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DPC: 24/30478891 DC

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Date: 04 September 2024
Origin: International

Latest date for receipt of comments: 28 October 2024

Project No. 2023/01929

Responsible committee: EH/2/2 Work place atmospheres

Interested committees:

Title: Draft BS ISO 30011 Workplace air. Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma mass spectrometry.

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

For national standards, comments will be reviewed by the relevant UK national committee and the resulting standards published as a British Standard.

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Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

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Date: xx/xx/20xx	Document: ISO/DIS xxxx

1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex **(e.g. 3.1)**	Paragraph/ Figure/Table/Note	**Type of comment**	**Commend (justification for change) by the MB**	**Proposed change by the MB**	**Secretariat observations on each comment submitted**
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

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DRAFT International Standard

ISO/DIS 30011

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

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

ICS: 13.040.30

ISO/TC **146**/SC **2**

Secretariat: **ANSI**

Voting begins on:
2024-09-04

Voting terminates on:
2024-11-27

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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).

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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 second edition cancels and replaces the first edition (ISO 30011:2010), which has been technically revised.

The main changes compared to the previous edition are as follows:

— references and definitions have been updated.

— data in Tables 2 and 4 have been updated.

— a new Annex B has been added containing example instrument operating parameters for standard and collision modes. The previous Annexes B and C are now Annexes C and D, respectively.

— a new Annex E has been added containing substrate-specific detection and quantification data.

— additional editorial changes have been made.

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.

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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 taking workplace air measurements. ISO 30011 has been published in order to make available a method for making valid ultra-trace exposure measurements for a wide range of metals and metalloids in use in industry. It is intended for: agencies concerned with health and safety at work; industrial hygienists and other public health professionals; analytical laboratories; and industrial users of metals and metalloids and their workers.

ISO 30011 specifies a method for determination of the mass concentration of metals and metalloids in workplace air using quadrupole inductively coupled plasma mass spectrometry (ICP-MS). For many metals and metalloids, analysis by ICP-MS is advantageous when compared to methods such as inductively coupled plasma atomic emission spectrometry, due to its sensitivity and the presence of fewer spectral interferences.

ISO 30011 gives requirements and test methods for analysis of sample solutions by ICP-MS. Users of ISO 30011 are referred to ISO 15202-1 for collection of samples of airborne particulate matter and to ISO 15202-2 for procedures for preparing sample solutions for analysis by ICP-MS.

It has been assumed in the drafting of ISO 30011 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 mass spectrometry

1 Scope

This document specifies a procedure for the use of quadrupole inductively coupled plasma mass spectrometry (ICP MS) for analysing test solutions prepared from samples of airborne particulate matter collected as specified in ISO 15202-1. Method development, performance checks, and a routine analysis method are specified.

The procedure in this document is for quadrupole ICP-MS, including single-quadrupole instruments and tandem ICP-MS/MS. Other types of ICP-MS (e.g., magnetic sector) are outside of the scope of this document.

Test solutions for analysis by this document are prepared as specified in ISO 15202-2.

This document is applicable to the assessment of workplace exposure to metals and metalloids for comparison with limit values (see e.g. EN 689,[10] ASTM E1370[8]).

This document 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 at least 2 l min^{-1} for sampling times in the range 0,25 h to 8 h and for assessment of exposure against the short-term exposure limits, where applicable.

The procedure is subject to no significant spectral interferences (see A.3), provided that suitable analytical isotopes are used. However, inadequate matrix-matching can adversely affect results.

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 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3585, *Borosilicate glass 3.3 — Properties*

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

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

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

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric reference measurement procedure for the determination of volume*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

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

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ISO 15202-2:2020, *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 2: Sample preparation*

ISO 18158, *Workplace air — Terminology*

ISO 20581, *Workplace air — General requirements for the performance of procedures for the measurement of chemical agents*

ISO 21832:2018, *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 terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1 Analytical definitions

3.1.1
blank solution
solution prepared by taking a reagent blank, laboratory blank or field blank through the same procedure used for sample dissolution

Note 1 to entry: A blank solution might need to be subjected to further operations, such as 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.

Note 2 to entry: Adapted from EN 14902:2005[11], 3.1.2.

3.1.2
calibration blank solution
calibration solution prepared without the addition of any stock standard solution or working standard solution

Note 1 to entry: The concentration of the analyte(s) of interest in the calibration blank solution is taken to be zero.

Note 2 to entry: Adapted from EN 14902:2005[11], 3.1.3.

3.1.3
calibration curve
plot of instrument response versus concentration of standards

Note 1 to entry: Adapted from United States Environmental Protection Agency (Reference [13]).

3.1.4
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) that is suitable for use in calibration of the analytical instrument

Note 1 to entry: The matrix-matching technique is normally used when preparing calibration solutions.

Note 2 to entry: Adapted from EN 14902:2005[11], 3.1.4.

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3.1.5
linear dynamic range
range of concentrations over which the calibration curve for an analyte is linear

Note 1 to entry: The linear dynamic range extends from the detection limit to the onset of calibration curvature.

3.1.6
reagent blank
all reagents used in sample dissolution, in the same quantities used for preparation of blank and sample solutions

Note 1 to entry: The reagent blank is used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample preparation.

3.1.7
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

Note 1 to entry: Adapted from EN 14902:2005[11], 3.1.25.

3.1.8
sample solution
solution prepared from a sample by the process of sample dissolution

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

Note 2 to entry: Adapted from EN 14902:2005[11], 3.1.22.

3.1.9
spiked media blank
media blank that is spiked with a known amount of the analyte(s) of interest

3.1.10
stock standard solution
solution used for preparation of working standard solutions or calibration solutions, containing the analyte(s) of interest at a certified concentration(s) traceable to national standards

Note 1 to entry: Adapted from EN 14902:2005[11], 3.1.26.

3.1.11
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 to entry: “Ready for analysis” includes any required dilution or addition 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 to entry: Adapted from EN 14902:2005[11], 3.1.30.

3.1.12
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 for preparation of calibration solutions than the concentration(s) of the analyte(s) in the stock standard solution(s)

Note 1 to entry: Adapted from EN 14902:2005[11], 3.1.32.

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3.2 ICP-MS definitions

3.2.1
collision cell
chamber in the ion path between mass-to-charge ratio (m/z) separation elements, or between ion source acceleration region and the first analyser, in tandem mass spectrometry in space configurations

Note 1 to entry: See Reference [14].

3.2.2
collision reaction cell
collision cell for removal of interfering ions by ion/neutral reactions in ICP-MS

Note 1 to entry: See Reference [14].

Note 2 to entry: Collision reaction cells make use of kinetic energy dispersion, reaction chemistry or a combination of both, to remove interfering species. A variety of applications of collision reaction cell technology are available.

3.2.3
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 (6.2.6)

3.2.4
ICP torch
device used to support and introduce sample into an ICP discharge

Note 1 to entry: An ICP torch usually consists of three concentric tubes, the outer two usually made from quartz.

3.2.5
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

3.2.6
injector
injector tube
centre tube
innermost tube of an ICP torch, through which the sample aerosol is introduced to the plasma

Note 1 to entry: The injector is usually made of quartz, ceramic material or platinum.

3.2.7
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 1 to entry: The inner argon flow rate is typically 0,5 l min^{-1} to 2 l min^{-1}.

3.2.8
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 1 to entry: The intermediate argon flow rate is typically 0 l min^{-1} to 2 l min^{-1}.

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3.2.9
internal standard
non-analyte element, present in all solutions analysed, the signal from which is used to correct for matrix interferences or improve analytical precision

3.2.10
load coil
length of metal 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

3.2.11
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

3.2.12
matrix-matching
technique used to minimize the effect of matrix interferences on the analytical results, involving the preparation of calibration solutions in which the concentrations of acids and other major solvents and solutes are matched with those in the test solutions

3.2.13
nebulizer
device used to create an aerosol from a liquid

3.2.14
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

Note 1 to entry: The outer argon flow rate is typically 7 l min^{-1} to 15 l min^{-1}.

3.2.15
spectral interference
interference caused by a species other than the analyte of interest; e.g. atomic, polyatomic, or doubly charged ion species

Note 1 to entry: An example of an atomic interference is $^{40}Ar^{+}$ on $^{40}Ca^{+}$. An example of a polyatomic interference is $^{40}Ar^{16}O^{+}$ on $^{56}Fe^{+}$. An example of a doubly charged ion interference is $^{48}Ti^{2+}$ on $^{24}Mg^{+}$ (Reference [14]).

3.2.16
spray chamber
device placed between a nebulizer and an inductively coupled plasma torch whose function is to separate out aerosol droplets in accordance with their size, so that only very fine droplets pass into the plasma and large droplets are drained or pumped to waste

3.2.17
tandem ICP-MS/MS
ICP-MS system with an additional quadrupole prior to the collision reaction cell[22]

Note 1 to entry: Tandem ICP/MS-MS is also known as triple-quadrupole ICP-MS or ICP-QQQ.

3.2.18
tuning
analysis of a solution containing a range of isotopic masses to establish ICP-MS mass-scale accuracy, mass resolution, signal intensity and precision prior to calibration

Note 1 to entry: See Reference [13].

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4 Principle

4.1 Airborne particles containing metals and metalloids are collected using the method specified in ISO 15202-1.

4.2 The collected sample and the filter are then treated to dissolve the metals and metalloids of interest using one of the sample dissolution methods specified in ISO 15202-2.

4.3 The resultant solutions are analysed for the metals and metalloids of interest using single-quadrupole ICP-MS or tandem ICP-MS/MS.

5 Requirements

The measuring procedure as a whole (specified in ISO 15202-1, ISO 15202-2 and this document) shall comply with applicable requirements of ISO 20581, ISO 21832, and with any relevant national standard that specifies performance requirements for procedures for measuring chemical agents in workplace air.

6 Reagents

6.1 Water, from a 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).

6.2 Mineral acids, concentrated.

6.2.1 General. Various types of mineral acid (6.2.2 to 6.2.6) are required for preparation of matrix-matched calibration solutions (see 6.4.2).

The concentration of the metals and metalloids of interest shall be less than 0,1 mg l^{-1}.

Mineral acids of higher purity may be required in order to obtain adequate detection limits for some metals and metalloids, e.g. beryllium.

6.2.2 Nitric acid, concentrated, ρ_{HNO3} ≈1,42 g ml^{-1}, w_{HNO3} ≈ 70 % mass fraction.

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 glasses, etc.) when working with concentrated or dilute nitric acid.

6.2.3 Perchloric acid, concentrated, ρ_{HClO4} ≈ 1,67 g ml^{-1}, w_{HClO4} ≈ 70 % mass fraction.

WARNING — Concentrated perchloric acid is corrosive and oxidizing and perchloric 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 glasses, etc.) when working with concentrated or dilute perchloric acid. For safety reasons, use perchloric acid in limited quantities.

For analysis of metals and metalloids that are subject to interference from polyatomic ions containing chlorine, the use of perchloric acid is not recommended unless a collision reaction cell is used.

6.2.4 Hydrochloric acid, concentrated, ρ_{HCl} ≈ 1,18 g ml^{-1}, w_{HCl} ≈ 36 % mass fraction.

WARNING — Concentrated hydrochloric acid is corrosive and oxidizing and hydrochloric 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 glasses, etc.) when working with concentrated or dilute hydrochloric acid. Handle open vessels containing concentrated hydrochloric

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