INTERNATIONAL
RECOMMENDATION

OIML R 100

Edition 1991 (E)

Atomic absorption spectrometers for measuring metal pollutants in water

Spectromètres d'absorption atomique pour la mesure des polluants métalliques dans l'eau

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OIML R 100 Edition 1991 (E)

ORGANISATION INTERNATIONALE
DE MÉTROLOGIE LÉGALE

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ATOMIC ABSORPTION SPECTROMETERS
for MEASURING METAL POLLUTANTS in WATER

1 Scope

1.1 This Recommendation provides requirements for defining, testing, and verifying the performance of atomic absorption spectrometers (AAS systems) when used for legal measurements of metal pollutants in water. It does not intend to exclude any other equivalent means of analysis. An AAS system can be applied simply and rapidly for the determination of a large variety of metals in drinking, surface and saline waters as well as domestic and industrial waste waters [1-5]. Measurement methods are beyond the scope of this Recommendation; however, reference to some relevant measurement methods is provided in Annex A.

1.2 The flame and electrothermal atomization (furnace) types of AAS atomizers are principally covered by this Recommendation. The hydride generation and the cold vapor techniques are also briefly addressed. The conditions of operation described for each atomizer type are intended to cover applications for trace analysis.

1.3 It is possible that the performance of an instrument can exceed the minimum criteria prescribed in this Recommendation for these applications. Higher accuracy and repeatability of measurements can be accomplished by careful attention to optimize the performance of each of an instrument's components. In such cases, success may also depend on the knowledge, skill, and experience of the analyst.

2 Application

2.1 This Recommendation applies to an AAS system used for measuring metal pollutants in water, in assessing water quality, and as a means of monitoring the effectiveness of pollution control programs mandated by national laws or regulations. Such control programs are usually designed to cover both toxic and non-toxic pollutants in potable water, surface water, ground water, and effluents to assess whether metal levels are below regulated limits.

2.2 Normally after simple acidification, water free of particulate matter may be analyzed directly by AAS systems. However, domestic and industrial wastes require processing to dissolve suspended material before analysis. Sludges, sediments and other types of solid samples may also be analyzed after proper pretreatment.

3 Terminology

Note: References [6] and [7] provide definitions of terms relevant to this Recommendation. The definitions of some terms presented here, however, have been modified for better interpretation of this application.

3.1 Absorbance

A value equal to the logarithm of the ratio of the incident light to the transmitted light and is dependent on the path length and the concentration of the absorbing substance.

Note: The principle of measurement of absorbance by the instrument is the Beer-Lambert law.

3.2 Atomizer

A device for converting the analyte into an atomic vapor.

3.3 Blank solution

3.3.1 Blank reference solution

A solution that is used to set the zero absorbance on the spectrometer and that normally consists of a pure solvent such as deionized water.

3.3.2 Blank test solution

A solution that contains all the chemicals except for the element to be determined in the same concentration as required for the preparation of a reference standard solution of that element.

3.4 Reference standard solution

A solution containing an accurately known concentration of a sample element or elements of interest and that is used for testing and calibration of the instrument.

3.5 Characteristic concentration/characteristic mass

The concentration/mass of the element being determined needed to produce a signal of 0.0044 absorbance which is equivalent to a 1 % decrease in transmitted radiation.

Note: Sensitivity has historically been used for this term.

3.6 Detection limit

The concentration of an element that provides a signal equivalent to three times the standard deviation of the baseline noise as determined on a statistical basis. It is the minimum quantity measured before it can be concluded that an element has been detected.

Note: In this Recommendation, this term refers to the determination of an element in a reference solution free of interferents. The baseline noise is established by measuring the output signal of several aliquots of a blank test solution.

3.7 Working range

The range of concentrations of an element of interest in solution that can be measured with repeatability within specified limits.

Note: Dilution, or concentration, of a sample solution may be necessary to bring the element of interest within the working range. The zero absorbance of the spectrometer is adjusted using a blank reference solution.

3.8 Nebulizer

A device that converts a liquid sample into an aerosol.

3.9 Repeatability

The closeness of agreement between the results of successive measurements of the same quantity being measured using the same instrument and under the same defined conditions within a relatively short period of time.

3.10 Temperature programming

A means of automatically controlling the rate and duration of the temperature rise of a furnace-type AAS system.

4 Description of the instrument

4.1 General

AAS is based on the absorption by an atom in its ground state of characteristic resonance radiation produced by atoms of the same element contained in a light source. Light absorbed by the atomized sample is measured and can be related to the concentration of the element in the sample. In flame atomization, a steady-state absorption signal is normally measured, while in furnace atomization, a measurement is made of either the peak height or peak area of a transient signal. Physical and chemical interferences of the light absorbed by the element can occur that are unrelated to the element's concentration. A number of techniques may be used to eliminate or minimize such interferences.

4.2 Flame AAS system

The main components of the instrument are illustrated in Figure 1. The important components are the light source (A), which emits the characteristic narrow-line spectrum of the element of interest; the flame (B) into which the sample solution to be analyzed is aspirated by the nebulizer (C) and the analyte molecules are then converted into atoms by thermal molecular dissociation; a monochromator (D) for dispersion of the light into its discrete spectral wavelengths that can be selected for analysis with slits of variable width; a photomultiplier or other suitable detector (E) that converts photons into an electrical signal; and an amplifier and a readout system (F) for displaying or storing the output signal and subsequent data processing. Automatic background correction may be necessary for a flame AAS system especially at wavelengths below 300 nm.

4.3 Furnace AAS system

This instrument has basically the same major components as a flame AAS system except the flame is replaced by a furnace which is usually a graphite tube. A sample in solution is deposited in the furnace either directly on the tube wall or on a graphite platform. In some cases, a matrix modifier may be required. The temperature of the furnace is then increased progressively in a programmed manner to dry the sample, char (or ash) the matrix material, and atomize the element. Since this process is subject to non-specific absorption, automatic background correction is necessary. The repeatability of measured results is best when using an automatic sample introduction device.

Note: The detection limit of a furnace AAS system is a factor of 10 to 10^3 lower than that of a flame AAS system; however, the repeatability of measured results by a flame AAS system is typically better than for those of a furnace AAS system. Flame analysis can be performed more quickly than furnace analysis and is less matrix dependent. The measurement performance can decrease with the number of firings (age). The change may depend on the nature of the graphite tube.

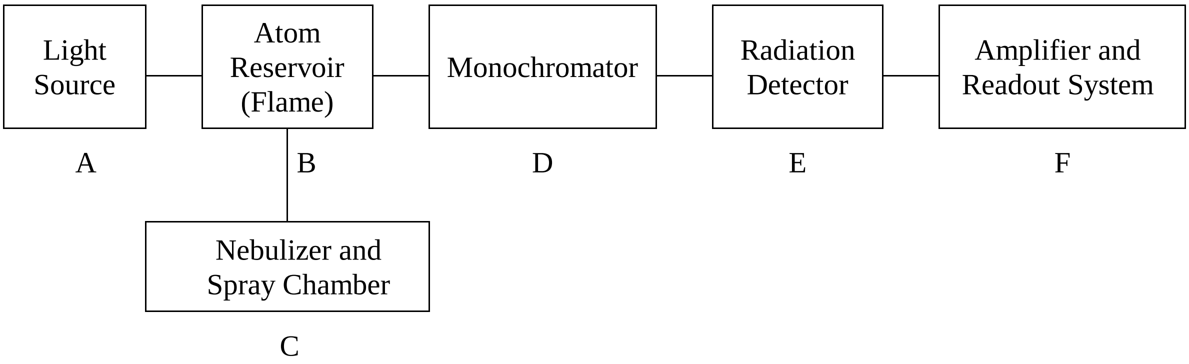

Figure 1. Diagram of a flame atomic absorption spectrometer

4.4 Other techniques

4.4.1 Hydride AAS system

This technique involves the generation of volatile hydrides of the element of interest in the sample solution undergoing analysis. The sample solution, an acid, and a reducing agent are reacted, either in a batch or continuous flow system, and the liberated hydride is transported to an atomizer by an inert carrier gas or by other means. The advantage of this technique over flame AAS is the separation and enrichment of the element to be determined and the significantly lower detection limit resulting from greater efficiency of sample introduction. This method may have more interference effects than the flame AAS system.

4.4.2 Cold vapor AAS system

This instrument applies only to mercury that can exist in a gaseous state at ambient temperature because of its relatively high vapor pressure. A reducing agent, usually either stannous chloride or sodium tetrahydrobromate, is mixed with an acidic sample solution containing trace elements of ionic mercury either in a batch or continuous flow system. The liberated atomic mercury vapor is then transported to an absorption cell by air or an inert gas. This technique for determining mercury is more sensitive than a flame AAS system.

5 Metrological requirements

5.1 For elements important in the analysis of water pollutants, the values for characteristic concentration (characteristic mass), detection limit, and working range at the appropriate wavelengths are provided for the various types of AAS systems in the following Tables: Table I for flame, Table II for furnace, and Table III for hydride and cold vapor techniques. An AAS system shall achieve these minimum performance requirements for elements to be determined under optimum measuring conditions consistent with the instrument's design and its rated operating conditions. Measurements shall be made, therefore, to confirm these values using blanks and reference standard solutions free of interferents.

Note: For sample solutions containing materials other than the element of interest, it may not be possible to achieve the values of characteristic concentration, detection limit, or working range listed in the Tables.

5.2 The working range of the instrument shall be that range of absorbance in which the repeatability of flame AAS measurements is within ± 2 % (relative standard deviation) and the repeatability of measurements using furnace and other sampling techniques is within ± 5 % (relative standard deviation).

Note:The range of absorbance required for most applications is usually from 0.05 to 0.5.

5.3 The spectrometer shall be able to cover the wavelength range from 190 nm to 770 nm.

TABLE I

METROLOGICAL CHARACTERISTICS OF A FLAME AAS SYSTEM

Element	Wavelength (nm)	Flame type[a]	Characteristic concentration[b] (mg/L)	Detection limit (mg/L)	Working range (mg/L)
Aluminium	309.3	NA	1.0	0.05	10 - 100
Antimony	217.6	AA	0.4	0.08	4- 40
Arsenic	193.7	AA or NA	1.0	0.25	10 - 100
Barium	553.6	NA	0.5	0.02	5 - 50
Beryllium	234.9	NA	0.03	0.002	0.3 - 3
Cadmium	228.8	AA	0.03	0.002	0.3 - 3
Calcium	422.7	AA or NA	0.1	0.004	1 - 10
Chromium	357.9	AA or NA	0.08	0.005	0.8 - 8
Cobalt	240.7	AA	0.12	0.008	1.2 - 12
Copper	324.7	AA	0.03	0.003	0.8 - 8
Iron	248.3	AA	0.1	0.006	1.0 - 10
Lead	283.3	AA	0.5	0.05	5 - 50
Lithium	670.8	AA	0.04	0.002	0.4 - 4
Magnesium	285.2	AA	0.008	0.0005	0.08 - 0.8
Molybdenum	313.3	NA	0.7	0.1	7 - 70
Nickel	232.0	AA	0.15	0.02	1.5 - 15
Selenium	196.0	AA or NA	0.6	0.2	6 - 60
Silver	328.1	AA	0.06	0.002	0.6 - 6
Sodium	589.0	AA	0.015	0.0008	0.15 - 1.5
Strontium	460.7	AA or NA	0.16	0.012	1.6 - 16
Vanadium	318.4	NA	2.0	0.1	20 - 200
Zinc	213.9	AA	0.02	0.002	0.2 - 2

a. The symbols AA and NA represent air-acetylene and nitrous oxide-acetylene flames, respectively.

b. This term has historically been called sensitivity.

TABLE II

METROLOGICAL CHARACTERISTICS OF A FURNACE AAS SYSTEM

Element	Wavelength (nm)	Characteristic Mass/Concentration		Detection limit[a]	Working range
		(pg)	(µg/L)[a]	(µg/L)	(µg/L)
Aluminium	309.3	8	0.4	0.2	4 - 40
Antimony	217.6	22	1.1	0.8	11 - 110
Arsenic	193.7	20	1	1	10 - 100
Barium	553.6	8	0.4	0.2	4 - 40
Beryllium	234.9	2	0.1	0.03	1 - 10
Cadmium	228.8	0.4	0.02	0.03	0.2 - 2
Chromium	357.9	8	0.4	0.1	4 - 40
Cobalt	240.7	16	0.8	0.2	8 - 80
Copper	324.7	8	0.4	0.2	4 - 40
Iron	248.3	6	0.3	0.2	3 - 30
Lead	283.3	24	1.2	0.3	12 - 120
Magnesium	285.2	0.2	0.01	0.004	0.1 - 1.0
Molybdenum	313.3	24	1.2	0.45	12 - 120
Nickel	232.0	40	2	1	20 - 200
Selenium	196.0	16	0.8	2.5	8 - 80
Silver	328.1	1	0.05	0.05	0.5 - 5
Vanadium	318.4	80	4	4	40 - 400
Zinc	213.9	0.6	0.03	0.03	0.3 - 3

a. A typical sample solution is from 10 to 20 µL. Values in this Table are based on the absolute mass and concentration for a sample solution of 20 µL. Therefore, for other volumes of the sample solution, the values should be adjusted accordingly.

TABLE III

METROLOGICAL CHARACTERISTICS OF AN AAS SYSTEM USING HYDRIDE AND COLD VAPOR TECHNIQUES[a]

Element	Wavelength (nm)	Characteristic concentration (µg/L)	Detection limit (µg/L)	Working range (µg/L)[b]
Arsenic	193.7	0.04	0.15	0.4 - 4
Antimony	217.6	0.08	0.24	0.8 - 80
Mercury	253.7	0.8	0.2	8 - 80
Selenium	196.0	0.06	0.24	0.6 - 6
Tin	286.3	0.4	1.0	4 - 40

a. Mercury is to be determined by the cold vapor technique, (see reference 8). The other elements listed are to be determined by the hydride technique. This Table applies to the determination of elements in sample solutions using a batch mode; however, instruments operating in continuous flow mode may have slightly different metrological characteristics than those presented.

b. Values in this table are based on a sample size of 50 mL and for the determination of the elements under optimum conditions. Because of the high sensitivity of the hydride technique, a sample solution of 50 mL is rarely required; sample solutions with volumes of 1 mL or less are generally sufficient to permit analysis in the working range (see reference 1).

6 Technical requirements

6.1 Atomizers

6.1.1 The flame atomizer shall be a nebulizer/premix type, in which the liquid sample is sprayed into a mixing chamber where the sample aerosol and the oxidant are mixed with the fuel. Analyses shall be carried out using either air-acetylene (for temperatures up to 2 300 °C) or nitrous oxide-acetylene (for temperatures up to 2 900 °C) flames. Table I gives some recommendations for the determination of various elements. Compressed air, acetylene, and nitrous oxide shall be available at sufficient pressure and purity. Compressed dry air shall be used; breathing air, or a mixture of nitrogen and oxygen, shall not be used; and copper tubing or connections shall not be used for acetylene lines.

6.1.2 A furnace AAS system shall have a means for achieving temperatures necessary for atomizing the element of interest in a sample. It shall be possible to program a variety of furnace temperatures to dry the sample, to remove the matrix material by pyrolysis or ashing, and to atomize the element of interest. Temperature programming of the furnace shall be possible in order to increase the temperature at a continuously variable rate and in a step-wise mode. A means for automatic background correction shall be available during analyses.

6.1.3 In the hydride technique, facilities should be available for atomizing hydrides by one of three ways:

(a) direct introduction to a cool flame (for example, argon-hydrogen)

(b) transported by argon into a long-optical-path silica tube heated by an air-acetylene flame, or

(c) similarly transported into a long-optical-path silica tube heated electrically [9].

6.1.4 In the cold vapor technique for mercury, the atoms produced should be transported to the absorption cell by one of the following methods:

(a) a closed circuit in which air is continuously pumped through the reaction vessel and absorption cell,

(b) a direct procedure of bubbling air or an inert gas through the reaction vessel to the absorption cell, or

(c) a continuous flow system in which an inert carrier gas transports mercury vapor to the absorption cell after separation of gaseous products from the reaction liquid by a gas/liquid separator [8].

Note: In the closed circuit and continuous flow system, the output signal rises to and remains at a plateau; whereas, in the direct procedure, the output signal rises to a peak and then returns to the baseline.

6.2 Light sources

6.2.1 The lamp used for determining an element in a sample shall be one of the following: hollow cathode, boosted discharge, or electrodeless discharge. Single or multi-element lamps may be used.

6.2.2 The light emitted by the source shall be modulated either electrically or mechanically before interacting with atoms from the atomizer. The detector shall be tuned to the frequency of modulation.

6.3 Optical system

6.3.1 The optical system used may be either single or double-beam type.

6.3.2 The monochromator shall be equiped with slits that can be preselected or adjusted to select the desired bandwidth for the wavelength of the element being determined to meet the requirements of 5.1.

6.4 The light detector used shall be selected so that its spectral response matches the wavelength range of the elements to be determined.

6.5 Readout systems

6.5.1 Digital or analog readout of the absorbance by the element being determined and variable integration times shall be available. Compensation for a non-linear relationship between absorbance and concentration should be available.

Note: A cathode-ray-tube display of the readout may also be used.

6.5.2 For connection to external computer systems, it should be possible to output data over a standard communication bus such as EIA RS-232C (9) or IEEE-488 (10). A computer data system may be used for storing and displaying AAS signal outputs, data handling and analysis and instrument control.

6.6 Markings shall be attached conspicuously to all major components of an AAS system as follows:

- name of the manufacturer,
- instrument model, serial number, and date (month and year) of manufacture,
- voltage, frequency, and current requirements.

Note: Labels or declarations concerning personnel safety and radio frequency interference emission should be provided according to national regulations.

7 Practical instructions

7.1 Atomic absorption spectrophotometers make use of high voltages and high temperatures during normal operation. Warning labels shall be placed conspicuously on the instrument to alert the user to these potential hazards (see 6.6). Instrument installation and operation, particularly with respect to flames and compressed gases, shall be consistent with the national safety regulations.

7.2 Manufacturers of an AAS system shall supply a manual that describes requirements for its installation, operation, and routine maintenance. Service manuals shall be available upon request.

Note: Manufacturers may provide analytical methods manuals.

7.3 Before installation of an AAS system, all laboratory environmental factors shall be considered. Manufacturers shall provide specifications for power consumption that include allowable variations from the rated mains voltage and frequency. Information shall also be provided for rated heat dissipation, and operating conditions for ambient temperature and humidity, and exhaust-gas venting.

8 Metrological controls

8.1 General considerations

8.1.1 An atomic absorption spectrometer is a complex instrument comprising a variety of atomizers, light sources, optics, and output devices. Major components used depend on the method specified as appropriate for analysis of a specific sample by the responsible national body for pollution control. Therefore, traditional legal metrology controls of initial and subsequent verification may not be practicable for these instruments. However, the responsible national body for pollution control should adopt as a minimum the control procedures specified in 8.2 as a means of assuring the continued metrological integrity of an AAS system.

8.1.2 Control procedures for specific analytical methods should also be established by the responsible national body for pollution control. When such control procedures are in place, the national body should consider means for assessing all laboratories that use an AAS system for measuring pollutants. Such means may include the following: accreditation of the user laboratory; self certification by the user laboratory, and proficiency testing through intercomparisons of the quality of measurements made among user laboratories.

8.2 Control procedures

8.2.1 A record shall be maintained that contains the following information in chronological order for each AAS system:

- results of initial, routine, and overall performance tests,
- identification of the reference standard solution, conditions of atomization, operating and measuring conditions, and the data system used for all tests,
- identification of the operator(s) of the instrument during tests,
- description of malfunctions and corrective actions taken,
- extent of maintenance and repair.

8.2.2 An initial test of an AAS system shall be performed upon installation according to the manufacturer's instructions. The results of this test shall be within the specifications provided by the manufacturer.

8.2.3 For a routine test, a reference standard solution should be used daily along with a quality control chart to verify the working range of the instrument over an extended period of time.

8.2.4 An overall test of the entire AAS system should be carried out frequently, for example once per work period, using reference standard solutions that are appropriate for the analytical method for the element or elements of interest. Annex B presents an appropriate performance test for the entire AAS system.

Note: Appropriate reference and certified reference materials may be available as indicated in references [12] and [13].

8.2.5 The responsible national body should publish control procedures that specify performance tests and calibration procedures for AAS systems appropriate for analytical methods for specific pollutants. The time interval between tests should also be specified as appropriate.

REFERENCES

[1] Welz, Bernhard, *Atomic Absorption Spectrometry*, Second Edition, VCH Publishers, Weinheim, FRG and Deerfield Beach, FL USA (1985). ISBN-0-89573-418-4

[2] Cantle, John Edward, Editor, *Atomic Absorption Spectrometry*, Elsevier, New York (1982). ISBN-0-444-42015-0

[3] Standing Committee of Analysts, Department of the Environment, U.K., "Atomic Absorption Spectrophotometry, An Essay Review", Her Majesty's Stationary Office (1979).

[4] Standing Committee of Analysts, "A General Introduction to Electrothermal Atomisation Atomic Absorption Spectrophotometry", Her Majesty's Stationary Office, London (1986).

[5] ASTM D3919-80, Practice for "Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry", Annual Book of Standards, Vol.11.01, ASTM, Philadelphia (1986). ISBN-0-8031-1140-1

[6] Joint Working Group of BIPM, IEC, ISO, and OIML, *International Vocabulary of Basic and General Terms in Metrology*, ISO (1984).

[7] ISO 6955:1982, *Analytical Spectroscopic Methods - Flame Emission, Atomic Absorption, and Atomic Fluorescence - Vocabulary*.

[8] ASTM D3223-80, Test Method for "Total Mercury in Water", Annual Book of Standards, Vol. 11.01, ASTM Philadephia (1986). ISBN-0-8031-1140-1

[9] Standing Committee of Analysts, Department of the Environment, U.K., "A Note on the Use of Hydride Generator Kits", Her Majesty's Stationary Office, London (1987).

[10] EIA RS-232-69, "Interface Between Data Terminal Equipment and Data Communication Equipment Employing Serial Binary Data Interchange" (Including Bulletin n^o 9, Application Notes; R 1981) Electronic Industries Association, Washington, D.C. EIA-232-0-1986

[11] IEEE 488-78, "Digital Interface for Programmable Instrumentation" (and Supplement 488A-80), Institute of Electrical and Electronic Engineers, New York, NY.

[12] ISO, *Directory of Certified Reference Materials*, Latest Edition, Geneva, Switzerland.

[13] United States National Institute of Standards and Technology (NIST), *Standard Reference Material Catalogue*, Latest Edition, Gaithersburg, MD 20899,USA.

ANNEX A

REFERENCES TO METHODS OF ANALYSIS

A.1 Joint Editorial Board, American Public Health Association, American Water Works Administration, and Water Pollution Control Federation, *Standard Methods for Examination of Water and Waste Water*, Latest Edition. ISBN-0-87553-131-8

A.2 U.S. Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020 (1979). PB-84-128677

A.3 M.W.Skougstad, et al. Editors, U.S.Geological Survey, *Methods for Determination of Inorganic Substances in Water and Fluvial Sediments*, USGS-TWSI Book 5, Chapter A1 (1979).

A.4 Association of Official Analytical Chemists, *Official Methods of Analysis of the Association of Official Analytical Chemists*, Methods Manual, Latest Edition. ISBN-0-935584-24-2

A.5 U.K. Standing Committee of Analysts, *Methods of the Examination of Waters and Associated Materials* (in series), HMSO, London.

A.6 ASTM, Annual Book of Standards on Water, Vol.11.01 (1986). ISBN-0-8031-1139-8

A.7 ISO, Standards of ISO/TC 147 on "Water Quality".

ANNEX B
(mandatory)

PERFORMANCE TEST FOR AN ENTIRE ATOMIC ABSORPTION SPECTROMETER SYSTEM

B.1 This test procedure involves measuring a trace amount of copper in a water solution.

B.2 The purpose of this test is to provide a performance test for an entire AAS system. It covers requirements for an AAS system when using either flame (B.3) or furnace (B.4) atomization of the sample. The test results should be consistent with the requirements of Section 5 of this Recommendation.

B.3 Test procedure for flame atomization

B.3.1 Equipment shall include: (a) copper hollow cathode lamp, (b) 2 mg/L copper reference standard solution in 0.1 % HNO_3, and (c) blank test solution of 0.1 % HNO_3.

B.3.2 Characteristic concentration check

B.3.2.1 Procedure. Set up the AAS system for a copper determination using the 324.7 nm wavelength, an air-acetylene flame, and set the instrument's parameters as recommended by the manufacturer. Aspirate the blank test solution into the flame, and set the instrument to zero. Aspirate the copper reference solution into the flame.

B.3.2.2 Acceptable results. The resulting signal from the 2 mg/L copper reference standard solution shall be at least an absorbance of 0.1 or better for a premix burner system with a flow spoiler and an absorbance of 0.2 or better for a premix burner system with an impact bead.

B.3.3 Repeatability check

B.3.3.1 Procedure. Use the same conditions of B.3.2 and set a 4 second integration time for the output signal. Take four separate sets of absorbance measurements of 10 aliquots each of the 2 mg/L copper reference standard solution. Calculate the mean and standard deviation of the absorbance for each of the four sets of data.

B.3.3.2 Acceptable results. The standard deviation, expressed as the coefficient of variation, for each set of data should be less than 3.5 %.

B.3.4 Detection limit check

B.3.4.1 Procedure. Use the conditions of B.3.2 and B.3.3 for measurement of the blank reference solution with 0.1 % HNO_3. Measure the absorbance or calibrate the instrument to read out 2.000 using the 2 mg/L copper reference standard solution. Take 4 sets of 10 readings of the output signal for the blank reference standard solution. Calculate the mean and standard deviation of each of the four sets of data.

B.3.4.2 Acceptable results. Multiply the standard deviation of each of the four sets of data by three. Each result, when referenced to the output signal for the copper reference standard solution of B.3.4.1 should be less than or equal to 0.003 mg/L.

B.4 Test procedure for furnace atomization

B.4.1 Equipment shall include: (a) copper hollow cathode lamp, (b) 2 µg/L and 10 µg/L copper reference standard solutions in 0.1 % HNO_3, (c) blank reference solution of 0.1 % HNO_3, and (d) a 20 µL micropipette, or automatic sampling device capable of delivering 20 µL.

B.4.2 Characteristic mass check

B.4.2.1 Procedure. Set up the AAS system using the 324.7 nm wavelength, and set the instrument's parameters as recommended by the manufacturer. Use a pyrolytically-coated graphite tube or an equivalent in the furnace; set the furnace operating parameters as recommended by the manufacturer, and set the background correction as necessary. Pipette a 20 µL aliquot of the 10 µg/L copper reference standard solution into the furnace, atomize this sample, and measure the peak height or peak area of the output signal of absorbance. Similarly, pipette a 20 µL aliquot of the blank reference solution of 0.1 % HNO_3 into the furnace, atomize, and measure the output absorbance.

B.4.2.2 Acceptable results. The difference in the output signals from the copper reference standard solution and the blank reference solution shall be an absorbance of 0.08 or greater.

B.4.3 Detection limit check

B.4.3.1 Procedure. Use the same conditions as B.4.2. Measure the absorbance or calibrate the instrument to read out 2.000 using a 20 µL aliquot of the 2 µg/L copper reference standard solution. Take 10 readings of the output signal from atomizing 20 µL aliquots of the 0.1 % HNO_3 blank reference solution. Determine the mean and the standard deviation of this set of data.

B.4.3.2 Acceptable results. The standard deviation obtained in B.4.3.1 should be multiplied by three. The results when referenced to the output signal of the copper reference standard solution of B.4.3.1 shall be less than or equal to 0.2 µg/L for the 20 µL aliquot.

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