Designation: ~~D4185 – 17~~ 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 ~~sensitivity, detection limit,~~estimated method detection limits and optimum working concentration ranges for ~~23~~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 and health~~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.

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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.
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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.

TABLE 1 FAAS ~~Instrumental~~ Detection Limits and Optimum Working Concentration for ~~23~~ Metals

Element	Detection Limit, ~~µg/mL (approximately three times standard deviation of blank)^A~~	Optimum Linear Range ~~Upper Limit, µg/mL~~	~~TLV, mg/m^3 (elements, compound classes, and oxides)^B~~
~~Ag~~	~~0.001~~	~~5~~	~~**0.1 (metal)** 0.01 (soluble compounds as Ag)~~
~~Al~~	~~0.04~~	~~50~~	~~**2.0 (soluble** salts and alkyls not otherwise classified) 10 (metal dust and oxide)~~ ~~5 (pyro powder and welding fume)~~
~~Ba~~	~~0.01~~	~~10~~	~~**0.5 (soluble** compounds)~~
~~Bi~~	~~0.03~~	~~10~~	~~**No Limit** expressed for this element~~
~~Ca~~	~~0.002~~	~~1~~	~~**2 (oxide** as CaO)~~
~~Cd~~	~~0.0008~~	~~1~~	~~**0.01 (elemental** and compounds—total dust)~~ ~~0.002 (elemental compounds—respirable fraction)~~
~~Co~~	~~0.009~~	~~5~~	~~0.02 **(elemental and** inorganic) 0.1 (carbonyl and hydrocarbonyl)~~
~~Cr~~	~~0.003~~	~~5~~	~~**0.5 (metal** and Cr III compounds) 0.05 (water soluble Cr VI compounds)~~ ~~0.01 (insoluble Cr VI compounds)~~
~~Cu~~	~~0.002~~	~~5~~	~~**0.2 (fume)** 1 (dust and mists as Cu)~~
~~Fe~~	~~0.005~~	~~5~~	~~**5 (iron** oxide fume) 5 (soluble salts as Fe)~~
~~In~~	~~0.03~~	~~50~~	~~**0.1 (metal** and compounds)~~
~~K~~	~~0.003~~	~~1~~	~~**No Limit** expressed for this element~~
~~Li~~	~~0.0008~~	~~1~~	~~**No Limit** expressed for this element~~
~~Mg~~	~~0.0002~~	~~0.5~~	~~**10 (as** MgO fume)~~
~~Mn~~	~~0.002~~	~~5~~	~~**0.2 (elemental** and inorganic compounds)~~
~~Na~~	~~0.0003~~	~~0.5~~	~~**No Limit** expressed for this element~~
~~Ni~~	~~0.006~~	~~5~~	~~**0.05 (elemental,** soluble and insoluble compounds)~~
~~Pb~~	~~0.02~~	~~10~~	~~**0.15 (inorganic** compounds, fume, dust)~~
~~Rb~~	~~0.003~~	~~5~~	~~**No Limit** expressed for this element~~
~~Sr~~	~~0.003~~	~~5~~	~~No Limit expressed for this element~~
~~Tl~~	~~0.02~~	~~50~~	~~0.1 (soluble compounds)~~
~~V~~	~~0.06~~	~~100~~	~~**0.05 (pentoxide, respirable dust or fume, as V_2O_5)**~~
~~Zn~~	~~0.002~~	~~1~~	~~**10 (oxide** dust as ZnO)~~ ~~5 (oxide fume as ZnO)~~

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)

Element	Method Detection Limit, µg/sample	Optimum Linear Range, mg/m^3	Reference(s)
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)

~~[A] These detection limits represent ideal laboratory conditions; variability due to sampling, digestion, reagents, and sample handling has not been taken into account.~~

~~[B] Threshold Limit Values of Airborne Contaminants and Physical Agents adopted by ACGIH for 1994–1995. Values are elemental concentrations except as noted.~~

3.2 *Definitions of Terms Specific to This Standard:*

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

~~3.2.2 *instrumental detection limit*—that concentration of a given element which produces a signal three times the standard deviation of the reagent blank signal.~~

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

~~NOTE 1—Values for instrumental detection limit may vary from instrument to instrument.~~

4. Summary of Test Method

4.1 Workplace air samples are collected ~~on membrane filters and treated with nitric acid~~ 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.

TABLE 2 FAAS Flame and Operating Conditions for Each Element

Element	Type of Flame	Analytical Wavelength, nm	Interferences^A	Remedy^A	Re
~~Ag~~	~~Air-C_2H_2 (oxidizing)~~	~~328.1~~	~~$I0_3^-$, WO_4^{-2}, MnO_4^{-2}, Cl^-, F^-~~	~~B~~	~~(2,~~
Ag	Air-C_2H_2 (oxidizing)	328.1	$I0_3^-$, WO_4^{-2}, MnO_4^{-2}, Cl^-, F^-	B	(1,
~~Al^C~~	~~N_2O-C_2H_2 (reducing)~~	~~309.3~~	~~ionization, SO_4^{-2}, V~~	~~B,D,E~~	~~(4)~~
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~~	~~(1,~~
Ba	N_2O-C_2H_2 (reducing)	553.6	ionization, large concentration Ca	D,F	(2,
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~~	~~(1,~~
Ca	Air-C_2H_2 (oxidizing)	422.7	ionization (slight) and chemical ionization	D,E	(2,
	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~~	~~(4)~~
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~~	~~(1,~~
Fe	Air-C_2H_2 (oxidizing)	248.3	high Ni concentration, Si	B	(2,
~~In~~	~~Air-C_2H_2 (oxidizing)~~	~~303.9~~	~~Al, Mg, Cu, Zn, $H_xPO_4^{x-3}$~~	~~B~~	~~(5)~~
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~~	~~(1,~~
K	Air-C_2H_2 (oxidizing)	766.5	ionization	D	(2,
~~Li~~	~~Air-C_2H_2 (oxidizing)~~	~~670.8~~	~~ionization~~	~~D~~	~~(6)~~
Li	Air-C_2H_2 (oxidizing)	670.8	ionization	D	(14
~~Mg~~	~~Air-C_2H_2 (oxidizing)~~	~~285.2~~	~~chemical ionization~~	~~D,E~~	~~(1,~~
Mg	Air-C_2H_2 (oxidizing)	285.2	chemical ionization	D,E	(2,
	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~~	~~(1,~~
Na	Air-C_2H_2 (oxidizing)	589.6	ionization	E	(2,
Ni	Air-C_2H_2 (oxidizing)	232.0	none known		
~~Pb~~	~~Air-C_2H_2 (oxidizing)~~	~~217.0~~ ~~283.3~~	~~Ca, high concentration SO_4^{-2}~~	~~B~~	~~(7)~~
Pb	Air-C_2H_2 (oxidizing)	217.0 283.3	Ca, high concentration SO_4^{-2}	B	(15
~~Rb~~	~~Air-C_2H_2 (oxidizing)~~	~~780.0~~		~~D~~ ~~ionization~~	~~(1,~~
Sb	Air-C_2H_2 (oxidizing)	217.6	Pb, Cu	G	(2)

Element	Type of Flame	Analytical Wavelength, nm	Interferences[A]	Remedy[A]	Re
	~~Sr~~		~~ionization~~ ~~Air-C_2H_2 (oxidizing)~~ ~~chemical~~	~~460.7~~ D,E	~~(1,~~
		231.2			
	~~N_2O-C_2H_2 (reducing)~~				
			~~ionization~~		
	Air-C_2H_2 (oxidizing)	276.8			
Tl			none known		
	~~N_2O-C_2H_2 (reducing)~~	~~318.4~~			
~~Va~~			~~ionization~~		
V	N_2O-C_2H_2 (reducing)	318.4			
			ionization		
	Air-C_2H_2 (oxidizing)	213.9			
Zn			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~~ 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 (~~1~~16).[3]

5.2 FAAS is capable of quantitatively determining ~~most~~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 (~~2~~11, ~~4~~12, ~~8~~17, ~~9~~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 (~~8~~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.

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

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 (**~~9~~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 ~~*Cellulose Ester or Cellulose Nitrate Membrane Filters,*~~ *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~~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—~~Appropriate workplace air samplers are described in Test Method~~Alternative sampling media, such as quartz fiber filters, may ~~D7035. The background metal content of the filters should be minimal (see Annex A1 of Test Method~~ be suitable. ~~D7035).~~

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 ~~1–5~~1 L ~~L/min~~ ⁄min to 5 L⁄min of air through ~~the 0.8-µm~~ 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.

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-gage,~~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-gage,~~two-gauge, two-stage