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Standard Practice for Determination of Elements by Graphite Furnace Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation E1184; 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 practice covers a procedure for the determination of microgram per milliliter ($\mu\text{g/mL}$) or lower concentrations of elements in solution using a graphite furnace attached to an atomic absorption spectrometer. A general description of the equipment is provided. Recommendations are made for preparing the instrument for measurements, establishing optimum temperature conditions and other criteria which should result in determining a useful calibration concentration range, and measuring and calculating the test solution analyte concentration.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.3 *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 hazard statements are given in Section 9.

1.4 *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](#)

[E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials](#)

¹ This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.20 on Fundamental Practices.

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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.

[E131 Terminology Relating to Molecular Spectroscopy](#)

[E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

[E406 Practice for Using Controlled Atmospheres in Atomic Emission Spectrometry](#)

3. Terminology

3.1 Refer to Terminologies [E131](#) and [E135](#) for the definitions of terms used in this practice.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *atomization, v*—the formation of ground state atoms that absorb radiation from a line emission source. The atomization process in graphite furnace atomic absorption spectrometry (GF-AAS) analysis is covered in [6.2](#).

3.2.2 *pyrolysis, n*—the process of heating a specimen to a temperature high enough to remove or alter its original matrix, but not so high as to volatilize the element to be measured. The purpose of the pyrolysis step in GF-AAS analysis is to remove or alter the original specimen matrix, thereby reducing or eliminating possible interferences to the formation of ground state atoms that are formed when the temperature is increased during the atomization step. Many publications and references will refer to pyrolysis as *charring* or *ashing*.

3.2.3 *pyrolytic graphite coating, n*—a layer of pyrolytic graphite that coats a graphite tube used in GF-AAS analysis. Pyrolytic graphite is formed by pyrolyzing a hydrocarbon, for example, methane, at 2000 °C.

3.2.4 *ramping, v*—a slow, controlled increase of the temperature in the graphite tube. Ramping will provide for an efficient but not too rapid removal or decomposition of the specimen matrix. Most graphite furnaces allow for ramping during the drying, pyrolysis, and atomization steps. It is usually employed during the drying and pyrolysis steps. However, some instrument manufacturers may recommend ramping during the atomization step depending on the specimen matrix and the element being measured (for example, the analysis of cadmium or lead in hair or blood). The power supplies for most instruments also allow the rate of the temperature increase to be varied.

4. Significance and Use

4.1 This practice is intended for users who are attempting to establish GF-AAS procedures. It should be helpful for establishing a complete atomic absorption analysis program.

5. Theory of Atomic Absorption Spectrometry (AAS)

5.1 In flame atomic absorption spectrometry (Flame-AAS), a test solution is aspirated into a flame through which passes radiation from a line emission source of the element sought. The radiation of the element sought is absorbed in proportion to the concentration of its neutral atoms present in the flame. The concentration of the analyte is obtained by comparison to calibrations.

5.2 The theoretical basis for using atomic absorption to determine analyte concentration can be found in texts on instrumental analysis in analytical chemistry and in the literature.

6. Theory of Graphite Furnace Atomic Absorption Spectrometry

6.1 *Basic Technique*—A discrete amount of test solution is heated in a graphite furnace to produce a cloud of neutral atoms. Light, emitted by a specific element from a line source at a specific wavelength, is passed through the cloud and neutral atoms of this same element in the cloud absorb some of this light. Thus, the intensity of the beam is decreased at the wavelengths characteristic of the element. This absorbance of radiation from the external light source depends on the population of the neutral atoms and is proportional to the concentration of the element in the test solution.

6.2 *Graphite Furnace Atomization*—Thermodynamic and kinetic theories must be considered to fully understand the atomization process that takes place in the graphite furnace. Jackson (1)³ and also Campbell and Ottaway (2) provide a complete discussion of the thermodynamic theory. They also discuss thermal dissociation of metal oxides, reduction of metal oxides, evaporation of metal oxides prior to atomization, and carbide formation. Several models have been proposed to explain the theory of kinetic atomization. A search of the literature will find discussions of atomization under increasing temperature, and atomization under isothermal conditions (3). Additional discussion and clarification of the kinetic atomization theory is provided by Paveri-Fontana et al. (4).

7. Apparatus

7.1 *Atomic Absorption Spectrometer*—Most flame atomic absorption spectrometers manufactured currently can be easily adapted for graphite furnace analysis.

7.1.1 Automatic background correction is necessary for all spectrometers used with graphite furnaces. When graphite furnaces are heated to high temperatures, background from absorption is produced within the graphite tube. Also, small amounts of particulate matter in the furnace contribute to the

background signal. Therefore, it is essential to correct or compensate for this background.

7.2 *Electrothermal Atomizers*—The most commonly used electrothermal atomizer is the graphite tube furnace. This atomizer consists of a graphite tube positioned in a water-cooled unit designed to be placed in the optical path of the spectrometer so that the light from the hollow cathode lamp passes through the center of the tube. The tubes vary in size depending upon a particular instrument manufacturer's furnace design. These tubes are available with or without pyrolytic graphite coating. However, because of increased tube life, tubes coated with pyrolytic graphite are commonly used. The water-cooled unit or atomizer head which holds the graphite tube is constructed in such a way that an inert gas, usually argon or nitrogen, is passed over, around, or through the graphite tube to protect it from atmospheric oxidation. The heating of all of these atomizers is controlled by power supplies which make it possible to heat the graphite tube to 3000 °C in less than 1 s. Temperatures and drying, pyrolysis, and atomization times are controlled by these power supplies (determination of these parameters is covered later in Section 10). The flow of the inert gas through the atomizer head also is controlled by the power supplies.

7.2.1 Other types of atomizers and accessories such as the graphite cup, graphite rod, L'vov platform, tantalum filament, and tantalum boat have been used and are covered in the literature. With the exception of the L'vov platform, they have not enjoyed the widespread and general use that the graphite tube atomizers have. Therefore, they will not be covered in detail within this practice. A good general description of these other units can be found in the literature.

7.3 *Signal Output System*—The output signal resulting from the atomization of a specimen may be displayed by a strip chart recorder, video display, digital computer, printer, or other suitable device depending on the electronic capability of the spectrometer employed.

7.3.1 If a strip chart recorder is used, it must have a full scale response of 0.5 s or less. Normally, when a strip chart recorder is used, the absorption is determined by measuring the peak height of the recorder tracing. This procedure is appropriate because the absorption signal generated by a graphite furnace atomizer usually results in a very narrow peak (absorption versus time). However, some specimen matrices may require instrumental parameters (for example, ramping), which will result in broad absorption versus time peaks. In such cases, peak area measurement may be more appropriate. The instrument manufacturer's manual should be consulted to determine which procedure is most suitable for the instrument being used.

8. Reagents and Materials

8.1 Picogram quantities of some elements can be determined by means of graphite furnace atomization. Therefore, ultra-pure acids and Type I (Specification D1193) water shall be used to prepare calibration solutions and test solutions.

9. Hazards

9.1 *Electrical Hazards*—The power supplies for graphite furnaces require high-voltage (greater than 200 V) electrical

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.

service. Electrical power shall be supplied as determined from load requirements in accordance with the latest revision of the National Electrical Code. The recommendations of the equipment manufacturers and local engineers should be followed in designing the electrical service.

9.2 Compressed Gas Hazard—The inert or non-oxidizing atmosphere required in the graphite furnace during heating cycles is usually maintained by using argon or nitrogen gas delivered from portable gas cylinders.

9.2.1 Sufficient space shall be provided for the cylinders, which shall be kept in a vertical position and always well secured. They shall not be used or stored near burners, hot plates, or in any area where the temperature exceeds 52 °C (125 °F). The contents shall be identified with labels or stencils and color coding.

9.2.2 Two-stage regulators with pressure gages should be used as part of the basic flow system to deliver required cylinder gas to the instrument at a reduced pressure. Practice **E406** and the manufacturer's instructions should be followed with regard to the types of regulators, flow-metering valves, and tubing for gas transport when designing a gas delivery system.

9.2.3 Reserve gas cylinders should not be stored in the laboratory area. Gas storage areas shall be adequately ventilated, fire-resistant, located away from sources of ignition or excessive heat, and dry. All cylinders shall be chained in place or placed in partitioned cells to prevent them from falling over. In all cases, storage areas shall comply with local, state, and municipal requirements as well as with the standards of the Compressed Gas Association and the National Fire Prevention Association. Access to gas storage areas should be limited to authorized personnel.

9.3 Chemical Hazard—Practice **E50** should be consulted for recommendations and precautions concerning chemical hazards.

9.4 Ventilation—A small hood is required to carry away any toxic fumes that may result from the atomization process. Follow the manufacturer's instructions for proper hood installation.

9.5 Laboratory—The laboratory in which the graphite furnace is operated shall be kept as clean as possible. Any procedures that may produce an atmosphere that is corrosive to the instrumentation or detrimental to the analysis of the specimen should be removed from the laboratory.

9.6 Laboratory Apparatus—It is imperative that all laboratory apparatus and containers used in the preparation of calibration and test solutions be acid cleaned. All laboratory ware, including plastic tips used on micropipets for the transfer of calibration solutions and test solutions to the graphite tube, should be acid rinsed before being used. Once laboratory ware is acid rinsed, all of the items that come in contact with analytical solutions shall be isolated from subsequent contact with fingers, clothing, bench tops, etc.

9.7 Magnetic Background Correction—If the graphite furnace atomic absorption unit is provided with a background correction that does or can produce a magnetic field, the unit

should not be operated by an individual who wears, internally or externally, a medical device such as a pacemaker, that can be affected by the magnetic field, without the approval of the prescribing or installing physician, or both. In addition an appropriate warning sign should warn visitors of the magnetic field.

10. Preparation of Apparatus

10.1 Graphite Furnace Parameters—All graphite furnaces are resistance-heated by power supplies that provide individually controlled heating stages for drying, pyrolysis, and atomization. The means to control the times and temperatures of these stages will vary with instrumentation. Most manufacturers provide a listing of the parameters required for the graphite furnace analysis of numerous elements in the most commonly encountered matrices. The recommended parameters for a particular element should be verified for the specific instrument being used with an appropriate solution. Also, for sample matrices that differ from those printed in the manufacturer's list, the most appropriate time and temperature setting for each stage must be calculated or determined experimentally (see **10.1.1**).

NOTE 1—Ramping is normally used during the drying and pyrolysis stages. Some procedures may also recommend that ramping be used during the atomization stage, depending upon the specimen matrix and the element being measured. Refer to the instrument manufacturer's manual of the particular instrument for the recommended ramp rates, if any, for the type of solution being analyzed.

10.1.1 Drying—The drying stage is a low temperature stage in which the graphite tube is heated to a temperature high enough to evaporate, but not boil, any solvent. The ideal drying temperature would be one just below the boiling point of the solvent. Specimen spattering may occur if the temperature is raised above the boiling point before evaporation is complete. The time, in seconds, required to completely dry a specimen may be calculated by multiplying 1.5 times to 2 times the volume of the specimen, measured in microliters (μL). For example, a 10-μL specimen would require a drying time of 15 s to 20 s. If an auto-sampling device is to be used, adjust it to deposit the desired volume (in microliters) in the graphite tube (see **10.1.2**). Deposit a measured amount of the reagent blank solution, prepared as directed in **11.1**, in the graphite tube. The volume should be identical to the test solution volume (see **10.1.2**). Cycle through the heating stages and adjust the readout system of the instrument to read zero absorbance during the atomization of the reagent blank solution. If the spectrometer has an auto-zero capability, the auto-zero should be activated at this time. Atomize a calibration solution, prepared as directed in **11.3**, containing the analyte at a concentration that will yield an absorbance of 0.1 to 0.3 and is anticipated to be within the linear absorbance range of the procedure. Where applicable, refer to the instrument manufacturer's instruction manual to determine an approximation of the linear concentration range for the analyte. Determine if adequate sensitivity (μg-mL/0.0044 absorbance) has been obtained by reference to the instrument instruction manual or to the analytical procedure utilized.

10.1.2 The appropriate volume, in microliters, of any solution deposited in a graphite tube may vary depending on the