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.

[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 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	IO_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.