Designation: E1184 – 02

Standard Practice for
Electrothermal (Graphite Furnace) Atomic Absorption Analysis[1]

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 millilitre (μg/mL) or lower concentrations of elements in solution using an electrothermal atomization device attached to an atomic absorption spectrophotometer. 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 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific safety hazard statements are given in Section 9.

NOTE 1—This practice is a companion to Practice E663.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
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 Spectrochemical Analysis
E416 Practice for Planning and Safe Operation of a Spectrochemical Laboratory[3]
E663 Practice for Flame Atomic Absorption Analysis
E863 Practice for Describing Atomic Absorption Spectrometric Equipment[3]

3. Terminology

3.1 Refer to Terminology E131 and E135 for the definition of terms used in this practice.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *atomization*—the formation of ground state atoms that absorb radiation from a line emission source (see Practice E663). The atomization process in electrothermal atomic absorption analysis is covered in greater detail in 6.2.

3.2.2 *pyrolysis*—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 electrothermal atomic absorption 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*—a layer of pyrolytic graphite that coats a graphite tube used in electrothermal atomic absorption analysis. Pyrolytic graphite is formed by pyrolizing a hydrocarbon, for example, methane, at 2000°C.

3.2.4 *ramping*—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 electrothermal atomizers 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

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[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.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

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 spectroscopists who are attempting to establish electrothermal atomic absorption procedures. Used in conjunction with Practice E663, it should be helpful for establishing a complete atomic absorption analysis program.

5. Theory of Atomic Absorption Spectroscopy

5.1 Practice E663 provides a brief discussion of the theory of flame atomic absorption spectroscopy. A more complete and detailed discussion of atomic absorption spectroscopy may be found in Dean and Rains (**1**).[4]

6. Theory of Electrothermal Atomic Absorption Spectroscopy

6.1 *Basic Technique*—A discrete amount of test solution is heated in an electrothermal device 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 absorbs 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 *Electrothermal Atomization Theory*—Thermodynamic and kinetic theories must be considered to fully understand the atomization process that takes place in the electrothermal atomizer. Fuller (**2**) and also Campbell and Ottaway (**3**) 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. Two studies provide a general summary of these models: one provides a discussion of atomization under increasing temperature (**4**), the other discusses atomization under isothermal conditions (**5**). Additional discussion and clarification of the kinetic atomization theory is provided by Paveri-Fontana et al (**6**) and Johnson et al (**7**).

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer*—Most flame atomic absorption spectrophotometers manufactured currently can be easily adapted for electrothermal analysis. These spectrophotometers are described in Practice E863.

7.1.1 Automatic background correction is necessary for all spectrophotometers used with electrothermal devices. When electrothermal atomizers, especially graphite furnaces, are heated to high temperatures, background from absorption is produced within the graphite tube. Also, small amounts of particular 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 spectrophotometer 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 are 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 is provided by Fuller (**2**).

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 spectrophotometer 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 an electrothermal 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 electrothermal atomization. Therefore, all acids and water used to prepare calibration solutions and test solutions shall be double distilled or ultra-pure in quality. All reagents used in the preparation of calibration solutions and test solutions shall be spectroscopically pure.

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