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

Part 2:

Sample preparation

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*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

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 15202-2 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This second edition cancels and replaces the first edition (ISO 15202-2:2001), which has been technically revised. The major changes in the second edition are as follows.

- Definitions have been updated.
- In Annex B, use of ammonium citrate leach solution has been eliminated.
- A new Annex H has been added to provide a method for sample dissolution using a 95 °C hot block dissolution system. The original Annex H is now Annex I.
- A new Annex J has been added to provide guidance regarding sampler wall deposits.

Annexes B through I form a normative part of this document. Annex A and Annex J are for information only.

ISO 15202 consists of the following parts, under the general title *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry*:

- *Part 1: Sampling*
- *Part 2: Sample preparation*
- *Part 3: Analysis*

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 part of ISO 15202 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 (i.e. this part) 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 part of ISO 15202 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 part of ISO 15202 may involve hazardous materials, operations and equipment. This part of ISO 15202 does not purport to address any safety problems associated with its use. It is the responsibility of the user of this part of ISO 15202 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

1.1 This part of ISO 15202 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 part of ISO 15202 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].

1.2 The method specified in Annex B is applicable when making measurements for comparison with limit values for soluble metal or metalloid compounds.

1.3 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.

1.4 The following is a non-exclusive list of metals and metalloids for which limit values have been set (see References [15] and [16]) and for which one or more of the sample dissolution methods specified in this part of ISO 15202 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	<i>Platinum</i>	Thallium	
Caesium	Lead	Potassium	Tin	
Cadmium	Lithium	<i>Rhodium</i>	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 referenced documents are indispensable for the application 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*

EN 13890, *Workplace exposure — Procedures for measuring metals and metalloids in airborne particles — Requirements and test methods*

3 Terms and definitions

For the purposes of this part of ISO 15202, the following terms and definitions apply.

3.1 analysis

all operations carried out after sample preparation to determine the amount or concentration of the analyte(s) of interest present in the sample

NOTE Adapted from EN 14902:2005^[14], 3.1.1.

3.2 analytical recovery

ratio of the mass of analyte measured in a sample to the known mass of analyte in that sample

NOTE The analytical recovery is usually given as a percentage.

[EN 1540:2011^[13]]

3.3**chemical agent**

any chemical element or compound, on its own or admixed as it occurs in the natural state or as produced, used, or released including release as waste, by any work activity, whether or not produced intentionally and whether or not placed on the market

[Council Directive 98/24/EC^[17], Art. 2(a)]

3.4**exposure by inhalation**

situation in which a chemical agent is present in the air that is inhaled by a person

NOTE Adapted from EN 1540:2011^[13].

3.5**occupational exposure limit value
limit value**

limit of the time-weighted average of the concentration of a chemical agent in the air within the breathing zone of a worker in relation to a specified reference period

[Council Directive 98/24/EC^[17], Art. 2(d)]

EXAMPLES Threshold Limit Values[®] (TLVs) established by the ACGIH^[15] and Indicative Occupational Exposure Limit Values (IOELVs) promulgated by the European Commission (Council Directive 2006/15/EC^[16]).

3.6**measuring procedure
measurement procedure**

set of operations, described specifically, for the sampling and analysis of chemical agents in air

NOTE 1 A measuring procedure for the sampling and analysis of chemical agents in air usually includes the following steps: preparation for sampling, sampling, transportation and storage, preparation of samples for analysis and analysis.

NOTE 2 Adapted from EN 1540:2011^[13].

3.7**air sampler
sampler**

device for separating chemical agents from the surrounding air

NOTE 1 Air samplers are generally designed for a particular purpose, e.g. for sampling gases and vapours or for sampling airborne particles.

NOTE 2 Adapted from EN 1540:2011^[13].

3.8**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

3.9**sample preparation**

all operations carried out on a sample, usually after transportation and storage, to prepare it for analysis, including transformation of the sample into a measurable state, where necessary

NOTE Adapted from EN 14902:2005^[14], 3.1.24.

3.10**sample solution**

solution prepared from a sample by the process of sample dissolution

NOTE 1 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.

NOTE 2 Adapted from EN 14902:2005^[14], 3.1.22.

**3.11
test solution**

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

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

NOTE 2 Adapted from EN 14902:2005^[14], 3.1.30.

**3.12
workplace**

designated area or areas in which the work activities are carried out

[EN 1540:2011^[13]]

4 Principle

4.1 Airborne particles containing metals and metalloids are collected by drawing a measured volume of air through 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 filter and collected sample 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 of this part of ISO 15202 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 might 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 comply with any relevant International, European or National Standards that specify performance requirements for measuring chemical agents in workplace air (for example EN 482^[12] and EN 13890).

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 part of ISO 15202. 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 Details of reagents that are required for use in Annexes B through I are given in the annex concerned.

NOTE 2 It might be necessary to use acids of higher purity in order to obtain an adequate detection limit for some metals and metalloids.

7.1 Water, complying with the requirements for ISO 3696^[3] grade 2 water (electrical conductivity less than 0,1 mS/m and resistivity greater than 0,01 M Ω ·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 M Ω ·m (usually expressed by manufacturers of water purification systems as 18 M Ω ·cm).

7.2 Nitric acid (HNO₃), concentrated, $\rho_{\text{HNO}_3} \approx 1,42 \text{ g ml}^{-1}$, $w_{\text{HNO}_3} \approx 70 \%$ mass fraction.

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

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.

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7.3 Nitric acid, diluted 1 + 9.

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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

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

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 complying with the requirements of ISO 1042^[1], made of borosilicate glass 3.3 complying 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, complying with the requirements of ISO 8655-1^[6] and tested in accordance with ISO 8655-6^[9], including **pipettors** complying with the requirements of ISO 8655-2^[7] and **dispensers** complying 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).

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.6.6.1) 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, it should be recognized that the particle size of a bulk sample could 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 EN 13890 (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 part of ISO 15202 if it can be demonstrated that its analytical recovery meets the requirements of EN 13890.

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

Prior to opening filter transport cassettes or samplers, consider the possibility that particles may have been deposited on the interior walls of the cassette or sampler during the sampling event, and actions that may be required to include such particles in the sample. 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.

NOTE The burning sensation associated with many concentrated acid burns is not immediately apparent on exposure to hydrofluoric acid and might 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 [15] and [16]), 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

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NOTE 1 The above list is based upon the applicability of the sample dissolution procedure reported in References [18], [19] and [20], 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.

B.2.2 Although the sample dissolution method for soluble compounds prescribed in this part of ISO 15202 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^[21].

B.3 Principle

B.3.1 Soluble metal and metalloid compounds are dissolved by treating the filter and collected sample with a suitable leach solution and agitating in a water bath at $37\text{ °C} \pm 2\text{ °C}$ for 60 min.