



Standard Test Method for Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma–Mass Spectrometry¹

This standard is issued under the fixed designation D 7439; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This standard test method specifies a procedure for sample preparation and analysis of airborne particulate matter for the content of metals and metalloids in workplace air samples using inductively coupled plasma–mass spectrometry (ICP-MS).

1.2 This standard test method assumes that samples will have been collected in accordance with Test Method D 7035.

1.3 This standard test method should be used by analysts experienced in the use of ICP-MS, the interpretation of spectral and matrix interferences and procedures for their correction.

1.4 This standard test method specifies a number of alternative methods for preparing test solutions from samples of airborne particulate matter. One of the specified sample preparation methods is applicable to the measurement of soluble metal or metalloid compounds. Other specified methods are applicable to the measurement of total metals and metalloids.

1.5 It is the user's responsibility to ensure the validity of the standard method for filters of untested matrices.

1.6 Table 1 provides a non-exclusive list of metals and metalloids for which one or more of the sample dissolution methods specified in this document is applicable.

1.7 This standard test method is not applicable to compounds of metals and metalloids that are present in the gaseous or vapor state.

1.8 No detailed operating instructions are provided because of differences among various makes and models of suitable ICP-MS instruments. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument. This test method does not address comparative accuracy of different devices or the precision between instruments of the same make and model.

1.9 The values stated in SI units are to be regarded as standard.

1.10 This standard test method contains notes that are explanatory and are not part of the mandatory requirements of the method.

1.11 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- D 1193 Specification for Reagent Water
 - D 1356 Terminology Relating to Sampling and Analysis of Atmospheres
 - D 4185 Practice for Measurement of Metals in Workplace Atmospheres by Flame Atomic Absorption Spectrophotometry
 - D 6785 Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry
 - D 7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
 - D 7202 Test Method for Determination of Beryllium in the Workplace Using Field-Based Extraction and Fluorescence Detection
 - E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
 - E 1613 Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques
- ### 2.2 ISO and European Standards:
- ISO 1042 Laboratory Glassware—One-Mark Volumetric Flasks³
 - ISO 3585 Borosilicate Glass 3.3—Properties³
 - ISO 8655 Piston-Operated Volumetric Apparatus (6 parts)³

¹ This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

TABLE 1 Applicable Metals and Metalloids

Element	Symbol	CASRN ^A	Element	Symbol	CASRN ^A	Element	Symbol	CASRN ^A
Aluminum	Al	7429-90-5	Antimony	Sb	7440-36-0	Arsenic	As	7440-38-2
Barium	Ba	7440-39-3	Beryllium	Be	7440-41-7	Bismuth	Bi	7440-69-9
Boron	B	7440-42-8	Cadmium	Cd	7440-43-9	Calcium	Ca	7440-70-2
Cesium	Cs	7440-46-2	Chromium	Cr	7440-47-3	Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8	Gallium	Ga	7440-55-3	<i>Germanium^B</i>	<i>Ge</i>	<i>7440-56-4</i>
Hafnium	Hf	7440-58-6	Indium	In	7440-74-6	Iron	Fe	7439-89-6
Lead	Pb	7439-92-1	Lithium	Li	7439-93-2	Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5	<i>Mercury^B</i>	<i>Hg</i>	<i>7439-97-6</i>	Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0	<i>Niobium^B</i>	<i>Nb</i>	<i>7440-03-1</i>	Phosphorus	P	7723-14-0
Platinum	Pt	7440-06-4	Potassium	K	7440-09-7	Rhodium	Rh	7440-16-6
Selenium	Se	7782-49-2	Silver	Ag	7440-22-4	Sodium	Na	7440-23-5
Tellurium	Te	13494-80-9	Thallium	Tl	7440-28-0	Tin	Sn	7440-31-5
Tungsten	W	7440-33-7	Uranium	U	7440-61-1	Vanadium	V	7440-62-2
Yttrium	Y	7440-65-5	Zinc	Zn	7440-66-6	Zirconium	Zr	7440-67-7

^ACASRN = Chemical Abstracts Service Registry Number

^B For the elements in italics, there is insufficient information available on the effectiveness of the sample dissolution procedures in Annex A1 through Annex A4.

ISO 15202 Workplace Air—Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (3 parts)³

ISO 17294 Water Quality—Application of Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (2 parts)³

EN 1540 Workplace Atmospheres—Terminology³

3. Terminology

3.1 *Definitions*—For definitions of other terms used in this standard test method, refer to Terminology **D 1356**.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *analytical recovery*—ratio of the mass of analyte measured to the known mass of analyte in the sample, expressed as a percentage. **D 6785**

3.2.2 *batch*—a group of field or quality control (QC) samples that are collected or processed together at the same time using the same reagents and equipment. **E 1613**

3.2.3 *blank solution*—solution prepared by taking a reagent blank, laboratory blank or field blank through the same procedure used for sample dissolution. **ISO 15202**

3.2.3.1 *Discussion*—A blank solution may 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.

3.2.4 *calibration blank solution*—calibration solution prepared without the addition of any stock standard solution or working standard solution. **ISO 15202**

3.2.4.1 *Discussion*—The concentration of the analyte(s) of interest in the calibration blank solution is taken to be zero.

3.2.5 *calibration curve*—a plot of instrument response versus concentration of standards (**1**).⁴

3.2.6 *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. **ISO 15202**

3.2.6.1 *Discussion*—The technique of matrix matching is normally used when preparing calibration solutions.

3.2.7 *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. **EN 1540/ISO 15202**

3.2.8 *collision/reaction system*—any system, such as a transmission collision cell, to which an oscillating radio frequency potential is applied that is used for charge exchange neutralization of interfering ions in inductively coupled plasma mass spectrometry (**2**).

3.2.8.1 *Discussion*—Some collision systems also have one or more reaction modes that can further reduce selected interferences.

3.2.9 *continuing calibration blank (CCB)*—a solution containing no analyte added, that is used to verify blank response and freedom from carryover. **E 1613**

3.2.9.1 *Discussion*—The CCB must be analyzed after the CCV (see **3.2.10**). The measured concentration of the CCB should not exceed 10 % of the applicable occupational exposure limit or minimum level of concern.

3.2.10 *continuing calibration verification (CCV)*—a solution (or set of solutions) of known analyte concentration used to verify freedom from excessive instrumental drift; the concentration is to be near the mid-range of a linear calibration curve. **E 1613**

3.2.10.1 *Discussion*—The CCV must be matrix matched to the acid content present in sample digestates or extracts. The CCV must be analyzed before and after all samples and at a frequency of not less than every ten samples. The measured value is to fall within $\pm 10\%$ of the known value.

3.2.11 *field blank*—sampling media (for example, an air filter) that is taken through the same handling procedure as a sample, except that no sample is collected (that is, no air is purposely drawn through the sampler), and is then returned to the laboratory for analysis. **ISO 15202/D 7035**

3.2.11.1 *Discussion*—Analysis results from field blanks provide information on the analyte background level in the sampling media, combined with the potential contamination experienced by samples collected within the batch resulting from handling.

3.2.12 *inductively coupled plasma (ICP)*—a high-temperature discharge generated by a flowing conductive gas,

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

normally argon, through a magnetic field induced by a load coil that surrounds the tubes carrying the gas. **ISO 15202**

3.2.13 *inductively coupled plasma (ICP) torch*—a device used to support and introduce sample into an ICP discharge. **ISO 15202**

3.2.14 *initial calibration blank (ICB)*—a standard containing no analyte that is used for the initial calibration. **E 1613**

3.2.14.1 *Discussion*—The ICB must be matrix matched to the acid content of sample extracts and digestates. The ICB must be measured during and after calibration. The measured concentration of the ICB should not exceed 10 % of the applicable occupational exposure limit or minimum level of concern.

3.2.15 *initial calibration verification (ICV)*—a solution (or set of solutions) of known analyte concentration used to verify calibration standard levels; the concentration of analyte is to be near the mid-range of the calibration curve that is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. **E 1613**

3.2.15.1 *Discussion*—The ICV must be matrix matched to the acid content of sample extracts or digestates. The ICV must be measured after calibration and before measuring any sample digestates or extracts. The measured value is to fall within $\pm 10\%$ of the known value.

3.2.16 *instrumental detection limit (IDL)*—the lowest concentration at which the instrumentation can distinguish analyte content from the background generated by a minimal matrix. **E 1613**

3.2.16.1 *Discussion*—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 (see 3.2.25).

3.2.17 *instrumental QC standards*—these provide information on measurement performance during the instrumental analysis portion of the overall analyte measurement process. They include CCBs, CCVs, ICB, and ICVs. **E 1613**

3.2.18 *internal standard*—non-analyte element, present in all solutions analyzed, the signal from which is used to correct for matrix interferences or improve analytical precision. **ISO 15202**

3.2.18.1 *Discussion*—The internal standard is added in known and constant amount(s) to all analyzed solutions. This is used to correct for instrument drift and some matrix effects by measuring the relative instrument response of the internal standard(s) to the other analytes that are components of the same solution. The element(s) selected for use as an internal standard must be initially absent from the sample solution.

3.2.19 *laboratory blank*—unused sample media (for example, an air filter), taken from the same batch used for sampling, that does not leave the laboratory. **ISO 15202**

3.2.20 *limit value*—reference figure for concentration of a chemical agent in air. **ISO 15202**

3.2.20.1 *Discussion*—An example of a limit value would be a Permissible Exposure Limit (PEL) such as those established by the U.S. Occupational Safety and Health Administration.

3.2.21 *linear dynamic range*—the range of concentrations over which the calibration curve for an analyte is linear. It extends from the detection limit to the onset of calibration

curvature.

ISO 15202

3.2.22 *load coil*—a length of metal tubing (typically copper) which is wound around the end of an inductively coupled plasma torch and connected to the radio frequency generator.

ISO 15202

3.2.23 *matrix interference*—interference of a non-spectral nature which is caused by the sample matrix. **ISO 15202**

3.2.24 *matrix matching*—a technique used to minimize the effect of the test solution matrix on the analytical results.

ISO 15202

3.2.24.1 *Discussion*—Matrix matching involves preparing calibration solutions in which the concentrations of acids and other major solvents and solutes are matched with those in the test solutions.

3.2.25 *method detection limit (MDL)*—the minimum concentration of an analyte that can be reported with a 99 % confidence that the value is above zero. **D 1356**

3.2.25.1 *Discussion*—The MDL is also known as the limit of detection (LOD) (1).

3.2.26 *method quantitation limit (MQL)*—the minimum concentration of an analyte that can be measured with acceptable precision. **D 7035**

3.2.26.1 *Discussion*—The MQL is also known as the limit of quantitation (1).

3.2.27 *nebulizer*—a device used to create an aerosol from a liquid. **ISO 15202**

3.2.28 *reagent blank*—solution containing all reagents used in sample dissolution, in the same quantities used for preparation of blank and sample solutions. **ISO 15202**

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

3.2.29 *sample dissolution*—the process of obtaining a solution containing the analyte(s) of interest from a sample. This may or may not involve complete dissolution of the sample.

D 6785/ISO 15202

3.2.30 *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. **ISO 15202**

3.2.31 *sample solution*—solution prepared from a sample by the process of sample dissolution. **ISO 15202**

3.2.32 *spectral interference*—an isobaric interference caused by a species other than the analyte of interest.

3.2.32.1 *Discussion*—Spectral interferences may involve an atomic, polyatomic, or doubly-charged ion species. An example of an atomic interference is $^{40}\text{Ar}^+$ on $^{40}\text{Ca}^+$. An example of a polyatomic interference is $^{40}\text{Ar}^{16}\text{O}^+$ on $^{56}\text{Fe}^+$. An example of a doubly-charged ion interference is $^{48}\text{Ti}^{2+}$ on $^{24}\text{Mg}^+$ (3).

3.2.33 *spiked reagent blank*—a reagent blank aliquot that is spiked with a known amount of analyte.

3.2.33.1 *Discussion*—Analysis results for spiked reagent blanks are used to provide information on the precision and bias of the overall analysis process.

3.2.34 *spiked media blank*—a reagent blank aliquot that includes the sampling media (that is, filter), but includes no actual sample, that is spiked with a known amount of analyte.