This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D4185 − 23

Standard Test Method for Measurement of Metals in Workplace Atmospheres by Flame Atomic Absorption Spectrophotometry[1]

This standard is issued under the fixed designation D4185; 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 test method covers the collection, dissolution, and determination of trace metals in workplace atmospheres, by flame atomic absorption spectrophotometry (FAAS).

1.2 The estimated method detection limits and optimum working concentration ranges for 21 metals are given in Table 1.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* (Specific safety precautionary statements are given in Section 9.)

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D3195 Practice for Rotameter Calibration

D5337 Practice for Setting and Verifying the Flow Rate of Personal Sampling Pumps

D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D8358 Guide for Assessment and Inclusion of Wall Deposits in the Analysis of Single-Stage Samplers for Airborne Particulate Matter

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blank signal, n*—that signal which results from all added reagents and clean sample media prepared and analyzed exactly in the same way as the samples.

3.2.2 *working range for an analytical precision better than 3 %, n*—the range of sample concentrations that will absorb 10 % to 70 % of the incident radiation (0.05 to 0.52 absorbance units).

4. Summary of Test Method

4.1 Workplace air samples are collected in samplers containing filters or filter capsules and are then treated with acid mixtures to destroy the organic matrix and to dissolve the metals present. The analysis is subsequently made by flame atomic absorption spectrophotometry (FAAS).

4.2 Samples and standards are aspirated the flame of an absorption spectrophotometer. A hollow cathode or electrodeless discharge lamp for the metal being determined provides a source of characteristic radiation energy for that particular metal. The absorption of this characteristic energy by the atoms of interest in the flame is related to the concentration of the metal in the aspirated sample. The flame and operating conditions for each element are listed in Table 2.

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[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.

Current edition approved Sept. 1, 2023. Published September 2023. Originally approved in 1990. Last previous edition approved in 2017 as D4185 – 17. DOI: 10.1520/D4185-23.

[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.

TABLE 1 FAAS Method Detection Limits and Optimum Working Concentration for 21 Metals

Element	Method Detection Limit, µg/sample	Optimum Linear Range, mg/m^3	Reference(s)
Ag	0.2	0.5 to 5 (100 L sample)	(1, 2)
Al	2	0.5 to 10 (100 L sample)	(3)
Ba	2	0.13 to 10 (200 L sample)	(4)
Bi	2.5	5 to 300 (400 L sample)	(2)
Ca	0.1	1 to 20 (85 L sample)	(5)
Cd	0.05	0.01 to 2 (250 L sample)	(6)
Co	0.6	0.01 to 0.3 (300 L sample)	(7)
Cr	0.06	0.05 to 2.5 (100 L sample)	(8)
Cu	0.1	0.05 to 50 (400 L sample)	(2)
Fe	0.5	0.3 to 50 (400 L sample)	(2)
In	2	1 to 500 (400 L sample)	(2)
K	0.2	0.2 to 20 (400 L sample)	(2)
Li	0.03	0.04 to 20 (400 L sample)	(2)
Mg	0.01	0.1 to 5 (400 L sample)	(2)
Mn	0.2	0.1 to 30 (400 L sample)	(2)
Na	0.02	0.09 to 10 (400 L sample)	(2)
Ni	0.2	1 to 50 (400 L sample)	(2)
Pb	2.6	0.05 to 1 (200 L sample)	(9)
Sb	8	1 to 10 (400 L sample)	(2)
Tl	3	0.5 to 200 (400 L sample)	(2)
Zn	3	1 to 10 (10 L sample)	(10)

TABLE 2 FAAS Flame and Operating Conditions for Each Element

Element	Type of Flame	Analytical Wavelength, nm	Interferences[A]	Remedy[A]	Reference
Ag	Air-C_2H_2 (oxidizing)	328.1	$I0_3^-$, WO_4^{-2}, MnO_4^{-2}, Cl^-, F^-	[B]	(1, 11)
Al[C]	N_2O-C_2H_2 (reducing)	309.3	ionization, SO_4^{-2}, V	[B,D,E]	(12)
Ba	N_2O-C_2H_2 (reducing)	553.6	ionization, large concentration Ca	[D,F]	(2, 12)
Bi	Air-C_2H_2 (oxidizing)	223.1	none known		
Ca	Air-C_2H_2 (oxidizing)	422.7	ionization (slight) and chemical ionization	[D,E]	(2, 12)
	N_2O-C_2H_2 (reducing)				
Cd	Air-C_2H_2 (oxidizing)	228.8	none known		
Co[C]	Air-C_2H_2 (oxidizing)	240.7	none known		
Cr[C]	Air-C_2H_2 (reducing)	357.9	Fe, Ni, oxidation state of Cr	[B]	(12)
Cu	Air-C_2H_2 (oxidizing)	324.8	none known		
Fe	Air-C_2H_2 (oxidizing)	248.3	high Ni concentration, Si	[B]	(2, 12)
In	Air-C_2H_2 (oxidizing)	303.9	Al, Mg, Cu, Zn, $H_xPO_4^{x-3}$	[B]	(13)
K	Air-C_2H_2 (oxidizing)	766.5	ionization	[D]	(2, 12)
Li	Air-C_2H_2 (oxidizing)	670.8	ionization	[D]	(14)
Mg	Air-C_2H_2 (oxidizing)	285.2	chemical ionization	[D,E]	(2, 12)
	N_2O-C_2H_2 (reducing)				
Mn	Air-C_2H_2 (oxidizing)	279.5	Si		
Na	Air-C_2H_2 (oxidizing)	589.6	ionization	[E]	(2, 12)
Ni	Air-C_2H_2 (oxidizing)	232.0	none known		
Pb	Air-C_2H_2 (oxidizing)	217.0	Ca, high concentration SO_4^{-2}	[B]	(15)
		283.3			
Sb	Air-C_2H_2 (oxidizing)	217.6	Pb, Cu	[G]	(2)
		231.2			
Tl	Air-C_2H_2 (oxidizing)	276.8	none known		
V	N_2O-C_2H_2 (reducing)	318.4	ionization		
Zn	Air-C_2H_2 (oxidizing)	213.9	none known		

[A] High concentrations of silicon in the sample can cause an interference for many of the elements in this table and may cause aspiration problems. No matter what elements are being measured, if large amounts of silica are extracted from the samples, the samples should be allowed to stand for several hours and centrifuged or filtered to remove the silica.
[B] Samples are periodically analyzed by the method of standard additions to check for chemical interferences. If interferences are encountered, determinations must be made by the standard additions method or, if the interferent is identified, it may be added to the standards.
[C] Some compounds of these elements will not be dissolved by the procedure described here. When determining these elements, one should verify that the types of compounds suspected in the sample will dissolve using this procedure (see 12.2).
[D] Ionization interferences are controlled by bringing all solutions to 1000 ppm cesium (samples and standards).
[E] 1000 ppm solution of lanthanum as a releasing agent is added to all samples and standards.
[F] In the presence of very large calcium concentrations (greater than 0.1 %) a molecular absorption from CaOH may be observed. This interference may be overcome by using background corrections when analyzing for barium.
[G] In the presence of high concentrations Pb or Cu, an alternative analytical wavelength of 231.2 nm should be used.

5. Significance and Use

5.1 The health of workers in many industries is at risk through exposure by inhalation to toxic metals. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposures, and this is generally achieved by making workplace air measurements. Exposure to some metal-containing particles has been demonstrated to cause dermatitis, skin ulcers, eye problems, chemical pneumonitis, and other physical disorders (**16**).[3]

5.2 FAAS is capable of quantitatively determining many metals in air samples at the levels required by federal, state, and local occupational health and air pollution regulations. The analysis results can be used for the assessment of workplace exposures to metals in workplace air. The suitability of FAAS for elemental analysis for exposure assessment purposes must be investigated prior to carrying out workplace air sampling, in consideration of relevant occupational exposure limit values (OELVs) for metals of concern.

6. Interferences

6.1 In FAAS the occurrence of interferences is less common than in many other analytical techniques. Interferences can occur, however, and when encountered are corrected for as indicated in the following sections. The known interferences and correction methods for each metal are indicated in Table 2. The methods of standard additions and background monitoring and correction (**11**, **12**, **17**, **18**) are used to identify the presence of an interference. Insofar as possible, the matrix of sample and standard are matched to minimize the possible interference.

6.2 Background or nonspecific absorption can occur from particles produced in the flame which can scatter light and produce an apparent absorption signal. Light scattering may be encountered when solutions of high salt content are being analyzed. They are most severe when measurements are made at shorter wavelengths (for example, below about 250 nm). Background absorption may also occur as the result of the formation of various molecular species which can absorb light. The background absorption can be accounted for by the use of background correction techniques (**17**).

6.3 Spectral interferences are those interferences which result from an atom different from the one being measured that absorbs a portion of the radiation. Such interferences are extremely rare in FAAS. In some cases multielement hollow cathode lamps may cause a spectral interference by having closely adjacent emission lines from two different elements. In general, the use of multielement hollow cathode lamps is discouraged.

6.4 Ionization interference occurs when easily ionized atoms are being measured. The degree to which such atoms are ionized is dependent upon the atomic concentration and the presence of other easily ionized atoms. This interference can be controlled by the addition of a high concentration of another easily ionized element which will buffer the electron concentration in the flame.

6.5 Chemical interferences occur in FAAS when species present in the sample cause variations in the degree to which atoms are formed in the flame, or when different valence states of a single element have different absorption characteristics. Such interferences may be controlled by adjusting the sample matrix or by the method of standard additions (**18**). Also, the use of lanthanum as a releasing element minimizes the interference from the formation of nonvolatile compounds in the flame. Lanthanum forms nonvolatile compounds preferentially with the interferent so that the analyte remains free.

6.6 Physical interferences may result if the physical properties of the samples vary significantly. Changes in viscosity and surface tension can affect the sample aspiration rate and thus cause erroneous results. Sample dilution or the method of standard additions, or both, are used to correct such interferences. High concentrations of silica in the sample can cause aspiration problems. No matter what elements are being determined, if large amounts of silica are extracted from the samples, they shall be allowed to stand for several hours and centrifuged or filtered to remove the silica.

6.7 This procedure describes a generalized method for sample preparation, which is applicable to the majority of samples. There are some relatively rare chemical forms of a few of the elements listed in Table 1 that will not be dissolved by this procedure. If such chemical forms are suspected, results obtained using this procedure shall be compared with those obtained using an appropriately altered dissolution procedure. Alternatively, the results may be compared with values obtained using a technique that does not require dissolving the sample (for example, X-ray fluorescence or neutron activation analysis).

7. Apparatus

7.1 *Sampling Apparatus:*

7.1.1 *Samplers,* containing mixed cellulose ester (MCE) or cellulose nitrate membrane filters, with a pore size of 0.8 µm mounted in a 25 mm or 37 mm diameter, two- or three-piece filter holder. MCE filters attached to cellulose acetate capsules, which are acid-soluble, are also suitable.

NOTE 1—Alternative sampling media, such as quartz fiber filters, may be suitable.

7.1.1.1 Appropriate workplace air samplers are described in Test Method D7035. The background metal content of the filters should be minimal (see Annex A1 of Test Method D7035).

7.1.2 *Portable, Battery-Operated Personal Sampling Pumps,* equipped with a flow-monitoring device (rotameter, critical orifice) or a constant-flow device, and capable of drawing 1 L/min to 5 L/min of air through a 0.8 µm mixed cellulose ester membrane filter for a period of at least 8 h.

7.2 *Analytical Apparatus:*

7.2.1 *Flame Atomic Absorption Spectrophotometer,* equipped with air/acetylene and nitrous oxide/acetylene burner heads.

[3] Boldface numbers in parentheses refer to the list of references appended to these methods.

7.2.2 *Hollow Cathode or Electrodeless Discharge Lamp,* for each element to be determined.

7.2.3 *Deuterium Continuum Lamp.*

7.2.4 *Compressed Air*—Appropriate pressure reducing regulator with base connections (see instrument manufacturer's instructions).

7.2.5 *Acetylene Gas and Regulator*—A cylinder of acetylene equipped with a two-gauge, two-stage pressure-reducing regulator with hose connections. (See instrument manufacturer instructions.)

7.2.6 *Nitrous Oxide Gas and Regulator*—A cylinder of nitrous oxide equipped with a two-gauge, two-stage pressure-reducing regulator and hose connections. Heat tape with the temperature controlled by a rheostat may be wound around the second stage regulator and hose connection to prevent freeze-up of the line. (See instrument manufacturer instructions.)

7.2.7 *Beakers,* Phillips or Griffin, 125 mL, borosilicate glass.

7.2.8 *Centrifuge Tubes,* 15 mL, graduated, borosilicate glass.

7.2.9 *Miscellaneous Borosilicate Glassware (Pipets and Volumetric Flasks)*—All pipets and volumetric flasks shall be calibrated Class A volumetric glassware.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used provided that it can be demonstrated that they are of sufficiently high purity to permit their use without decreasing the accuracy of the determinations.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean Type II reagent water conforming to Specification D1193.

8.3 *Hydrochloric Acid (HCl)*—Concentrated hydrochloric acid, 12 *N*, specific gravity 1.19.

8.4 *Nitric Acid (HNO_3)*—Redistilled, concentrated nitric acid, 16 *N*, specific gravity 1.42.

8.5 *Standard Stock Solutions* (1000 µg/mL) for each of the metals listed in Table 1. These solutions are stable for at least one year when stored in polyethylene bottles, and can be obtained from commercial sources or prepared in the laboratory in the following manner:

8.5.1 *Stock Aluminum Solution*—Dissolve 1.000 g of aluminum wire in a minimum volume of 1 + 1 HCl. Dilute to volume in a 1 L flask with purified water.

8.5.2 *Stock Barium Solution*—Dissolve 1.779 g of barium chloride ($BaCl_2 \cdot 2H_2O$) in water. Dilute to volume in a 1 L flask with purified water.

8.5.3 *Stock Bismuth Solution*—Dissolve 1.000 g of bismuth metal in a minimum volume of 6 *N* HNO_3. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.4 *Stock Cadmium Solution*—Dissolve 1.000 g of cadmium metal in a minimum volume of 6 *N* HCl. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.5 *Stock Calcium Solution*—To 2.497 g of primary standard calcium carbonate ($CaCO_3$) add 50 mL of distilled water. Add dropwise a minimum volume of HCl (approximately 10 mL) to dissolve the $CaCO_3$. Dilute to volume in a 1 L flask with purified water.

8.5.6 *Stock Chromium Solution*—Dissolve 3.735 g of potassium chromate (K_2CrO_4) in distilled water. Dilute to volume in a 1 L flask with purified water.

8.5.7 *Stock Cobalt Solution*—Dissolve 1.000 g of cobalt metal in a minimum volume of 1 + 1 HCl. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.8 *Stock Copper Solution*—Dissolve 1.000 g of copper metal in a minimum volume of 6 *N* HNO_3. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.9 *Stock Indium Solution*—Dissolve 1.000 g of indium metal in a minimum volume of 1 + 1 HCl. Addition of a few drops of HNO_3 and mild heating will aid in dissolving the metal. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.10 *Stock Iron Solution*—Dissolve 1.000 g of iron wire in 50 mL of 6 *N* HNO_3. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.11 *Stock Lead Solution*—Dissolve 1.598 g of lead nitrate ($Pb(NO_3)_2$) in 2 % (v/v) HNO_3. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.12 *Stock Lithium Solution*—Dissolve 5.324 g of lithium carbonate (Li_2CO_3) in a minimum volume of 6 *N* HCl. Dilute to volume in a 1 L flask with purified water.

8.5.13 *Stock Magnesium Solution*—Dissolve 1.000 g of magnesium ribbon in a minimum volume of 6 *N* HCl. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.14 *Stock Manganese Solution*—Dissolve 1.000 g of manganese metal in a minimum volume of 6 *N* HNO_3. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.15 *Stock Nickel Solution*—Dissolve 1.000 g of nickel metal in a minimum volume of 6 *N* HNO_3. Dilute to volume in a 1 L flask with 2 % (v/v) HNO_3.

8.5.16 *Stock Potassium Solution*—Dissolve 1.907 g of potassium chloride (KCl) in purified water. Dilute to volume in a 1 L flask with purified water.

8.5.17 *Stock Rubidium Solution*—Dissolve 1.415 g of rubidium chloride (RbCl) in distilled water. Dilute to volume in a 1 L flask with purified water.

8.5.18 *Stock Silver Solution*—Dissolve 1.575 g of silver nitrate ($AgNO_3$) in 100 mL of purified water. Dilute to volume in a 1 L volumetric flask with 2 % (v/v) HNO_3. The silver nitrate solution will deteriorate in light and must be stored in an amber bottle away from direct light. New stock silver solution shall be prepared every few months.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.