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

Part 3:

Analysis

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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 —*

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Partie 3: Analyse



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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 15202-3 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*

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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, etc.

This part of ISO 15202 specifies a generic method for determination of the mass concentration of metals and metalloids in workplace air using inductively coupled plasma atomic emission spectrometry (ICP-AES) and gives requirements and test methods for analysis of sample solutions.

Part 1 of ISO 15202 gives details of relevant international, European and national standards which specify characteristics, performance requirements and test methods relating to sampling equipment; augments guidance provided elsewhere on assessment strategy and measurement strategy; and specifies a method for collecting samples of airborne particulate matter for subsequent chemical analysis.

Part 2 of ISO 15202 describes a number of procedures for preparing sample solutions for analysis by ICP-AES.

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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 3: Analysis

1 Scope

This part of ISO 15202 specifies a procedure for the use of inductively coupled plasma atomic emission spectrometry for analysing test solutions prepared as prescribed in ISO 15202-2 from samples of airborne particulate matter collected as specified in ISO 15202-1. Method development, performance checks and a routine analysis method are specified.

This part of ISO 15202 is applicable for the assessment of workplace exposure to metals and metalloids for comparison with limit values (see e.g. EN 689^[1], ASTM E 1370^[2], etc.).

The following is a non-exclusive list of metals and metalloids for which limit values have been set (see Reference [3]), and for which one or more of the sample dissolution methods specified in ISO 15202-2 and the analytical procedure described in this part of ISO 15202 are applicable. However, there is no information available on the effectiveness of any of the sample dissolution methods specified in ISO 15202-2 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	

NOTE ISO 15202 is not applicable to determination of elemental mercury, since mercury vapour is not collected using the sampling method specified in ISO 15202-1.

The procedure is suitable for assessment of exposure against the long-term exposure limits for most of the metals and metalloids listed above when sampling at a typical flow rate of 2 l·min⁻¹ for sampling times in the range 30 min to 8 h and for assessment of exposure against the short-term exposure limits, where applicable (see 10.4).

The procedure suffers from no significant spectral interferences (see 10.5), provided that suitable analytical wavelengths are used. However, inaccurate background correction and/or inadequate matrix-matching can adversely affect results.

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 648:1977, *Laboratory glassware — One-mark pipettes*

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

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

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

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric methods for the determination of measurement error*

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 15202-2:2001, *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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3 Terms, definitions and abbreviated terms

For the purposes of this document, the following terms and definitions apply.

3.1 General definitions

3.1.1

chemical agent

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

3.1.2

measuring procedure

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

3.1.3

time-weighted average concentration

TWA concentration

concentration of a chemical agent in the atmosphere, averaged over the reference period

NOTE A more detailed discussion of TWA concentrations has been published by the American Conference of Government Industrial Hygienists^[3].

3.1.4

limit value

reference figure for concentration of a chemical agent in air

NOTE An example is the Threshold Limit Value[®] (TLV) for a given substance in workplace air, as established by the ACGIH^[3].

3.1.5

reference period

specified period of time stated for the limit value of a specific chemical agent

NOTE Examples of limit values for different reference periods are short-term and long-term exposure limits, such as those established by the ACGIH^[3].

3.1.6

workplace

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

[EN 1540^[4]]

3.2 Analytical definitions

3.2.1

blank solution

solution prepared by taking a reagent blank, laboratory blank or field blank through the same procedure used for sample dissolution

NOTE A blank solution might need to be subjected to further operations, e.g. addition of an internal standard, if the sample solutions are subjected to such operations in order to produce test solutions that are ready for analysis.

3.2.2

calibration blank solution

calibration solution prepared without the addition of any stock standard solution or working standard solution

NOTE The concentration of the analyte(s) of interest in the calibration blank solution is taken to be zero.

3.2.3

calibration solution

solution prepared by dilution of the stock standard solution(s) or working standard solution(s), containing the analyte(s) of interest at a concentration(s) suitable for use in calibration of the analytical instrument

NOTE The technique of matrix-matching is normally used when preparing calibration solutions.

3.2.4

field blank

filter that is taken through the same handling procedure as a sample, except that it is not used for sampling, i.e., it is loaded into a sampler, transported to the sampling site and then returned to the laboratory for analysis

3.2.5

laboratory blank

unused filter, taken from the same batch used for sampling, that does not leave the laboratory

3.2.6

linear dynamic range

range of concentrations over which the calibration curve for an analyte is linear

NOTE The linear dynamic range extends from the detection limit to the onset of calibration curvature.

3.2.7

reagent blank

solution containing all reagents used in sample dissolution, in the same quantities used for preparation of laboratory blank, field blank and sample solutions

3.2.8

sample dissolution

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

3.2.9

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

3.2.10

sample solution

solution prepared from a sample by the process of sample dissolution

NOTE A sample solution might need to be subjected to further operations, e.g. dilution and/or addition of an internal standard, in order to produce a test solution that is ready for analysis.

3.2.11

stock standard solution

solution used for preparation of working standard solutions and/or calibration solutions, containing the analyte(s) of interest at a certified concentration(s) traceable to national standards

3.2.12

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

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NOTE 1 "Ready for analysis" includes dilution and/or the addition of an internal standard.

NOTE 2 The blank test solution is the blank solution and the sample test solution is the sample solution if these solutions are not subjected to any further operations before analysis.

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3.2.13

working standard solution

solution, prepared by dilution of the stock standard solution(s), that contains the analyte(s) of interest at a concentration(s) better suited to preparation of calibration solutions than the concentration(s) of the analyte(s) in the stock standard solution(s)

3.3 ICP-AES definitions

3.3.1

axial plasma
end-on plasma

plasma that is viewed end-on by the optical detection system

3.3.2

background correction

process of correcting the intensity at an analytical wavelength for the intensity due to the underlying spectral background

3.3.3

background equivalent concentration

concentration of an analyte that results in an emission signal of an intensity equivalent to the background emission signal at the analytical wavelength

3.3.4

corrosion-resistant sample introduction system

sample introduction system that features a nebulizer, spray chamber and torch injector tube that are resistant to corrosion by hydrofluoric acid

3.3.5**excitation interference**

matrix interference that manifests itself as a change in sensitivity due to a change in plasma conditions when the matrix of a calibration or test solution is introduced into the plasma

3.3.6**ICP torch**

device used to support and introduce sample into an ICP discharge

NOTE An ICP torch usually consists of three concentric tubes, the outer two usually made from quartz.

3.3.7**inductively coupled plasma****ICP**

high-temperature discharge generated in flowing argon by an alternating magnetic field induced by a radio-frequency (RF) load coil that surrounds the tube carrying the gas

3.3.8**injector****injector tube****centre tube**

innermost tube of an ICP torch, through which the sample aerosol is introduced to the plasma

NOTE The injector is usually made of quartz or ceramic material.

3.3.9**inner argon flow****nebulizer argon flow****sample argon flow**

flow of argon gas that is directed through the nebulizer and carries the sample aerosol through the injector and into the plasma

NOTE The inner argon gas flow rate is typically $0,5 \text{ l}\cdot\text{min}^{-1}$ to $2,0 \text{ l}\cdot\text{min}^{-1}$.

3.3.10**inter-element correction****interference correction**

spectral interference correction technique in which emission contributions from interfering elements that emit at the analyte wavelength are subtracted from the apparent analyte emission after measuring the interfering element concentrations at other wavelengths

3.3.11**intermediate argon flow****auxiliary argon flow**

flow of argon gas that is contained between the intermediate and centre (injector) tubes of an ICP torch

NOTE The intermediate argon gas flow rate is typically $0 \text{ l}\cdot\text{min}^{-1}$ to $2,0 \text{ l}\cdot\text{min}^{-1}$.

3.3.12**internal standard****reference element**

non-analyte element, present in all solutions analysed, the signal from which is used to correct for matrix interferences or improve analytical precision

3.3.13**internal standardization****reference element technique**

technique that uses the signal from an internal standard to correct for matrix interferences

3.3.14

load coil

length of tubing wound around the end of an ICP torch and connected to the radio-frequency (RF) generator, used to inductively couple energy from the RF generator to the plasma discharge

3.3.15

matrix interference

matrix effect

non-spectral interference

interference of a non-spectral nature caused by a difference between the matrix of the calibration and test solutions

3.3.16

matrix-matching

technique used to minimize the effect of matrix interferences on analytical results, involving the preparation of calibration solutions in which the concentrations of acids and other major solutes are matched with those in the test solutions

3.3.17

nebulizer

device used to create an aerosol from a liquid

3.3.18

outer argon flow

plasma argon flow

coolant argon flow

flow of argon gas that is contained between the outer and intermediate tubes of an ICP torch

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NOTE The outer argon flow is typically 7 l·min⁻¹ to 15 l·min⁻¹.

3.3.19

pneumatic nebulizer

nebulizer that uses high-speed gas flows to create an aerosol from a liquid

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3.3.20

radial plasma

plasma that is viewed from the side by the optical detection system

3.3.21

spray chamber

device, placed between a nebulizer and an ICP torch, whose function is to separate out aerosol droplets according to their size, so that only very fine droplets pass into the plasma and large droplets are drained or pumped to waste

3.3.22

spectral interference

interference caused by the emission from a species other than the analyte of interest

3.3.23

transport interference

transport effect

matrix interference caused by a difference in viscosity, surface tension or density between the calibration and test solutions that produces a change in nebulizer efficiency and hence in the amount of analyte reaching the plasma

NOTE A transport interference can be due to a difference in dissolved solids, type and concentration of acid, etc., between the calibration and the test solutions.

3.3.24**ultrasonic nebulizer**

nebulizer in which the aerosol is created by flowing a liquid across a surface that is oscillating at an ultrasonic frequency

3.3.25**viewing height****observation height**

(radial plasma) position in a radial plasma from where the emission measured originates

NOTE The viewing height is generally given as the distance, in millimetres, above the load coil.

3.3.26**x-y centring**

(axial plasma) horizontal and vertical adjustment of an axial plasma to establish optimum viewing conditions, such that only emission from the central channel of the plasma is measured

3.4 Statistical terms**3.4.1****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

3.4.2**bias**

consistent deviation of the results of a measurement process from the true value of the air quality characteristic itself

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NOTE Adapted from ISO 6879^[5]. [ISO 15202-3:2004](https://standards.iteh.ai/catalog/standards/sist/184060c7-5ccb-410f-9ea8-97adb8decc5b/iso-15202-3-2004)

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3.4.3**overall uncertainty**

(of a measuring procedure or of an instrument) quantity used to characterize as a whole the uncertainty of a result given by an apparatus or measuring procedure.

NOTE The overall uncertainty is calculated based on a combination of bias and precision, usually in accordance with Equation (1) and is expressed as a percentage:

$$\frac{|\bar{x} - x_{\text{ref}}| + 2s}{x_{\text{ref}}} \times 100 \quad (1)$$

where

- \bar{x} is the mean value of results of a number (n) of repeated measurements;
- x_{ref} is the true or accepted reference value of concentration;
- s is the standard deviation of the measurements.

[EN 482^[6]]

3.4.4**precision**

closeness of agreement of results obtained by applying the method several times under prescribed conditions

NOTE Adapted from ISO 6879^[5].