
**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*

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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 30011 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

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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 International Standard 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.

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

This International Standard 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]).

The following is a non-exclusive list of metals and metalloids for which limit values have been set (see Reference [15]) and for which one or more of the sample preparation methods specified in ISO 15202-2 and the analytical procedure described in this International Standard are applicable. However, there is no information available on the effectiveness of any of these sample preparation methods for those elements listed in italics.

aluminium	caesium	lead	platinum	tungsten
antimony	chromium	lithium	potassium	uranium
arsenic	cobalt	magnesium	rhodium	vanadium
barium	copper	manganese	selenium	yttrium
beryllium	gallium	<i>mercury</i>	silver	zinc
bismuth	<i>germanium</i>	molybdenum	sodium	zirconium
boron	hafnium	nickel	tellurium	
cadmium	indium	<i>niobium</i>	thallium	
calcium	iron	phosphorus	tin	

This International Standard 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 0,5 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 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 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: 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, *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 1: Sampling*

ISO 15202-2:—, *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 and definitions

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

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3.1 General definitions

3.1.1

breathing zone

⟨general definition⟩ space around the worker's face from where he or she takes his or her breath

NOTE Adapted from EN 1540:—^[11], 2.4.5.

3.1.2

breathing zone

⟨technical definition⟩ hemisphere (generally accepted to be 0,3 m in radius) extending in front of the human face, centred on the midpoint of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head, and the larynx

NOTE 1 The definition is not applicable when respiratory protective equipment is used.

NOTE 2 Adapted from EN 1540:—^[11], 2.4.5.

3.1.3

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^[16], Art. 2(a)]

3.1.4**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:—^[11], 2.4.1.

3.1.5**occupational exposure 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^[16], Art. 2(d)]

EXAMPLES Threshold Limit Values® (TLVs) established by the ACGIH (Reference [15]) and Indicative Occupational Exposure Limit Values (IOELVs) promulgated by the European Commission (Council Directive 2006/15/EC^[17]).

3.1.6**measuring procedure for the sampling and analysis of chemical agents in air**

measurement procedure for the sampling and analysis of chemical agents in air
set of operations, described specifically, used 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 ISO/IEC Guide 99:2007^[4].

3.1.7**reference period**

specified period of time for which the limit value of a chemical agent applies

NOTE 1 The reference period is usually 8 h for long-term measurements and 15 min for short-term measurements.

NOTE 2 Adapted from EN 1540:—^[11], 2.4.7. <https://standards.iteh.ai/catalog/standards/sist/ef00dc90-6cf5-4def-8ab2-c547efb76d/iso-30011-2010>

3.1.8**workplace**

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

[EN 1540:—^[11], 2.5.2]

3.2 Analytical definitions**3.2.1****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^[13], 3.1.1.

3.2.2**blank solution**

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

NOTE 1 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 Adapted from EN 14902:2005^[13], 3.1.2.

3.2.3

calibration blank solution

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

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

NOTE 2 Adapted from EN 14902:2005^[13], 3.1.3.

3.2.4

calibration curve

plot of instrument response versus concentration of standards

NOTE Adapted from United States Environmental Protection Agency (Reference [18]).

3.2.5

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

NOTE 2 Adapted from EN 14902:2005^[13], 3.1.4.

3.2.6

field blank

sampling medium 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

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3.2.7

instrumental detection limit

IDL
lowest concentration at which the instrumentation can distinguish analyte content from the background generated by a minimal matrix

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NOTE The IDL can be determined from blank, acidified, deionized, or ultrapure water as the matrix and from the same calculation methods used to determine a method detection limit.

3.2.8

laboratory blank

media blank
unused sampling medium, taken from the same batch used for sampling, that does not leave the laboratory

3.2.9

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.

3.2.10

method detection limit

MDL
minimum concentration of an analyte that can be reported with 99 % confidence that the value is above zero

3.2.11

quantification limit

quantitation limit
QL
minimum concentration of an analyte that can be measured with acceptable precision

3.2.12**reagent blank**

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

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

3.2.13**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 Adapted from EN 14902:2005^[13], 3.1.25.

3.2.14**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

NOTE Adapted from EN 14902:2005^[13], 3.1.24.

3.2.15**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 of an internal standard(s), in order to produce a test solution.

NOTE 2 Adapted from EN 14902:2005^[13], 3.1.22.

3.2.16**sampling medium**

sampling substrate

collection medium

collection substrate

medium on which airborne chemical or biological agents are collected for subsequent analysis

EXAMPLES Filters and polyurethane foams.

3.2.17**spiked media blank**

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

3.2.18**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 Adapted from EN 14902:2005^[13], 3.1.26.

3.2.19**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 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^[13], 3.1.30.

3.2.20

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 Adapted from EN 14902:2005^[13], 3.1.32.

3.3 ICP-MS definitions

3.3.1

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 See Reference [19].

3.3.2

collision reaction cell

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

NOTE 1 See Reference [20].

NOTE 2 Collision reaction cells make use of kinetic energy dispersion, reaction chemistry or a combination of both, to remove interfering species. A variety of reaction chemistry techniques are available.

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3.3.3

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

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3.3.4

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.

3.3.5

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

3.3.6

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, ceramic material or platinum.

3.3.7

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 The inner argon flow rate is typically 0,5 l min⁻¹ to 2 l min⁻¹.

3.3.8**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 The intermediate argon flow rate is typically 0 l min⁻¹ to 2 l min⁻¹.**3.3.9****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

3.3.10**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

3.3.11**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

3.3.12**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

3.3.13**nebulizer**

device used to create an aerosol from a liquid

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plasma argon flow

coolant argon flow

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

NOTE The outer argon flow rate is typically 7 l min⁻¹ to 15 l min⁻¹.**3.3.15****spectral interference**

isobaric interference caused by a species other than the analyte of interest

NOTE Interferences can involve an atomic, polyatomic or doubly charged ion species. An example of an atomic interference is ⁴⁰Ar⁺ on ⁴⁰Ca⁺. An example of a polyatomic interference is ⁴⁰Ar¹⁶O⁺ on ⁵⁶Fe⁺. An example of a doubly charged ion interference is ⁴⁸Ti²⁺ on ²⁴Mg⁺ (Reference [21]).**3.3.16****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