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

Part 2:

Sample preparation

iTeh STANDARD PREVIEW
(standards.iteh.ai)

*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 —*

<https://standards.iteh.ai/catalog/standards/sist/d336e7c8-d51-4a00-979c-7c78a9b7619f/iso-15202-2-2001>
Partie 2: Préparation des échantillons



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 15202-2:2001

<https://standards.iteh.ai/catalog/standards/sist/d336e7c8-da51-4a00-979c-7c78a9b7619f/iso-15202-2-2001>

© ISO 2001

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

	Page
Foreword.....	iv
Introduction.....	v
1 Scope	1
2 Normative references	2
3 Terms and definitions	2
4 Principle.....	3
5 Requirements.....	4
6 Reactions.....	4
7 Reagents.....	4
8 Laboratory apparatus.....	4
9 Procedure	5
9.1 Soluble metal and metalloid compounds.....	5
9.2 Total metals and metalloids and their compounds.....	5
9.3 Mixed exposure.....	5
10 Special cases	6
10.1 Action to be taken if there is doubt about the effectiveness of the selected sample dissolution method.....	6
10.2 Action to be taken when particles have become dislodged from the filter during transportation	6
11 Laboratory records.....	6
Annex A (informative) Safety precautions to be observed when using hydrofluoric and perchloric acids.....	7
Annex B (normative) Sample dissolution method for soluble metal and metalloid compounds.....	8
Annex C (normative) Sample dissolution using nitric acid and hydrochloric acid on a hotplate.....	14
Annex D (normative) Sample dissolution using hydrofluoric and nitric acids and ultrasonic agitation.....	17
Annex E (normative) Sample dissolution based on the use of sulfuric acid and hydrogen peroxide on a hotplate	20
Annex F (normative) Sample dissolution based on the use of nitric acid and perchloric acid on a hotplate	24
Annex G (normative) Sample dissolution in a closed vessel microwave digestion system.....	28
Annex H (normative) Action to be taken when there is visible, undissolved, particulate material after sample dissolution	33
Bibliography	37

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 3.

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 part of ISO 15202 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

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*

ITeH STANDARD PREVIEW
(standards.iteh.ai)
ISO 15202-2:2001
<https://standards.iteh.ai/catalog/standards/sist/d336e7c8-da51-4a00-979c-7c78a9b7619f/iso-15202-2-2001>

Annexes B to H form a normative part of this part of ISO 15202. Annex A is for information only.

Introduction

The health of workers in many industries is at risk through exposure by inhalation to 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 the 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.

This part of ISO 15202 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.

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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
<https://standards.iteh.ai/catalog/standards/sist/d336e7c8-da51-4a00-979c-7c78a9b7619f/iso-15202-2-2001>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 15202-2:2001

<https://standards.iteh.ai/catalog/standards/sist/d336e7c8-da51-4a00-979c-7c78a9b7619f/iso-15202-2-2001>

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

Part 2: Sample preparation

1 Scope

1.1 This part of ISO 15202 specifies a number of alternative 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.

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) and inductively coupled plasma mass spectrometry (ICP-MS).

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 G 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 reference [1] in the bibliography) 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, since mercury vapour is not collected using the sampling method specified in ISO 15202-1.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 15202. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 15202 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3585:1998, *Borosilicate glass 3.3 — Properties.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 8655-1:—¹⁾, *Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations.*

ISO 8655-2:—¹⁾, *Piston-operated volumetric apparatus — Part 2: Piston pipettes.*

ISO 8655-5:—¹⁾, *Piston-operated volumetric apparatus — Part 5: Dispensers.*

ISO 8655-6:—¹⁾, *Piston-operated volumetric apparatus — Part 6: Gravimetric test methods.*

ISO 11174:1996, *Workplace air — Determination of particulate cadmium and cadmium compounds — Flame and electrothermal atomic absorption spectrometric method.*

ISO 15202-1:2000, *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 1: Sampling.*

ISO 15202-3:—¹⁾, *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 3: Analysis.*

EN 13890:—¹⁾, *Workplace atmospheres — 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

analytical recovery

ratio of the mass of analyte measured when a sample is analysed to the known mass of analyte in that sample, expressed as a percentage

3.2

exposure

(by inhalation)

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

3.3

limit value

reference figure for concentration of a chemical agent in air

1) To be published

3.4**measuring procedure**

procedure for sampling and analysing one or more chemical agents in air, including storage and transportation of the sample

3.5**sampling instrument****sampler**

device for collecting airborne particles

NOTE Instruments used to collect airborne particles are frequently referred to by a number of other terms, e.g. sampling heads, filter holders, filter cassettes etc.

3.6**sample dissolution**

process of obtaining a solution containing the analytes of interest from a sample

NOTE This may or may not involve complete dissolution of the sample.

3.7**sample preparation**

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

3.8**sample solution**

solution prepared by the process of sample dissolution, but needing to be subjected to further operations in order to produce a test solution which is ready for analysis

3.9**test solution**

solution prepared by the process of sample dissolution and subjected to any further operations required to bring it into a state in which it is ready for analysis

3.10**workplace**

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

[EN 1540:1998] [2]

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 G, 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 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.

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^[3] 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 clause 10).

7 Reagents

Details of reagents that are required for individual methods are given in annexes B through H.

During the analysis, use only reagents of analytical grade and only water as specified in 7.1.

7.1 Water, complying with the requirements for ISO 3696 grade 2 water (electrical conductivity less than 0,1 mS/m and resistivity greater than 0,01 M Ω ·m at 25 °C).

NOTE 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, ρ about 1,42 g/ml, about 70 % (m/m).

The concentration of the metals and metalloids of interest shall be less than 0,1 μ g/ml.

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

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 tap water to contaminate the contents of the bottle. When 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 is required for individual methods are given in annexes B through H.

Ordinary laboratory apparatus and

8.1 Disposable gloves, impermeable, 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, made of borosilicate glass 3.3 complying with the requirements of ISO 3585, 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).

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

8.3 Forceps, flat-tipped, for unloading filters from samplers or from filter transport cassettes.

8.4 Piston-operated volumetric instruments, complying with the requirements of ISO 8655-1 and tested in accordance with ISO 8655-6; pipettors complying with the requirements of ISO 8655-2 and dispensers complying with the requirements of ISO 8655-5, for dispensing leach solution, acids etc.

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

NOTE 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 and/or metalloid compounds, 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 and/or metalloids 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 sample dissolution methods for total metals and metalloids and their compounds prescribed in annexes C through G, and compare results with the limit value for the soluble metals and/or metalloids concerned.

NOTE The methods prescribed in annexes C through G are not specific for soluble metal and/or metalloid compounds. 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 the results are required for comparison with limit values for total metals and/or metalloids and their compounds, select a suitable sample dissolution method from those specified in annexes C through G. Take into consideration the applicability of each method for dissolution of the metals and metalloids of interest from materials which 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 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 frequently much more easily dissolved than bulk amounts.

10.1.2 If the analytical recovery is less than the minimum acceptable value prescribed in EN 13890, 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, consider whether to discard the sample as invalid or whether to wash the internal surfaces of the filter transport cassette or sampler into the sample dissolution vessel in order to recover the material concerned.

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 1 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.

NOTE 2 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 whilst 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.

(standards.iteh.ai)

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.

A.2.3 Perform sample digestions 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.