

Determination of metal-containing components of airborne particles

Air Monitoring Method – Translation of the German version from 2019

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Abstract

In addition to the gravimetric determination of airborne particles (total concentration), it is often necessary to selectively determine metals and their compounds in particle fractions because of their toxicological relevance. Usually, the total metal concentration is determined independently of the type of binding or oxidation state in a sample. From an occupational medical and toxicological point of view it makes sense to distinguish between different compounds of a metal, because type and extent of the toxic effect of metals depend considerably on their binding type and their solubility in the human body. In addition to the limit values of the respirable and inhalable particle fraction that must be complied with, many metals have an OEL (occupational exposure limit) or MAK value that has to be checked and complied with, too. For carcinogenic compounds the exposure-risk relationship has to be considered. Analysis for metals and their compounds predominantly resorts to methods which

require that the dust particle sample is brought into solution. That means the metals and their compounds contained in the sample need to be extracted, dissolved or digested. Aim of the sample preparation is the complete solution of all relevant substances to be analysed. Common digestion methods are for example acid digestion, which uses an acid mixture to digest the sample, and the suspension method, in which acetone is used to suspend the sample. An alternative sample preparation method is the microwave-assisted pressure digestion with acid/acid mixture. In this chapter the different digestion methods are presented, discussed and compared, taking into account recent developments, in particular microwave-assisted digestion.

1 Introduction

In addition to the gravimetric metal determination (total concentration) in a dust sample collected on a sample carrier described in this method collection (Hebisch et al. 2005), selective determination of occupationally relevant metals and their compounds in dusts is often necessary. Many metals have a limit value (Occupational Exposure Limit or MAK value) and tests must be carried out to ensure compliance with the limit values as well as those limits for the respirable and the inhalable dust fractions. Several metals are classified as carcinogenic and have in part very low evaluation levels that were derived as acceptance and tolerance concentrations according to the concept of an Exposure-Risk-Relationship (AGS 2020 b). Furthermore, it may be necessary to collect the metals to be determined in the respirable as well as the inhalable dust fraction, as an evaluation in both fractions may be required due to the binding limit values.

Metals and metal compounds relevant to workplaces are listed in Table 8 in the Appendix. The currently valid evaluation criteria for metals and metal compounds can be found in TRGS 900 “Occupational Exposure Limits” (AGS 2020 a) and TRGS 910 “Risk-related concept of measures for activities involving carcinogenic hazardous substances” (AGS 2020 b). In addition, international limit values exist, that can be found in the “GESTIS – International limit values for chemical agents” database (IFA 2020).

Generally, the total metal content (total concentration) in a dust sample is determined independently of the type of bond or oxidation state. From the point of view of occupational medicine and toxicology it makes sense to differentiate between the various compounds of an individual metal, as the type and extent of the toxic effect of metals depends to a large extent on their bonding state and their solubility in the body. Furthermore, differences in effect of the same compound may be dependent on the type of exposure. In most cases, this requirement for differentiation cannot be fulfilled by means of analysis or can be achieved only by using considerable resources (labour and cost).

In order to determine metals and/or their compounds, on the one hand the collection media must permit the determination of the mass of the collected dust fraction by means of gravimetric analysis, on the other it must also allow the quantitative conversion of the metals contained in the dust to a form that can be analysed. In practice the collected quantity of dust may not be sufficient for the subsequent gravimetric determination of the respirable or the inhalable dust fraction. However, the considerably more sensitive analytical method still enables determination of the individual metal content. Therefore, when sampling is still at the planning stage, the subsequent preparation of the analytical samples should be taken into consideration.

As a rule, methods for analysing metals and/or metal compounds require that the dust samples are brought into solution, that means metals and/or metal compounds contained in the dust must be extracted, dissolved or digested. The aim of sample preparation is to achieve complete dissolution of all the substances to be analysed. However, it is difficult to consistently implement this requirement since the composition of the dust samples is usually largely unknown. On-site research provides information on metals and metal compounds that may be expected in higher concentrations in the dust. Generally, there is insufficient information on concomitant substances that often occur in higher concentrations than the substances to be determined (analytes).

The largely standardised digestion procedures described below have proved successful in practice, whereby recent developments – in particular digestion using microwave technology – are also taken into consideration. The digestion conditions for most metals and metal compounds were selected so that maximum solubility can be assumed with regard to their “dissolution behaviour” in the human body. It should be noted that it is not always necessary to dissolve the total amount of the material to be investigated. Therefore, the procedure described here represents a convention. It is important to ensure that the methods described here provide comparable measurement results that allow a reliable evaluation of exposure to metals at the workplace.

Alternatively, there are specific measurement methods for the determination of individual metals and metal compounds that do not necessarily require digestion (e.g. “Method for the determination of vapour from mercury and its inorganic compounds in workplace air using atomic absorption spectrometry” (Hebisch et al. 2018)).

2 Requirements for analytical methods

2.1 General requirements

The digestion and extraction methods used should fulfil certain requirements that will be described in more detail in the following sections. Thus, the analytes to be determined must be completely dissolved, whereby composite methods and multi-step digestion should be avoided. Composite methods or multi-step digestions (cascade digestions) with many individual steps increase random errors as well as heighten the risk of systematic errors and contamination. Furthermore, it is important to ensure that all process steps are carried out in the same reaction vessel if possible.

Equipment and chemicals used should be of the highest possible quality in order to minimise contamination. Losses due to the formation of volatile compounds should be prevented by adopting suitable measures (e.g. use of a reflux condenser, microwave digestion). Moreover, concentrations of other salts in the digestion solution should be as low as possible in order to avoid interference during measurement (matrix effects).

2.2 Specific requirements and influencing factors

The requirements and influencing parameters that are of great significance in the evaluation of measurement results are described in the following sections. This includes collection media suitable for sampling, the particle size, the composition of the sample material to be investigated as well as its components if necessary. The digestion or extraction agent used, the temperature as well as the duration of sample digestion or extraction play an important role in sample preparation.

2.2.1 Collection media

Metal-containing dusts are generally collected on filters. As some metals have been assigned very low limit values, it is advisable to use filters that exhibit no blank values or blank values that are as low as possible and constant. Experience has shown that blank value concentrations are batch-dependent, therefore a test certificate giving information on relevant components of the filters should be available (e.g. from the manufacturer or supplier). The test certificate should contain information on the level of the metal content and their bandwidth. Only filters from a single batch should be used in the course of a measurement series.

The filter size has a significant influence on the performance of the digestion. This applies in particular to the volume of the digestion medium and the size of the digestion vessel used. Filters should not be divided, as the homogeneity of the dust deposition cannot be generally assured. The commonly used collection media and their areas of application are presented below (Breuer 2012).

2.2.1.1 Cellulose nitrate/Cellulose acetate membrane filters

Cellulose nitrate/cellulose acetate membrane filters have a sponge-like but pressure-resistant structure. They act as multi-layered, fine-meshed sieves that enable surface deposition. Due to their good retention capacity, including for small particle sizes, they are well suited for dust sampling.

Membrane filters are very suitable for subsequent analysis of metals in dust, as they exhibit very low blank values and can be readily digested by acids.

Nonetheless, blank values cannot be disregarded, even for membrane filters. Blank values can be traced back to the use of natural, already contaminated cellulose in the manufacture of filter materials. In the case of very sensitive analytical methods they may cause interference in the detection of low concentrations. A regular check for blank values is necessary.

2.2.1.2 Quartz glass fibre filters

Quartz glass fibre filters used as depth filters are non-woven liners made of quartz glass. They are suitable for dust sampling due to their good retention capacity.

As a result of their exceedingly low and relatively constant blank values, they are substantially more suitable than glass fibre filters for the screening of dusts for metallic components. However, soluble siliceous components can also interfere with the analysis when using sensitive analysis techniques.

2.2.1.3 Glass fibre filters

Glass fibre filters used as depth filters are non-woven liners made of borosilicate glass. They are suitable for dust sampling due to their good retention capacity.

The high and often fluctuating blank values of this filter material can adversely affect the analysis of metals in dust. These filters are not suitable or only partially suitable for the determination of zinc and iron, as the strongly fluctuating blank values lead to high limits of quantification. Problems also arise in the determination of aluminium, calcium and barium. Similarly, the glass fibre filters made of borosilicate and free from binding agents with reduced blank values, which are now available, are less suitable for sensitive analytical methods (ET-AAS, ICP-MS).

2.2.1.4 Fluoropolymer filters

Fluoropolymer filters are membrane filters made of fluoropolymers (e.g. PTFE). They exhibit high chemical and thermal durability and are not dissolved by common digestion agents.

Their blank value concentrations are low, however, this filter material must be tested before use, as it may contain small amounts of metals. Furthermore, the high flow resistance of this filter material must be taken into consideration.

2.2.1.5 Other filter materials

Additionally, other filter materials can also be used such as polycarbonate or polyvinyl chloride (PVC) filters. Polycarbonate filters, in particular, have a very good resistance to chemicals.

2.2.1.6 Polyurethane foams

Polyurethane foams (PU foams) can be manufactured with various pore sizes and are suitable for dividing collections of particles into fractions (BIA 2002; Möhlmann et al. 2003). Due to the manufacturing process, PU foams may contain many impurities from auxiliary components (e.g. organic tin compounds), pre-cleaning of the material is crucial to minimise blank values. The limited solubility of the material during digestion can also lead to interference.

2.2.2 Particle size

As a general rule, the particle size range is unambiguously set by the limit values (inhalable, respirable fraction) and the sampling method is to be applied according to this requirement.

2.2.3 Components in the collected dust

Generally, the composition of dust samples is unknown. Depending on the origin of the sample, concomitant substances containing, e.g. silicate or organic concomitant substances, can interfere with digestion and the subsequent analysis. Furthermore, some elements can form poorly soluble compounds, such as AgCl, SnO₂ or tungstates during sample preparation. Other elements – such as selenium – are volatile or form volatile compounds during digestion, so that evaporation of the solvent must be prevented during sample preparation.

2.2.4 Digestion or extraction agents

When selecting digestion or extraction agents, it must be ensured that these do not form poorly soluble compounds with the dust components to be analysed. HNO₃, HCl, HF, H₂SO₄, HClO₄ or mixtures of these are commonly used acids. The digestion or extraction agents used should be ultrapure (with a low metal content) and each batch must be certified by the manufacturer.

2.2.5 Temperature and treatment period

At present two digestion techniques are widely used.

- Open digestion methods with aqueous acids/acid mixtures. In some cases, these digestions are carried out under reflux.
- High-pressure microwave digestion with aqueous acids/acid mixtures that are heated under pressure.

It should be noted that temperatures are lower during open digestion than in high-pressure microwave digestion and therefore a longer treatment period is necessary. The digestion temperature is critical to ensure that digestion is as complete as possible.

3 Equipment, analysis techniques and chemicals

3.1 Equipment and aids for digestion

Equipment and aids for the digestion of metals and metal compounds in dust samples are listed below. Inert materials should be used for digestion, which are as free of metals as possible; if necessary, they should be suitably cleaned before use.

3.1.1 Open digestion

- Heating block thermostat made of metal or graphite with time/temperature control
- The digestion vessels are preferably made of quartz glass (see Table 9) or of comparable quality in accordance with the requirements of DIN 12353 (DIN 1981) for reaction vessels.
- Air cooler preferably made of quartz glass with standard inner and outer ground glass joints for mounting on a digestion vessel or of comparable quality in accordance with the requirements of DIN 12353 (DIN 1981) and DIN 12242 (DIN 1980).
- Boiling rods preferably made of quartz glass with replaceable endpieces (e.g. PTFE tube)

3.1.2 High-pressure microwave digestion

- High-pressure microwave digestion system
- Suitable digestion vessels for the microwave, for instance made of quartz glass or PTFE

3.1.3 Suspension method

- Test tube shaker, variable speed setting up to approx. 1500 rpm
- Suitable digestion vessels, e.g. made of quartz glass

3.2 Analytical techniques

Spectrometric analytical procedures (Table 1) are employed for the analysis of metals and/or their compounds. Electrochemical analysis methods do not fall within the scope of this collection of methods.

Tab. 1 Frequently used analytical techniques for the determination of metals in measurements at the workplace

Measurement principle	Analytical technique
Atomic absorption spectrometry (AAS)	Flame (F-AAS)
	Graphite furnace technique (GF-AAS or ET-AAS ^{a)})
	Hydride technique
	Cold vapour technique
ICP techniques	Atomic emission spectrometry (AES), inductively coupled plasma with optical emission spectrometry (ICP-OES)
	Mass spectrometry (MS), inductively coupled plasma with mass spectrometric detection (ICP-MS)
Atomic fluorescence spectrometry (AFS)	
X-ray fluorescence analysis (XRF)	Energy dispersive
	Wavelength dispersive
	Total reflection

^{a)} ET-AAS: electrothermal atomic absorption spectrometry

The spectrometric methods include atomic absorption spectrometry (AAS) that is used for the quantitative determination of individual metals and metalloids. The techniques are differentiated into flame, graphite furnace, hydride and cold vapour techniques according to the type of atomisation source. Atomic spectrometric methods, such as inductive coupled plasma (ICP), as ICP-OES (optical emission spectrometry) or ICP-MS (mass spectrometry), are increasingly being used today. The ICP technique is a multiple-element procedure. Normally this involves an aerosol of the digested sample being transferred to an argon plasma and excited. In the case of the ICP-OES method the excited atoms of the elements contained in the sample emit a characteristic electromagnetic radiation. In the ICP-MS procedure the element ions are diverted into a mass spectrometer where they are separated according to their mass/charge ratio and detected. The multiple-element procedure of X-ray fluorescence analysis, which is free of interference, is based on excitation of the element ions by X-ray radiation.

In addition to the use of inert products and materials during sample preparation, the entire sampling equipment should be composed of inert materials or should be appropriately selected for the element(s) to be analysed in order to avoid contamination.

The elemental content of the relevant metal is determined by all of the analytical techniques listed here. Element-specific analysis, e.g. the speciation of chromium(VI) compounds (DGUV 2017), can also be carried out with photometric, ion chromatographic or liquid chromatographic analytical methods. The species analysis will be presented in a special section.

3.3 Chemicals

Only batch-related certified chemicals may be used for digestion. The content of the analytical standards for calibration and quality assurance must be certifiably traceable to standard reference materials (see Table 10 in the Appendix). When commercially available standard solutions are used, it is essential to comply with the expiry date or the recommended period of use specified by the manufacturer.

- Ultrapure water ($\rho \geq 18.2 \text{ M}\Omega \times \text{cm}$ at 25 °C), low metal content and an especially low content of boron and alkalis
- Nitric acid; low metal content, content certified for each batch by the manufacturer, e.g.: 65% nitric acid, Suprapur®, Merck KGaA, Darmstadt
- Nitric acid; low metal content, content certified for each batch by the manufacturer, e.g.: 70% nitric acid, ULTREX II Ultrapure Reagent, J. T. Baker, Phillipsburg, USA
- Hydrochloric acid; low metal content, content certified for each batch by the manufacturer, e.g.: 30% hydrochloric acid, Suprapur®, Merck KGaA, Darmstadt
- Hydrochloric acid; low metal content, content certified for each batch by the manufacturer, e.g.: 36% hydrochloric acid, ULTREX II Ultrapure Reagent, J. T. Baker, Phillipsburg, USA
- Acetone, p.a., e.g. EMSURE® ACS, Merck KGaA, Darmstadt

Rare-earth elements, such as scandium, yttrium and terbium, are generally used as internal standards in ICP-MS methods, as these metals are generally of no significance at workplaces. In the case of XRF analysis e.g. gallium or rubidium are suitable as internal standards. As a rule, multiple-element standard solutions are employed for the simultaneous analytical determination by means of ICP-OES or ICP-MS.

Suitable single-element standard solutions can be used for calibration of AAS analytical methods.

4 Sample preparation methods for dust samples to determine “soluble metal compounds”

The sample preparation methods described below are suitable for the determination of such metals and metal compounds whose limit values are associated with the term “soluble” (Table 2). There is no clear and consistent definition for the term “soluble compounds”. As a rule, “soluble metallic compounds” are defined by the specific extraction agents and conditions (DIN 2010) described in measurement methods. Dilute hydrochloric acid is recommended as an extraction agent (Hahn 2000). When water is used as an extraction agent, strong fluctuations in the physicochemical properties (e.g. pH value and redox potential of the resulting solutions) may be caused by concomitant substances in the collected dust. Under certain circumstances, this may lead to changes in the behaviour of solutions of identical metal compounds and thus lead to scatter in the measured results.

Tab. 2 Limit values of soluble metals in dusts

Substance	Limit value (inhalable fraction) [mg/m ³]	
	List of MAK and BAT Values (DFG 2019)	TRGS 900 (AGS 2020 a)
Barium compounds, soluble (except barium oxide and barium hydroxide) (calculated as Ba)	0.5	0.5

These problems can be avoided by the use of dilute hydrochloric acid, as most metal chlorides are readily soluble. The use of buffer solutions or synthetic body fluids is also not advisable in everyday practice due to the various problems that may arise in the preparation of dusts, as treatment over a long period is often required when these extraction agents are used (Hahn 2000).

4.1 Determination of soluble metal compounds by means of open digestion

The methods described below can be used to determine soluble metal/metalloid elements and their compounds.

The loaded filter is transferred to the digestion vessel. Then the filter is covered by the recommended quantity of digestion mixture, whereby the volume to be added depends on the selected sampling conditions (see Table 3). In addition, it must be ensured that the loaded filter is completely immersed in the digestion agent. The digestion vessel is equipped with a boiling rod and an air cooler (approx. 40 cm long) and is subsequently kept in a thermostatically controlled heating block with suitable bore holes for two hours under reflux (block temperature approx. 125 °C). After cooling and sedimentation of the residue, the volume is read off and an aliquot of the supernatant solution is analysed. In the case of incomplete sedimentation an aliquot of the solution is filtered before analysis. The procedure described here may be modified depending on the apparatus available. The operational conditions for the determination of soluble metal compounds by means of digestion are summarised in Table 3.

Tab. 3 Operational conditions for the determination of soluble compounds

Parameter	Operational conditions		
	Filter diameter up to 37 mm	Filter diameter up to 70 mm	Filter diameter up to 150 mm
Digestion agent	0.1 M Hydrochloric acid		
Volume of digestion agent	20 ml	40 ml	80 ml
Pressure	Normal pressure	Normal pressure	Normal pressure
Temperature	Boiling temperature	Boiling temperature	Boiling temperature
Duration of treatment ^{a)}	2 h	2 h	2 h
Digestion vessel	Graduated digestion cylinder with a standard inner ground opening, mounted cooler and boiling rod		
	Volume: 25 ml Graduation: 0.2 ml Ground glass joint: NS 19/26	Volume: 50 ml Graduation: 0.5 ml Ground glass joint: NS 19/26	Volume: 100 ml Graduation: 0.5 ml Ground glass joint: NS 29/32

^{a)} The treatment duration can be reduced, if it can be demonstrated that comparable results are achievable.

5 Preparation methods for dust samples to determine the “total metal content”

The preparation methods described in the following sections should precede the determination of such metals and metal compounds whose limit values are based on the total metal content in dust.

Different preparation methods, with which the total metal content in dust samples can be determined, will be presented. The various procedures were developed in projects, compared through tests and finally evaluated. All the digestion methods described here yield comparable results and are suitable for determination of the total metal content. The suitability of the preparation methods for different metals and metal compounds is shown in Table 4.

Tab. 4 Suitability of the preparation methods

Element	Open digestion	High-pressure microwave digestion	Suspension method ^{a)}
Aluminium	+	+	+
Antimony	+	-	-
Arsenic	+	+	-
Barium	+	+	-
Beryllium	+	+	-
Lead	+	+	+
Boron	+	+	-
Cadmium	+	+	-
Chromium	+	+	+
Cobalt	+	+	+
Iron	+	+	+
Indium	+	+	-
Copper	+	+	+
Manganese	+	+	+
Molybdenum	+	+	+
Nickel	+	+	+
Selenium	+	+	+
Tellurium	+	+	-
Thallium	+	+	+
Titanium	+	+	+
Vanadium	+	+	+
Yttrium	+	+	+
Zinc	+	+	+

These methods are not suitable for certain compounds of these elements, e.g. Al₂O₃, TiO₂ and VC.

^{a)} The suspension method and the analytical determination of metals by means of TXRF permit further elements to be quantitatively determined under the conditions given in Section 5.4. These include Ag, Au, Ba, Bi, Ge, Hf, Nb, Pd, Pt, Re, Rh, Ru, Sr, Ta, Sn, W and Zr.

Relatively short treatment periods at a high temperature – possibly under pressure and with microwave treatment – have proved successful in practice. Possible interference by components of the dust is more easily prevented in this way.

The use of concentrated acids/acid solutions described in this section results in viscous solutions. The analytical samples must be appropriately diluted for determination of the metal content (e.g. by means of AAS or ICP-MS). The degree of dilution depends on the analytical device and the analytical method.

The elements contained in the dust to be analysed are converted to soluble salts by the sample digestion procedure, whereby the accompanying matrix is largely destroyed. Depending on the dust samples and the metals contained in them that are to be determined, different types of digestion are carried out. The conventions for the digestion methods will be described in the following sections. Open digestion and high-pressure microwave digestion can be

applied as equivalent procedures. A method involving suspension has proved suitable in combination with TXRF. This is described in more detail in Section 5.4.

5.1 Open digestion methods

In the case of open digestion, a mixture of hydrochloric acid and nitric acid is used (see Table 5). Nitric acid is effective due to its good dissolution properties and its oxidative effect, whereas hydrochloric acid serves as a solubiliser. The method described here is suitable for the metals/metalloids and their compounds in Table 4.

5.1.1 Sample preparation for open digestion

The loaded filter is transferred to the digestion vessel. Then the filter is covered by the recommended quantity of the digestion agent, whereby the quantity to be added depends on the selected sample preparation (see Table 5). It is important to ensure that the loaded filter is completely immersed in the digestion agent. The digestion vessel is equipped with a boiling rod and an air cooler (approx. 40 cm long) and is subsequently kept in a thermostatically controlled heating block with suitable bore holes for two hours under reflux (block temperature approx. 125 °C). After cooling, ultrapure water is cautiously added to the digestion vessel through the cooler and heating is repeated. After cooling again, the volume of the sample digestion solution is read off. Alternatively, after heating for 2 hours, the sample digestion solution can be transferred to a suitable volumetric flask (e.g. a 20 ml volumetric flask), rinsed with ultrapure water if necessary and then the flask is filled to the mark with ultrapure water. The sample solution is then homogenised once again. As a rule, a further dilution step is required before analysis. The degree of dilution depends on the analytical procedure used and on the selected method of measurement.

5.1.2 Operational conditions for open digestion

The operational conditions for the preparation of dust samples by means of open digestion are listed in Table 5.

Tab. 5 Operational conditions for the open digestion method

Parameter	Operational conditions		
	Filter diameter up to 37 mm	Filter diameter up to 70 mm	Filter diameter up to 150 mm
Digestion agent	2 parts by volume of nitric acid ($\geq 65\%$) and 1 part by volume of hydrochloric acid (25%)		
Volume of digestion agent	10 ml	20 ml	40 ml
Addition of ultrapure water after digestion	10 ml	20 ml	40 ml
Pressure	Normal pressure	Normal pressure	Normal pressure
Temperature	Boiling temperature	Boiling temperature	Boiling temperature
Treatment duration ^{a)}	2 h	2 h	2 h
Digestion vessel	Graduated digestion cylinder with standard inner ground joint, mounted cooler and boiling rod		
	Volume: 25 ml Graduation: 0.2 ml Ground glass joint: NS 19/26	Volume: 50 ml Graduation: 0.5 ml Ground glass joint: NS 19/26	Volume: 100 ml Graduation: 0.5 ml Ground glass joint: NS 29/32

^{a)} The treatment duration can be reduced if it can be demonstrated that comparable results are achievable.

The procedure described in this section may be modified depending on the apparatus available.

It is important to consider that some metals form readily volatile compounds and when open digestion methods are used, they could lead to diminished results.

5.2 High-pressure microwave digestion

High-pressure microwave digestion can be carried out as an alternative to open digestion methods. This procedure involves a closed system, in which the temperature and time can be varied.

Metals/metalloids and their compounds suitable for high-pressure microwave digestion are listed in Table 4.

Note: It is advisable to limit the diameter of the filter to be digested to a maximum of 70 mm on account of the dimensions of the commercially available digestion vessels.

Cellulose nitrate/acetate filters with a diameter of > 70 mm can be digested only by exercising special care. The resulting mass of filter material in the digestion vessel can lead to very high pressure in the microwave system, so that a successful high-pressure microwave digestion cannot be guaranteed. Processing of filter material masses > 200 mg is not advisable from the safety point of view.

5.2.1 Sample preparation for high-pressure microwave digestion

The loaded filter is transferred into the digestion vessel. Then the recommended quantity of the digestion agent is added to the filter. After digestion, the sample is transferred in its entirety to a suitable volumetric flask (e.g. 25 ml) and the flask is filled to the mark with ultrapure water. The sample is filtered and diluted if necessary and then transferred to the analytical instrument. The degree of dilution depends on the analytical technique used and on the selected measurement method.

5.2.2 Operational conditions for high-pressure microwave digestion

Table 6 shows the operational conditions for preparation by means of high-pressure microwave digestion in the comparative tests that were performed in this case.

Tab. 6 Operational conditions for the high-pressure microwave digestion method

Parameter	Operational conditions
	Filter diameter up to 70 mm
Digestion agent	Nitric acid ($\geq 65\%$)
Volume of digestion agent	10 ml
Addition of ultrapure water after digestion	Fill to 25 ml
Power	1100 Watt
Temperature	Max. 240 °C
Treatment duration	1 h (of which e.g. 15 min are set at 0.5 bar/s pressure increase)
Digestion vessel	High-pressure microwave digestion vessel made of PTFE

Similar programs that have been checked for their suitability can also be used.

5.3 Comparison of open digestion and high-pressure microwave digestion

The comparability of open digestion and high-pressure microwave digestion methods was checked by proficiency tests. The tests were carried out in different laboratories of members of the “Air Analyses” group of the Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area of the Deutsche Forschungsgemeinschaft (MAK Commission) (Pitzke et al. 2018 b).

In these investigations, various deposited dust samples were collected from real workplaces and further processed. Two types of dust from the collected dust samples were selected and homogenised for the proficiency tests. For

this purpose, the dust samples were ground with a mortar and sieved to give a particle size of $< 100 \mu\text{m}$ and the metals contained in the dust were qualitatively and quantitatively determined (Pitzke et al. 2018 a).

The studies of the participant laboratories were intended to simulate as closely as possible the investigation of real digestion of workplace samples. In addition to the dusts, a membrane filter (blank value) was also prepared in the same manner as the dust samples. Nine metals were chosen for analysis, i.e. cadmium, cobalt, chromium, copper, iron, manganese, nickel, lead and zinc. A total of ten experiments that encompassed the digestion conventions described here as well as further open and high-pressure microwave digestions were carried out (Pitzke et al. 2018 b). The results for most of the metals to be analysed show good agreement, except for the metals antimony, tin and tungsten, c.f. Table 4. The following conclusion can be drawn: high-pressure microwave digestion is an equally suitable digestion procedure for the determination of the total metal content in dust samples.

Furthermore, digestion methods whose parameters deviate from those given here can be used, provided the method has been tested and yields comparable results.

The results of the publication (Pitzke et al. 2018 b) also show that other digestion procedures lead to comparable results.

5.4 Suspension method

Suspension methods can also be employed as alternatives to open and high-pressure microwave digestion methods to determine the total metal content. In this case dust collected on a cellulose nitrate filter is suspended in acetone. The suspension method is suitable for the metals/metalloids and their compounds indicated as appropriate in Table 4. The quantitative analysis is subsequently performed by means of total reflection X-ray fluorescence (TXRF).

Further metals/metalloids can be quantitatively determined under the conditions stated in Section 5.4.2. These include the metals silver, gold, barium, bismuth, germanium, hafnium, niobium, palladium, platinum, rhenium, rhodium, ruthenium, strontium, tantalum, tin, tungsten and zirconium.

5.4.1 Sample preparation for the suspension method

The loaded filter is transferred to a digestion vessel made of quartz glass. First internal standards are added and then acetone as a dispersion agent. It is important to ensure that the loaded filter is completely immersed. The sample digestion vessel is sealed using a quartz glass ground stopper and shaken for 30 minutes on a laboratory shaker. The dust is kept in suspension by constant shaking of the sample solution and can thus be applied to a quartz glass sample carrier (already tested for blank values). A pipette is used to apply $3 \mu\text{l}$ of the sample suspension to each of five sample carriers, whereby the acetone already evaporates during application of the samples to the quartz glass sample carrier due to its high vapour pressure. In addition to the dust samples, a filter (blank value) is prepared and analysed in the same manner as the analytical samples in each case.

5.4.2 Operational conditions for the suspension method

Table 7 shows the operational conditions for the preparation by means of the suspension method.

Tab. 7 Operational conditions for the suspension method

Parameter	Operational conditions		
	Filter diameter up to 37 mm	Filter diameter up to 70 mm	Filter diameter up to 150 mm
Dispersion agent	Acetone p.a.		
Volume of dispersion agent	2 ml	6 ml	20 ml
Internal Standard	Gallium (1000 mg/l)	40 µl	80 µl
	Rubidium (1000 mg/l)	20 µl	40 µl
Treatment duration	30 min in the test-tube shaker		
Digestion vessel	Graduated digestion vessel with standard inner ground glass joint		
	Volume: 10 ml Graduation: 0.1 ml Ground glass joint: NS 14.5/20	Volume: 10 ml Graduation: 0.1 ml Ground glass joint: NS 14.5/20	Volume: 50 ml Graduation: 0.5 ml Ground glass joint: NS 29/32

6 Remarks on the described digestion methods

The digestion methods described here are conventional procedures. Experience has shown that results with good accordance are achieved in the majority of investigations. These methods have also proved successful in view of the resources and labour required for sample preparation. For the reproducibility of the measured values, a standardised sample work up is advantageous. In addition to the digestion procedures described here, methods for determination of the total metal content using other acids/acidic mixtures (hydrochloric acid, nitric acid, perchloric acid, hydrofluoric acid) were also tested as part of a round robin test (Pitzke et al. 2018 a). Volumes, concentrations and mixture ratios were thereby varied. The experiments showed that the concentration of the digestion medium as well as the digestion temperature and the duration of the digestion were crucial. Moreover, further proficiency tests (Breuer 1997) have demonstrated that the accuracy of the analytical results do not significantly differ from those of much more complex methods, even when the deposited dust cannot be completely dissolved. However, visibly residue-free solutions are always desirable.

In how far further-reaching research of a residue is absolutely necessary for the evaluation of the exposure situation at the workplace must be decided case-by-case. For special samples or analytes extended or different digestion methods may lead to better results, especially when additional information on the dust composition is available.

Thus, the volume, concentration and mixture ratio of acids or acidic mixtures (hydrochloric acid, nitric acid, hydrofluoric acid, perchloric acid) can be varied. Pressure digestion in closed systems (microwave) at higher temperatures with acid mixtures adapted to the metal/metalloid and its compounds may be suitable for poorly soluble alloy components. Melt digestion and subsequent dissolution of the melt cake are rarely necessary for airborne dusts.

The documentation “Empfohlene Analysenverfahren für Arbeitsplatzmessungen” [Recommended Analytical Methods for Measurements at the Workplace] (GA 13) (Hebisch and Poppek 2008) issued by the Bundesanstalt für Arbeitsschutz und Arbeitsmedizin [Federal Institute for Occupational Safety and Health] (BAuA) gives valuable advice on methods for quantitative investigation of metal-containing airborne dust. The digestion and measurement methods described here, such as e.g. AAS, ICP-OES/ICP-MS or XRF, permit only the elemental content of the metals and metal compounds in dust samples to be determined. This is sufficient for most investigations, as many

limit values are based on the metal concentration (on the basis of the elemental content of the relevant metal). The frequently required differentiation between compounds of the metals or oxidation states (species analysis) for toxicological reasons cannot be achieved by analysis in many cases. In such circumstances the “worst case” scenario should be considered, i.e. comparison of the elemental concentration in the air (after conversion with the corresponding stoichiometric factor) with the limit value. However, it is advisable to resort to recognised, specific methods, e.g. Cr(VI) by means of UV/VIS or IC, or Sb(III, V) by means of the hydride technique if these are available (DGUV 2009, 2017).

7 Quality control

The complete measurement method should be subject to regular quality control. This can take the form of internal quality assurance measures or by participation in proficiency tests in which filters loaded with dust or dust samples are tested (Nürnberg et al. 2017). The internal quality assurance can be achieved by repeated analysis of certified reference materials (dusts). Thus, the precision and accuracy of the analytical method can be checked. However, the influence of sampling on the measurement result is not taken into account in this case (see Hebisch et al. 2012).

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Appendix

Tab. 8 List of metals and metal compounds with their evaluation category

Name	EC No.	CAS No.
Arsenic compounds (classified as Carc. 1A, Carc. 1B (AGS 2020 b))		
Arsine	232-066-3	7784-42-1
Barium compounds, soluble (except barium oxide and barium hydroxide)		
Beryllium and its inorganic compounds		7440-41-7
Boric acid and sodium borate	233-139-2	10043-35-3
Boron trifluoride	231-569-5	7637-07-2
Boron trifluoride dihydrate	231-569-5	13319-75-0
Cadmium and Cd compounds (classified as Carc. 1A, Carc. 1B (AGS 2020 b))		
Calcium cyanamide	205-861-8	156-62-7
Calcium dihydroxide	215-137-3	1305-62-0
Calcium oxide	215-138-9	1305-78-8
Calcium sulphate	231-900-3	7778-18-9
Chromium(VI) compounds		
Chromium and inorganic chromium(II) and (III) compounds	231-157-5	7440-47-3
Cobalt and Cobalt compounds (classified as Carc. 1A, Carc. 1B (AGS 2020 b))		
Decaborane	241-711-8	17702-41-9
Dihydrogen selenide (selenium hydride)	231-978-9	7783-07-5
Indium, indium oxide, indium hydroxide and indium phosphide (the OEL is based in the elemental content of indium)		7440-74-6 1312-43-2 20661-21-6 or 55326-87-9 22398-80-7
Potassium cyanide (as CN)	205-792-3	151-50-8
Potassium hydroxide		
Lithium hydride	231-484-3	7580-67-8
Lithium and strongly irritating lithium compounds (such as lithium amide, lithium hydride, lithium hydroxide lithium nitride, lithium oxide, lithium aluminium hydride, lithium tetrahydroborate)		
Lithium compounds, inorganic, with the exception of lithium and strongly irritating lithium compounds		
Manganese and its inorganic compounds	231-105-1	7439-96-5
Sodium azide	247-852-1	26628-22-8
Sodium 2-biphenylate	205-055-6	132-27-4
Sodium cyanide (as CN)	205-599-4	143-33-9
Sodium fluoroacetate	200-548-2	62-74-8
Sodium hydroxide		1310-73-2
Sodium trichloroacetate		650-51-1

Tab. 8 (continued)

Name	EC No.	CAS No.
Metallic nickel	231-111-4	7440-02-0
Nickel compounds (classified as Carc. 1A, Carc. 1B (AGS 2020 b))		
Platinum (metallic)	231-116-1	7440-06-4
Mercury	231-106-7	7439-97-6
Mercury compounds, inorganic		
Selenium	231-957-4	7782-49-2
Selenium compounds, inorganic		
Silver	231-131-3	7440-22-4
Silver compounds, inorganic		
Tetraethyllead	201-075-4	78-00-2
Tetraethyl silicate (TEOS)	201-083-8	78-10-4
Tetramethyllead	200-897-0	75-74-1
Vanadium compounds, inorganic, valence 4+ and 5+ (e.g. divanadium pentaoxide)	e.g. 215-239-8	e.g. 1314-62-1
Tin(II) compounds, inorganic		
Tin(IV) compounds, inorganic		
Tin compounds, organic		
– n-Butyltin compounds		
Mono-n-butyltin compounds,		
Di-n-butyltin compounds,		
Tri-n-butyltin compounds and		
Tetra-n-butyltin	215-960-8	1461-25-2
– Methyltin compounds,		
Monomethyltin and dimethyltin compounds with the exception of the separately named compounds		
Triisooctyl-2,2',2''-(methylstannylidyne)tris(thio)triacetate,	259-374-0	54849-38-6
Bis[methyltindi(isooctylmercaptoacetate)]sulphide,		
Bis[methyltindi(2-mercaptoethyloleate)]sulphide		59118-99-9
Diisooctyl-2,2'-((dimethylstannylene)bis(thio))diacetate,	247-862-6	26636-01-1
2-Ethylhexyl 10-ethyl-4,4-dimethyl-7-oxo-8-oxa-3,5-dithia-4-stannatetradecanoate,	260-829-0	57583-35-4
Bis[dimethyltin(isooctylmercapto-acetate)]sulphide,		
Bis[dimethyltin(2-mercaptoethyloleate)]sulphide		
Trimethyltin compounds and tetramethyltin	209-833-6	594-27-4
– n-Octyltin compounds		
Mono-n-octyltin compounds,		
Di-n-octyltin compounds,		
Tri-n-octyltin compounds and		
Tetra-n-octyltin	222-733-7	3590-84-9
– Phenyltin compounds		

Tab. 8 (continued)

Name	EC No.	CAS No.
Ziram (zinc bis(<i>N,N</i> -dimethyldithiocarbamate))	205-288-3	137-30-4
Zirkonium and compounds that are insoluble in water	231-176-9	7440-67-7

Remarks on Table 8:

Lithium compounds, inorganic, with the exception of lithium and strongly irritating lithium compounds

The term “strongly irritating” is not explained in TRGS 900 (AGS 2020 a). Which compounds are strongly irritating can be found only in the MAK Documentation. Lithium amide, lithium hydride, lithium hydroxide, lithium nitride, lithium oxide, lithium tetrahydroaluminate and lithium tetrahydroborate are named as examples of strongly irritating compounds there

Zirkonium and compounds that are not soluble in water

Solubility is not defined

Tab. 9 Examples of filter materials and collection phases for dust sampling

Filter materials	Type	Manufacturer/supplier
Cellulose nitrate filter	Type 11301	Sartorius, 37079 Göttingen, Germany
	AE99	Whatman GmbH, 37586 Dassel, Germany
Cellulose acetate filter	Type 11104	Sartorius, 37079 Göttingen, Germany
Quartz fibre filter	QM-A	Whatman GmbH, 37586 Dassel, Germany
	Quartz microfibre filter T293	Sartorius, 37079 Göttingen, Germany
	Ederol T293	Binzer & Munktell Filter GmbH, 35088 Battenberg, Germany
	Quartz microfibre filter MK 360	Binzer & Munktell Filter GmbH, 35088 Battenberg, Germany
Glass fibre filter	MN 85/90 BF	Macherey & Nagel, 52313 Düren, Germany
Fluoropolymer filter	Zefluor	Pall GmbH, 63303 Dreieich, Germany
Other filter materials	Polycarbonate	Nuclepore Filter
	Polyvinyl chloride	Whatman GmbH, 37586 Dassel, Germany
Polyurethane foam	Bulpren S 31048	Eurofoam Deutschland GmbH Schaumstoffe, 65203 Wiesbaden, Germany
	Bulpren BF 38049	Eurofoam Deutschland GmbH Schaumstoffe, 65203 Wiesbaden, Germany

Tab. 10 Examples of suitable chemicals and chemical manufacturers

Chemical	Type	Manufacturer
Nitric acid	65% Suprapur [®]	Merck KGaA, 64293 Darmstadt, Germany
	65% for metal trace analysis	AppliChem GmbH, 64291 Darmstadt, Germany
	65% ROTIPURAN [®] , p.a., ISO	Carl Roth GmbH + Co. KG, 76185 Karlsruhe, Germany
	70% ULTREX II Ultrapure Reagent	J. T. Baker, (supplied by Fisher Scientific, 58239 Schwerte, Germany)
Hydrochloric acid	25% ROTIPURAN [®] , p.a., ISO	Carl Roth GmbH + Co. KG, 76185 Karlsruhe, Germany
	30% Suprapur [®]	Merck KGaA, 64293 Darmstadt
	37% p.a., ACS, ISO	AppliChem GmbH, 64291 Darmstadt, Germany
	37% ROTIPURAN [®]	Carl Roth GmbH + Co. KG, 76185 Karlsruhe, Germany
	36% ULTREX II Ultrapure Reagent	J. T. Baker, (supplied by Fisher Scientific, 58239 Schwerte, Germany)
Acetone	p.a. EMSURE [®] , ACS, ISO	Merck KGaA, 64293 Darmstadt, Germany
Standard	1000 mg Element Titrisol ^{®a)}	Merck KGaA, 64293 Darmstadt, Germany
	ICP Multi-Element Standard Solution, 1000 mg/l, CertiPUR ^{®a), c)}	Merck KGaA, 64293 Darmstadt, Germany
	Multi-element Solution, 10 mg/l ^{a)}	Spex CertiPrep, Metuchen, USA
	Multi-element Calibration Standard, 10 mg/l ^{a)}	PerkinElmer LAS, 63110 Rodgau, Germany
	Element Plasma Standard Solution, 1000 mg/l Specpure ^{®a), b), c)} (as an Internal standard e.g.: Sc, Y, Tb, Ga, Rb)	Alfa Aesar GmbH & Co KG, 76185 Karlsruhe, Germany
	ICP Quality Control Standard, 100 mg/l ^{c)}	Spex CertiPrep, Metuchen, USA
	Multi-element Quality Control for ICP, 1 mg/l ^{c)}	VWR International GmbH, 64295 Darmstadt, Germany

a) Used as a calibration standard

b) Used as an internal standard

c) Used as a quality control standard