anode mud, a by-product of the electrolytic refining of copper and nickel.

Due to its versatile properties selenium is used in many branches of industry. Selenium is used in the electronics industry in rectifiers, photocells, light-sensitive alarm systems as well as in xerography on account of its electrical conductivity and its semi-conducting properties. In addition, in the glass and ceramics industry it is used as a pigment and decolourising agent and in the chemical industry for vulcanisation and polymerisation in the manufacture of rubber. Selenides are used as catalysts for mineral oil processing, hydrogenation of coal and as lubricants. Furthermore, they are employed in pharmaceutics, the food industry as well as in agriculture.

Selenium has an Occupational Exposure Limit (OEL) of 0.05 mg/m³ for the inhalable fraction (I), the short-term exposure limit is assigned to Peak Limitation Category II with an excursion factor of 1 (AGS 2018). In the List of MAK and BAT values, the MAK value for selenium is 0.02 mg/m³ I with an excursion factor of 8 (DFG 2018). Detailed information on the toxicity of selenium and its inorganic compounds can be found in the toxicological-occupational health documentation of MAK values (Hartwig 2014).

3   General principles

The analytical procedure permits the simultaneous determination of selenium dusts and selenium compounds that occur in particulate form in workplace air in a concentration range of 0.1 times up to twice the currently valid Occupational Exposure Limit (OEL) of 0.05 mg/m³ I (AGS 2018; DIN 2015).

A suitable pump draws a defined volume of air through a membrane filter (mixed cellulose ester) for the sampling procedure. After acid digestion of the selenium deposited on the filter in the form of dust and particles, it is deter-

mined using a graphite furnace atomic absorption spectrometer (GF-AAS). The quantitative evaluation is based on a calibration function, whereby the concentrations of selenium in the calibration standards are plotted versus the peak areas obtained by means of an integration program.

4 Equipment, chemicals and solutions

4.1 Equipment

For sampling:

- Sampling pump, suitable for a flow rate of 1.4 l/min, (e.g. SG 4000 or SG 5200, from GSA, Gesellschaft für Schadstoffanalytik, 40880 Ratingen, Germany)

- Filter cartridge for membrane filters (mixed cellulose ester), ID 37 mm, (e.g. Supelco No. 23370-U)

- Membrane filter (mixed cellulose ester), ID 37 mm, pore size 0.8 μm (e.g. P/N 64678, from PALL Filtersysteme, 55543 Bad Kreuznach, Germany)

For sample preparation and analysis:

- VAO wet ashing device (from Hans Kürner Analysentechnik, 83004 Rosenheim, Germany)

- Digestion vessels made of quartz glass

- Atomic absorption spectrometer with graphite furnace and autosampler (e.g. PerkinElmer AAnalyst 600, from PerkinElmer LAS, 63110 Rodgau, Germany)

- Volumetric flasks made of glass, 10 ml and 100 ml for the standard and calibration solutions

- Volumetric flasks made of glass, 25, 50, 100 and 1000 ml for the acid solutions

- Measuring cylinder made of glass, graduated up to 250 ml

- Selenium EDL lamp

- 50 ml sample tube (114 × 28 mm) with screw caps made of polypropylene (PP), graduated, (e.g. from Sarstedt, 51582 Nümbrecht, Germany, Order No. 62.548.004)

- Two "reference" piston pipettes, variable, 10–100 μl, 100–1000 μl (e.g. from Eppendorf, 22339 Hamburg, Germany)

- "Research" piston pipette, variable, 500–5000 μl (e.g. from Eppendorf)

- Gas meter or stopwatch and bubble flowmeter

4.2 Chemicals

- Selenium ICP standard 1000 mg/l, traceable to SRM from NIST, SeO_2 in HNO_3 2–3%, from Merck, Darmstadt, Germany, 100 ml, Order No. 1.70350.01000

- Ultrapure selenium (99.999%), from Retorte, 90552 Röthenbach a.d. Pegnitz, Germany

- Nitric acid (65%), Suprapur®, e.g. from Merck, Darmstadt, Germany, Order No. 1.00441.1000

- Hydrochloric acid (30%), Suprapur®, e.g. from Merck, Darmstadt, Germany, Order No. 1.00318.1000

- Hydrochloric acid (25%), EMSURE®, e.g. from Merck, Darmstadt, Germany, Order No. 1.00316.1000

- Ascorbic acid: L(+)-Ascorbic acid, NORMAPUR®, from VWR International, 85737 Ismaning, Germany, Order No. 20150.184

- Palladium(II) nitrate dihydrate (40% Pd), for synthesis, e.g. from Merck, Darmstadt, Germany, Order No. 8.14573.0001

- Magnesium nitrate hexahydrate $Mg(NO_3)_2 \times 6\ H_2O$, Suprapur®, from Merck, Darmstadt, Germany, Order No. 1.05855.0050

- Bidistilled water (Ultrapure water unit, e.g. from Millipore, 65760 Schwalbach am Taunus, Germany)

- Argon 4.6 (purity 99.996%)

4.3   Solutions

The following solutions are prepared using the chemicals listed in Section 4.2:

Acidic digestion mixture: Two parts by volume of nitric acid (65%) (HNO_3) and one part by volume of hydrochloric acid (25%) (HCl)

Two parts by volume of nitric acid (65%) and one part by volume of hydrochloric acid (25%) are carefully mixed in a 200 ml glass beaker.

Nitric acid (2 molar):
Approx. 500 ml of bidistilled water are placed into a 1000 ml measuring cylinder. Then 138.9 ml of 65% nitric acid are added. After the solution has cooled to 20 ℃, the measuring cylinder is filled up to 1000 ml with bidistilled water.

Hydrochloric acid (2 molar):
Approx. 300 ml of bidistilled water are placed into a 1000 ml measuring cylinder. Then 210.1 ml of 30% hydrochloric acid are added. After the solution has cooled to 20 ℃, the measuring cylinder is filled up to 1000 ml with bidistilled water.

Acid mixture for preparation of the calibration solutions:
25 ml of the 2 molar nitric acid and 25 ml of the 2 molar hydrochloric acid are added to a 100 ml volumetric flask into which approx. 30 ml of bidistilled water have been previously placed. After the solution has cooled to 20 ℃, the volumetric flask is filled up to the mark with bidistilled water.

Ascorbic acid solution: (50 g/l)
5 g of ascorbic acid are weighed into a 100 ml volumetric flask; the flask is then filled up to the mark with bidistilled water and shaken.

Modifier stock solution: (3.1% Pd(II) nitrate dihydrate and 1.3% Mg nitrate hexahydrate)

31 mg of Pd(II) nitrate dihydrate and 13 mg of Mg nitrate hexahydrate are weighed into a 50 ml glass beaker and dissolved in 1.0 ml of bidistilled water by shaking repeatedly.

Modifier ready-to-use solution: Modifier stock solution (3%)

30 µl of modifier stock solution and 970 µl of bidistilled water (ratio 3 : 100) are mixed in plastic laboratory tubes with 3 ml volume (e.g. Sarstedt tubes, 3.0 ml, Order No. 60.549/1000 pcs).

The ready-to-use modifier solution must be freshly prepared every working day!

4.4 Calibration standards

Calibration standards are prepared by dilution of the selenium ICP standard (1000 mg/l) as follows:

Selenium stock solution: Dilution 1:100 ≙ 10 mg/l

1.0 ml of the selenium ICP standard is pipetted into a 100 ml volumetric flask into which approx. 50 ml of the acid mixture have been previously placed. The volumetric flask is then filled up to the mark with acid mixture and shaken.

Calibration standards

Calibration standards with selenium concentrations of 50, 100, 150, 200, 250 and 300 µg/l are prepared from the selenium stock solution (10 mg/l) in 10 ml volumetric flasks. The volumetric flasks are subsequently filled up to the mark with the acid mixture. The dosing scheme for the preparation of the calibration solutions is shown in Table 1.

Tab. 1   Concentrations of selenium in the calibration solutions

Calibration solution [µg selenium/l]	Volume of stock solution [µl]	Total volume [ml]	Spiked mass [µg selenium/filter]
50	50	10	0.5
100	100	10	1.0
150	150	10	1.5
200	200	10	2.0
250	250	10	2.5
300	300	10	3.0

5 Sampling and sample preparation

5.1 Sampling

Sampling can be carried out as stationary or personal sampling. Samples are collected in the breathing zone in the case of personal sampling. It is important to ensure that the inlet aperture of the filter cartridge is freely accessible.

The filter cartridge is opened and connected with the pump. The pump is used to continuously draw ambient air through the filter over a time period of 2 hours at a flow rate of 1.4 l/min. This is equivalent to an air sample volume of 168 l (0.168 m³) at a sampling period of 2 hours. The important parameters for the determination of the concentration in air (sample volume, temperature, air pressure and relative humidity) are documented in the sampling record.

After sampling, the flow rate must be tested for constancy. If the deviation from the adjusted flow rate is greater than ±5%, it is advisable to repeat the measurement (DIN 2014). The filter cassette with the loaded filter is sealed

with the designated caps and transported to the laboratory for analysis with as little vibration as possible. The air samples must be stored in the refrigerator at 4 to 6 ℃ until analysis.

5.2   Sample preparation

The loaded filter is carefully removed from the filter cassette using a tweezer and transferred to a digestion vessel made of quartz glass. At the same time at least two unused filters from the same batch are also prepared as blank filter samples in another digestion vessel. 6.0 ml of the acidic digestion mixture (see Section 4.3) are added to each of the filters. Then the digestion vessels are furnished with an air cooler and a glass rod, placed in a thermostatically controlled heating block and kept at boiling temperature over a time period of 2 hours.

After digestion vessels have cooled, the sample solutions are transferred into graduated and appropriately labelled tubes with screw caps and filled up to 10 ml with bidistilled water. The sample solutions are then analysed with AAS.

An initial test injection with a dilution at a ratio of approx. 1:50 of each sample is recommended to be able to correctly estimate the dilution required to protect the graphite tubes/furnace against contamination.

6   AAS operating conditions

Apparatus:	Atomic absorption spectrometer A600 with graphite furnace (graphite tube with platform as well as background correction by Zeeman effect) and autosampler from PerkinElmer
Measured wavelength:	196.0 nm
Spectral slit width:	2.0
Lamp power rating:	According to the manufacturer's instructions
Signal evaluation mode:	Peak area
Inert gas:	Argon (4.6)
Injection volume:	20 µl
Injection volume of the modifier ready-to-use solution:	20 µl

The temperature/time program of the graphite tube is shown in Table 2.

Tab. 2   Temperature/time program

Program step	Furnace temperature [℃]	Temperature ramp time [s]	Dwell time [s]	Inert gas flow rate [ml/min]
1	110	5	25	250
2	130	30	30	250
3	1300	20	50	250
4	2100	0	5	0
5	2450	1	3	250

7   Analytical determination

Before analysis with AAS, 100 µl of ascorbic acid solution are added to each of the samples prepared according to Section 5.2. In order to obtain the calibration graph the samples are prepared as follows:

Reagent blank value:	100 µl of ascorbic acid solution + 900 µl of the acid mixture
Calibration solutions:	100 µl of ascorbic acid solution + 900 µl of the calibration solution (100/200/300 µg/l)
Filter samples:	100 µl of ascorbic acid solution + 900 µl of the acid digestion mixture

The addition of the ready-to-use modifier is controlled by the autosampler program. Initially the modifier is pipetted into the graphite tube by means of the "Perkin Elmer AS 800" autosampler used. There it is dried during the first two steps of the temperature program (see Section 6 and Table 2).

After the furnace has cooled to approx. 20 ℃, a pipette is used to add the sample, then it undergoes the complete temperature program. If the samples exceed the selenium concentration of 300 µg/l, they must be diluted accordingly with acid mixture and measured again.

Note: *The samples must always be freshly prepared and analysed immediately. If analysis cannot be carried out at once, then reduced values are to be anticipated!*

8   Calibration

Loading of the calibration samples should occur in the lowest measurement range (one tenth to twice the OEL) of selenium (DIN 2015).

The calibration standards of selenium in the concentration range of 50 to 300 µg/l (see also Table 1) prepared according to Section 4.4, are measured in order to obtain the calibration function. Any reagent blank values are subtracted from the absorbances obtained for the selenium standards and the absorbances are plotted versus the concentrations. The calibration graph is automatically generated by the data evaluation unit of the AAS device by means of linear regression.

The calibration graph is linear under the stated conditions and should be checked regularly during routine analysis. Calibrations previously carried out must be performed anew if the analytical conditions change or the quality control results indicate that this is necessary. Figure 1 shows the calibration graph of selenium.
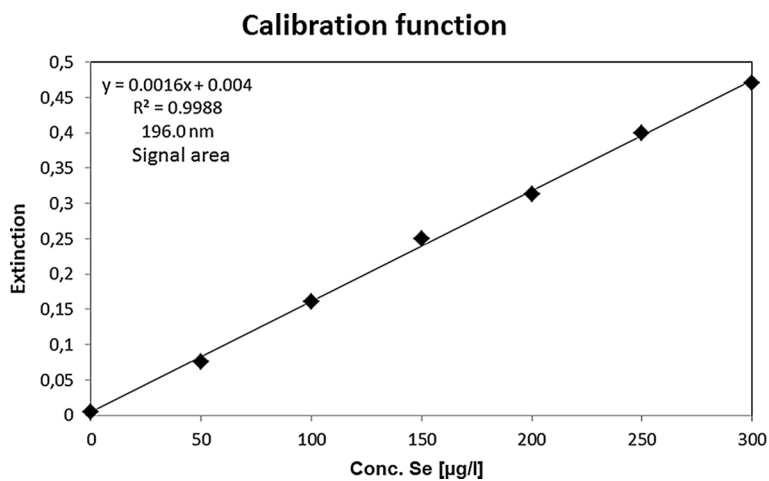
Calibration function

Fig. 1 Calibration function of selenium in the lowest measurement range

9   Calculation of the analytical result

The data evaluation unit calculates the selenium concentration in the workplace air from the concentration of selenium in the measured solution using the calibration function obtained by means of linear regression for this purpose. The concentration of selenium in the workplace air is calculated from the selenium concentrations, taking the corresponding dilutions and the air sample volume into account. The mass concentration (ρ) of selenium is calculated according to Equation (1) as follows:

$$\rho = \frac{(C - C_{\text{Blank}}) \times 0.001 \times f_v \times V}{V_{\text{air}}} \tag{1}$$

where:

ρ	is the mass concentration of selenium in the air sample in mg/m^3
C	is the concentration of selenium in the measurement solution in µg/l
C_{Blank}	is the concentration of the blank value (mean value) in µg/l
0.001	is the conversion factor [µg $\rightarrow$ mg]
f_v	is the dilution factor
V	is the volume of the sample solution in l
V_{air}	is the air sample volume in m^3

10 Reliability of the method

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

10.1 Precision and expanded uncertainty

In order to determine the precision and expanded uncertainty, six membrane filters were each spiked with different masses of selenium (2, 20 and 200 µg selenium/filter) and then subjected to the complete analytical method (see Sections 5.2, 6 and 7).

For this purpose, six filters were each spiked with 200 µl of the selenium ICP standard solution (1000 mg/l) (equivalent to a Se content of 200 µg) and six filters with 20 µl each (equivalent to 20 µg of selenium). The lowest spiked mass was prepared by diluting the selenium ICP standard solution with 3% nitric acid at a ratio of 1:5, this results in a concentration of 200 mg/l. 10 µl each of this solution is applied to six further filters (equivalent to 2 µg of selenium). The spiked filters were prepared and analysed in the same manner as the sample solutions. For an air sample volume of 168 l these spiked masses are equivalent to selenium concentrations in air of 10 µg/m³, 100 µg/m³ and 1000 µg/m³.

Before analysis by AAS, the extraction solutions with spiked masses of 20 µg and 200 µg were diluted with the acid mixture by a factor of 10 and 100, respectively. Furthermore, two unused filters (blank filters) as well as a sample without filter (reagent blank value) were subjected to the complete analytical method. The precision data shown in Table 3 were calculated from these results.

Tab. 3 Standard deviation (rel.) and expanded uncertainty *U* for *n* = 6 determinations

Mean value of the spiked mass of selenium	Standard deviation (rel.)	Expanded uncertainty *U*
[µg]	[%]	[%]
2.48	1.4	27
21.0	3.2	23
225	4.7	22

The expanded uncertainty is obtained by estimation of all the relevant influencing parameters. The uncertainty of the result is based on two important steps, the uncertainty components for sampling and for analysis.

In order to estimate the uncertainty components of sampling, the uncertainty associated with the air sample volume and the sampling effectiveness for inhalable dusts were determined according to Appendix B in DIN EN 13890 (DIN 2010). The combination of all random and non-random uncertainty components results in an uncertainty of 9.3% when sampling inhalable dusts for two hours.

The uncertainty components of the analysis encompass the entire analytical preparation including digestion, dilution, calibration, recovery and precision. The concentration-dependent uncertainties of the analysis for this method for the mean value of the spiked masses of selenium are 2.48 µg at 9.6%, 21 µg at 6.3% and 225 µg at 6.2%.

The combination of all uncertainty contributions results in the concentration-dependent combined uncertainties of the entire method. The values for the expanded uncertainty of the entire method listed in Table 3 are obtained by multiplying with the expansion factor k = 2.

10.2 Recovery

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).

The recovery was calculated from the measurement results for the determination of the precision (Section 10.1). The recoveries shown in Table 4 were obtained, whereby the mean recovery was 113.9%.

Tab. 4   Recovery of selenium for n = 6 determinations

Spiked mass of selenium	Mean value and standard deviation for the spiked mass of selenium	Recovery
[µg/filter]	[µg]	[%]
2	2.48 ± 0.03	124.1
20	21.0 ± 0.7	105.2
200	225 ± 11	112.5

In order to realistically determine the recovery, 200 µg of ultrapure selenium in powder form (99.999%) were each weighed onto 13 filters. The spiked filters were prepared and analysed in the same manner as the sample solutions. Furthermore, one unused filter (blank filter) as well as a sample without filter (reagent blank value) were subjected to the complete analytical procedure. The measurement results were compared to the weighed amounts. A recovery of 95.8 ± 25.8% was determined in this case.

10.3   Limit of quantification

During development of the method the limit of quantification was determined based on DIN 32645 (DIN 2008) according to the calibration line method. For this purpose, calibration solutions with selenium concentrations of 0.5; 5; 7.5; 10; 12.5; 15; 17.5; 20; 22.5 and 25 µg/l were subjected to the complete analytical method together with one unloaded filter in each case in a triplicate determination. The response values were determined according to the standard method for the calculation of the detection limit. A k value of 3 was used for the calculation of the limit of quantification.

On the basis of this procedure a detection limit of 2.22 µg/l of digestion solution and a limit of quantification of 6.55 µg/l of digestion solution were determined. The limit of quantification was 0.0004 mg of selenium/m^3 based on a sample volume of 168 l.

10.4   Storage stability

The storage period of the loaded filters at room temperature should not exceed one month.

10.5   Selectivity

The selectivity of the method depends largely on the selection of the wavelength and thus on spectral interference. Spectral interference is mostly caused by emission lines from interferents and molecules in the sample matrix.

11   Discussion

Selenium and inorganic selenium compounds can be determined in workplace air in a concentration range of a tenth up to twice the currently valid OEL value of 0.05 mg/m^3 I and MAK value of 0.02 mg/m^3 I using the analytical method described here. The analytical method is suitable for checking compliance with the short-term value.

Sampling can not only be carried out using the filter cartridge described here, but also with a GSP sampling head (DGUV 2016). Principally all working conditions, in particular the sample dosing and the temperature program, must be adapted to the respective AAS device used. The examiner of the method described here was able to achieve stable analytical conditions by using a modifier containing nickel, despite oxidative conditions (without ascorbic

acid). If applicable, this may lead to a risk of contamination should the same AAS device also be used for nickel analysis. If such contamination can be ruled out, then the use of the nickel modifier is preferable to the use of ascorbic acid. This applies in particular to cases where the measurement solution contains large amounts of halides.

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