
**Workplace air — Determination of
metals and metalloids in airborne
particulate matter by inductively
coupled plasma atomic emission
spectrometry —**

**Part 2:
Sample preparation**

*Air des lieux de travail — Détermination des métaux et métalloïdes
dans les particules en suspension dans l'air par spectrométrie
d'émission atomique avec plasma à couplage inductif —*

Partie 2: Préparation des échantillons

ISO 15202-2:2020

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This third edition cancels and replaces the second edition (ISO 15202-2:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

- Definitions that appear in ISO 18158 have been removed from ISO 15202-2, with ISO 18158 being added as a reference (replacing references to EN 1540).
- References to EN 482 have been replaced with ISO 20581, and references to EN 13890 have been replaced with ISO 21832.
- Information regarding digestion of acid-soluble internal capsules has been added to [Annexes C, D, E, F, G and H](#).
- The text has been editorially updated.

A list of all parts in the ISO 15202 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The health of workers in many industries is at risk through exposure by inhalation of toxic metals and metalloids. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure, and this is generally achieved by making workplace air measurements. This document has been published in order to make available a method for making valid exposure measurements for a wide range of metals and metalloids in use in industry. It will be of benefit to agencies concerned with health and safety at work, industrial hygienists and other public health professionals, analytical laboratories, industrial users of metals and metalloids and their workers.

ISO 15202, published in three parts, specifies a generic method for the determination of the mass concentration of metals and metalloids in workplace air using inductively coupled plasma atomic emission spectrometry (ICP-AES).

- ISO 15202-1 gives details of relevant International, European and National Standards which specify characteristics, performance requirements and test methods relating to sampling equipment. It also augments guidance provided elsewhere on assessment strategy and measurement strategy, as well as specifying a method for collecting samples of airborne particulate matter for subsequent chemical analysis.
- ISO 15202-2 describes a number of procedures for preparing sample solutions for analysis by ICP-AES.
- ISO 15202-3 gives requirements and test methods for the analysis of sample solutions by ICP-AES.

The sample preparation methods described in this part of ISO 15202 are generally suitable for use with analytical techniques other than ICP-AES; e.g. atomic absorption spectroscopy (AAS) and inductively coupled plasma mass spectrometry (ICP-MS).

It has been assumed in the drafting of this document that the execution of its provisions and the interpretation of the results obtained are entrusted to appropriately qualified and experienced people.

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Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry —

Part 2: Sample preparation

WARNING — The use of this document may involve hazardous materials, operations and equipment. This document does not purport to address any safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies a number of suitable methods for preparing test solutions from samples of airborne particulate matter collected using the method specified in ISO 15202-1, for subsequent determination of metals and metalloids by ICP-AES using the method specified in ISO 15202-3. It contains information about the applicability of the methods with respect to the measurement of metals and metalloids for which limit values have been set. The methods can also be used in the measurement of some metals and metalloids for which limit values have not been set but no information about its applicability is provided in this case.

NOTE The sample preparation methods described in this document are generally suitable for use with analytical techniques other than ICP-AES, e.g. atomic absorption spectrometry (AAS) by ISO 8518^[5] and ISO 11174^[10] and inductively coupled plasma mass spectrometry (ICP-MS) by ISO 30011^[11].

The method specified in [Annex B](#) is applicable when making measurements for comparison with limit values for soluble metal or metalloid compounds.

One or more of the sample dissolution methods specified in [Annexes C](#) through [H](#) are applicable when making measurements for comparison with limit values for total metals and metalloids and their compounds. Information on the applicability of individual methods is given in the scope of the annex in which the method is specified.

The following is a non-exclusive list of metals and metalloids for which limit values have been set (see References [\[14\]](#) and [\[15\]](#)) and for which one or more of the sample dissolution methods specified in this document are applicable. However, there is no information available on the effectiveness of any of the specified sample dissolution methods for those elements in italics.

Aluminium	Calcium	Magnesium	Selenium	Tungsten
Antimony	Chromium	Manganese	Silver	<i>Uranium</i>
Arsenic	Cobalt	Mercury	Sodium	Vanadium
Barium	Copper	Molybdenum	Strontium	Yttrium
Beryllium	<i>Hafnium</i>	Nickel	<i>Tantalum</i>	Zinc
Bismuth	<i>Indium</i>	Phosphorus	Tellurium	Zirconium

Boron	Iron	Platinum	Thallium
Caesium	Lead	Potassium	Tin
Cadmium	Lithium	Rhodium	Titanium

ISO 15202 is not applicable to the determination of elemental mercury or arsenic trioxide, since mercury vapour and arsenic trioxide vapour are not collected using the sampling method specified in ISO 15202-1.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 15202-1, *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 1: Sampling*

ISO 15202-3, *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 3: Analysis*

ISO 18158, *Workplace air — Terminology*

ISO 21832, *Workplace air — Metals and metalloids in airborne particles — Requirements for evaluation of measuring procedures*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org>

3.1 sample dissolution

process of obtaining a solution containing all analytes of interest from a sample, which might or might not involve complete dissolution of the sample

3.2 sample solution

solution prepared from a sample by the process of *sample dissolution* (3.1)

Note 1 to entry: A sample solution might need to be subjected to further operations, e.g. dilution, or addition, or both, of an internal standard(s), in order to produce a *test solution* (3.3).

SOURCE: Adapted from EN 14902:2005, 3.1.22.

3.3 test solution

blank solution or *sample solution* (3.2) that has been subjected to all operations required to bring it into a state in which it is ready for analysis

Note 1 to entry: “Ready for analysis” includes any required dilution or addition, or both, of an internal standard. If a blank solution or *sample solution* (3.2) is not subject to any further operations before analysis, it is a test solution.

SOURCE: EN 14902:2005, 3.1.30.

4 Principle

4.1 Airborne particles containing metals and metalloids are collected by drawing a measured volume of air through a collection substrate, typically a filter mounted in a sampler designed to collect an appropriate size fraction of airborne particles, using the method specified in ISO 15202-1.

4.2 An appropriate and suitable sample dissolution method is selected from those specified in [Annexes B](#) through [H](#), taking into consideration the metals and metalloids which are to be determined, the limit values that have been set for those metals and metalloids, the applicability of the methods for dissolution of the metals and metalloids of interest from materials which could be present in the test atmosphere and the availability of the required laboratory apparatus.

4.3 The sample and collection substrate are then treated to dissolve the metals and metalloids of interest using the selected sample dissolution method.

4.4 The resultant test solution is subsequently analysed for the metals and metalloids of interest by inductively coupled plasma-atomic emission spectrometry using the method specified in ISO 15202-3.

NOTE Sample preparation methods described in [Annexes B](#) through [H](#) are generally suitable for use with analytical techniques other than ICP-AES, e.g. AAS by ISO 8518^[5] and ISO 11174^[10] and ICP-MS by ISO 30011^[11]. For ICP-MS, changes could be required in the concentrations of acids or the dilution factors used to prepare test solutions. Furthermore, some acids, such as hydrochloric acid, are not recommended for test solutions for analysis by ICP-MS.

5 Requirements

The measuring procedure as a whole (covered by ISO 15202-1, ISO 15202-2 and ISO 15202-3) shall conform to any relevant performance requirements for measuring chemical agents in workplace air (for example ISO 20581^[12] and ISO 21832).

6 Reactions

In general, the majority of particulate metals and metalloids and particulate metal and metalloid compounds which are commonly of interest in samples of workplace air are converted to water-soluble ions by one or more of the sample dissolution methods specified in this document. However, if there is any doubt about whether a method will exhibit the required analytical recovery for a particular application, it is necessary to investigate this before proceeding with the method (see [10.1](#)).

7 Reagents

During the analysis, use only reagents of analytical grade and only water as specified in [7.1](#).

NOTE 1 Safety precautions to be observed when using hydrofluoric and perchloric acids are given in [Annex A](#).

NOTE 2 Details of reagents that are required for use in [Annexes B](#) through [I](#) are given in the annex concerned.

NOTE 3 It could be necessary to use acids of higher purity in order to obtain an adequate detection limit for some metals and metalloids.

7.1 Water, conforming with the requirements for ISO 3696^[3] grade 2 water (electrical conductivity less than 0,1 mS/m and resistivity greater than 0,01 MΩ·m at 25 °C).

It is recommended that the water used be obtained from a water purification system that delivers ultrapure water having a resistivity greater than 0,18 MΩ·m (usually expressed by manufacturers of water purification systems as 18 MΩ·cm).

7.2 Nitric acid (HNO_3), concentrated, $\rho_{\text{HNO}_3} \approx 1,42 \text{ g ml}^{-1}$, $w_{\text{HNO}_3} \approx 70 \text{ \%}$ mass fraction.

The concentration of the metals and metalloids of interest shall be less than $0,1 \text{ }\mu\text{g ml}^{-1}$.

WARNING — Concentrated nitric acid is corrosive and oxidizing, and nitric acid fumes are irritant. Avoid exposure by contact with the skin or eyes, or by inhalation of fumes. Use suitable personal protective equipment (including suitable gloves, face shield or safety spectacles, etc.) when working with the concentrated or dilute nitric acid and carry out sample dissolution with concentrated nitric acid in open vessels in a fume hood.

7.3 Nitric acid, diluted 1 + 9.

Carefully and slowly begin adding 50 ml of concentrated nitric acid (7.2) to 450 ml of water (7.1) in a 1 l polypropylene bottle (8.5). Add the acid in small aliquots. Between additions, swirl to mix and run cold tap water over the side of the bottle to cool the contents. Do not allow the tap water to contaminate the contents of the bottle. When the addition of the concentrated nitric acid is complete, swirl the bottle to mix the contents, allow to cool to room temperature, close the bottle with its screw cap and mix thoroughly.

8 Laboratory apparatus

Usual laboratory apparatus and, in particular, the following.

8.1 Disposable gloves, impermeable and powder free, to avoid the possibility of contamination from the hands and to protect them from contact with toxic and corrosive substances. PVC gloves are suitable.

8.2 Glassware, beakers and one-mark volumetric flasks conforming to the requirements of ISO 1042^[1], made of borosilicate glass conforming with the requirements of ISO 3585^[2], cleaned before use by soaking in 1 + 9 nitric acid (7.3) for at least 24 h and then rinsing thoroughly with water (7.1).

Alternatively, the glassware may be cleaned with a suitable laboratory detergent using a laboratory washing machine.

8.3 Flat-tipped forceps, non-metallic (e.g. plastic or plastic-coated), for unloading filters from samplers or from filter transport cassettes.

8.4 Piston-operated volumetric instruments, conforming with the requirements of ISO 8655-1^[6] and tested in accordance with ISO 8655-6^[9], including **pipettors** conforming with the requirements of ISO 8655-2^[7] and **dispensers** conforming with the requirements of ISO 8655-5^[8], for dispensing leach solution, acids, etc.

8.5 Polypropylene bottle, 1 l capacity, with leakproof screw cap.

A bottle made of an alternative plastic may be used provided that it is suitable for the intended use (see 7.3).

NOTE Details of laboratory apparatus that are required for use in Annexes B through I are given in the annex concerned.

9 Procedure

9.1 Soluble metal and metalloid compounds

9.1.1 If results are required for comparison with limit values for soluble metal or metalloid compounds, or both, use the sample dissolution method specified in Annex B to prepare test solutions for analysis by the method specified in ISO 15202-3.

9.1.2 Alternatively, if it is known that no insoluble compounds of the metals or metalloids, or both, of interest are used in the workplace and that none are produced in the processes carried out, prepare test solutions for analysis by the method specified in ISO 15202-3, using one of the sample dissolution methods for total metals and metalloids and their compounds prescribed in [Annexes C](#) through [H](#), and compare the results with the limit value for the soluble metals or metalloids, or both, concerned.

The methods prescribed in [Annexes C](#) through [H](#) are not specific for soluble metal or metalloid compounds, or both. However, in the circumstances described above, they may be used as an alternative to the method described in [Annex B](#), if this is more convenient.

9.2 Total metals and metalloids and their compounds

9.2.1 If results are required for comparison with limit values for total metals or metalloids, or both, and their compounds, select a suitable sample dissolution method from those specified in [Annexes C](#) through [H](#). Take into consideration the applicability of each method for dissolution of the metals and metalloids of interest from materials that could be present in the test atmosphere (refer to the clause on the effectiveness of the sample dissolution method in the annex in which the method is specified) and the availability of the required laboratory apparatus.

9.2.2 Use the selected sample dissolution method to prepare test solutions for analysis of total metals and metalloids and their compounds by the method specified in ISO 15202-3.

9.3 Mixed exposure

9.3.1 If results are required

- for comparison with limit values for soluble metal and/or metalloid compounds **and** with limit values for metals and/or metalloids and their insoluble compounds, or
- for comparison with limit values for soluble metal and/or metalloid compounds **and** with limit values for total metals and/or metalloids and their compounds,

follow the instructions given in [9.3.2](#) and [9.3.3](#).

9.3.2 Use the sample dissolution method specified in [Annex B](#) to prepare test solutions for the determination of soluble metal and metalloid compounds by the method specified in ISO 15202-3.

9.3.3 Select a suitable sample dissolution method for total metals and metalloids and compounds (see [9.2](#)). Use this to treat undissolved material from the method for soluble metal and metalloid compounds (see [B.6.7.1](#)) and prepare test solutions for determination of metals and metalloids and their insoluble compounds by the method specified in ISO 15202-3.

10 Special cases

10.1 Action to be taken if there is doubt about the effectiveness of the selected sample dissolution method

10.1.1 If there is any doubt about whether the selected sample dissolution method will exhibit the required analytical recovery when used for dissolution of the metals and metalloids of interest from materials which could be present in the test atmosphere, determine its effectiveness for that particular application. For total metals and metalloids, this may be achieved by analysing a bulk sample of known composition which is similar in nature to the materials being used or produced in the workplace, e.g. a certified reference material. For soluble metals and metalloids, analytical recovery is best determined by analysing filters spiked with solution containing a known mass of the soluble compound of interest.

NOTE In designing an experiment to determine the effectiveness of a sample dissolution method, the particle size of a bulk sample can have a significant influence on the efficiency of its dissolution. Furthermore, microgram amounts of relatively insoluble material are normally much more easily dissolved than milligram amounts.

10.1.2 If the analytical recovery is less than the minimum acceptable value prescribed in ISO 21832 (analytical recovery at least 90 % with a coefficient of variation less than 5 %), investigate the use of an alternative sample dissolution method. This may be a method not specified in this document if it can be demonstrated that its analytical recovery meets the requirements of ISO 21832.

10.1.3 Do not use a correction factor to compensate for an apparently ineffective sample dissolution method, since this might equally lead to erroneous results.

10.2 Action to be taken when particles have become dislodged from the filter during transportation

When the filter transport cassettes or samplers are opened, it is advisable to look for evidence that particles have become dislodged from the filter during transportation. If this appears to have occurred, wash the internal surfaces of the filter transport cassette or sampler in the sample dissolution vessel in order to recover the material concerned. Before analysis is carried out, inform the originator of the sample of the condition in which it was received so that the originator can make a judgement as to whether it is to be analysed.

10.3 Action to be taken regarding sampler wall deposits

Particles are frequently found to have deposited on the interior walls of samplers, and for many samplers these particles comprise an integral, and often substantial component of the sample. In such cases, action shall be taken to include these deposits in the analysis. Additional information is provided in [Annex J](#).

11 Laboratory records

11.1 Record details of all reagent sources (lot numbers) used for sample preparation.

11.2 Record details of laboratory apparatus used for sample preparation, where this is relevant, e.g. the serial number of equipment when there is more than one item of equipment of the same type in the laboratory.

11.3 Record any deviations from the specified methods.

11.4 Record any unusual events or observations during sample preparation.

Annex A

(informative)

Safety precautions to be observed when using hydrofluoric and perchloric acids

A.1 Special precautions to be observed when using hydrofluoric acid

A.1.1 Take extreme care when using hydrofluoric acid. Ensure that the nature and seriousness of hydrofluoric acid burns is understood before commencing work with this substance.

The burning sensation associated with many concentrated acid burns is not immediately apparent on exposure to hydrofluoric acid and may not be felt for several hours. Relatively dilute solutions of hydrofluoric acid can also be absorbed through the skin, with serious effects similar to those resulting from exposure to the concentrated acid.

When using hydrofluoric acid, it is recommended that a pair of disposable gloves is worn underneath suitable rubber gloves to provide added protection for the hands.

A.1.2 Carry hydrofluoric acid burn cream (containing calcium gluconate) at all times while working with hydrofluoric acid and for 24 h afterwards. Apply the cream to any contaminated skin, after washing the affected area with copious amounts of water. Obtain medical advice immediately in case of an accident. Calcium gluconate cream has a limited lifetime and should be replaced prior to its expiration date.

A.2 Special precautions to be observed when using perchloric acid

A.2.1 Perchloric acid forms explosive compounds with organics and with many metal salts. When performing sample dissolution using this acid, ensure that any organic material present is destroyed, e.g. by heating with nitric acid before addition of perchloric acid.

A.2.2 Do not allow perchloric acid solutions containing high concentrations of metal salts to boil dry, as solid perchlorates are shock-sensitive and can explode.

A.2.3 Perform sample dissolution using a special fume cupboard designed for the use of perchloric acid and incorporating a scrubbing system to remove acid vapours from exhaust gases so as to prevent the possibility of potentially explosive material accumulating in ducts.

Annex B (normative)

Sample dissolution method for soluble metal and metalloid compounds

B.1 Scope

B.1.1 This annex specifies a method for the dissolution of soluble metal and metalloid compounds using a suitable leach solution.

B.1.2 The method is applicable in all instances, except when use of a specific leach solution or leach conditions, or both, is prescribed in National Standards or Regulations.

B.1.3 Metals for which limit values for soluble compounds have typically been set (see References [14] and [15]), and for which the sample dissolution method specified in this annex is applicable, are listed below:

Aluminium	Molybdenum	Platinum	Silver	Tungsten
Barium	Nickel	Rhodium	Thallium	Uranium

NOTE 1 The above list is based upon the applicability of the sample dissolution procedure reported in References [17], [18] and [19], with adaptation based on expert judgement. Furthermore, the list is not comprehensive and the procedure will be effective for some metals and metalloids that are not listed.

NOTE 2 The sample dissolution method specified in this annex can also be used for the dissolution of soluble zinc compounds, e.g. for determination of zinc chloride in the presence of zinc oxide in galvanizing fume.

B.2 Effectiveness of the sample dissolution method

B.2.1 Soluble compounds of metals and metalloids are essentially defined by the specific leach solutions and leach conditions used in the measurement methods prescribed for their determination. (This is because, except for compounds that are very soluble or very insoluble in water, solubility can be dependent upon the nature of the leach solution and parameters such as particle size, solute/solvent ratio, pH, temperature, etc.) Consequently, the sample dissolution method, by definition, gives the desired result.

NOTE The repeatability and reproducibility of water leach for soluble metals has been demonstrated for welding fume in an interlaboratory study[43].

B.2.2 Although the sample dissolution method for soluble compounds prescribed in this document is design-based, there are circumstances in which it can give incorrect results. In particular, this can occur if a soluble compound reacts with the filter material, or a contaminant on the filter, to produce an insoluble compound. For example, a low recovery will be obtained for soluble silver compounds if the filter used is contaminated with chloride. It is therefore important that proper consideration is given to chemical compatibility when selecting a filter for collecting samples of soluble compounds (see ISO 15202-1). If it is believed that there could be a chemical compatibility problem, tests should be performed to confirm that analytical recovery is satisfactory before samples are collected (see 10.1.1). Low recoveries for soluble silver can also occur if samples are exposed to light[20].