



Designation: E 863 – 98

## Standard Practice for Describing Atomic Absorption Spectrometric Equipment<sup>1</sup>

This standard is issued under the fixed designation E 863; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers those features that are important for evaluating atomic absorption spectroscopy equipment. It also discusses performance characteristics and identifies parameters that should be recorded in analytical procedures.

1.2 *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 practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in the Note in 5.3 and specific precautionary statements are given in Section 7.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials<sup>2</sup>
- E 416 Practice for Planning and Safe Operation of a Spectrochemical Laboratory<sup>3</sup>
- E 520 Practice for Describing Detectors in Emission and Absorption Spectroscopy<sup>3</sup>
- E 1770 Practice for Optimization of Electrothermal Atomic Absorption Spectrometric Equipment<sup>3</sup>
- E 1812 Practice for Optimization of Flame Atomic Absorption Spectrometric Equipment<sup>3</sup>

### 3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology E 135.

### 4. Significance and Use

4.1 This practice provides criteria for instrument selection and should be useful for setting up an atomic absorption facility.

### 5. Description of Equipment

5.1 *Optical System*—Both single-beam and double-beam optical systems are in use. A single-beam system employs one optical path to measure both incident and transmitted radiation. The two measurements are separated in time. A double-beam system separates the incident radiation into two portions. One of these portions (often designated  $I$ ) is measured after traversing the analytical cell; the other portion (often designated  $I_o$ ) represents the radiation incident on the analytical cell. Signals from the two portions are used to compensate for baseline variability. In single-beam systems, instability of either the incident intensity or the detector-amplifier sensitivity-gain contributes to the variability of the signal. Double-beam systems can correct completely for either type of instability. Neither single-beam or double-beam systems can correct for fluctuations occurring in the flame.

5.2 *Radiation Sources*—The most widely used source of absorbing lines is the hollow cathode lamp. It emits narrow spectral lines, has low background, and is applicable to virtually all elements amenable to atomic absorption analysis. The high-frequency electrodeless discharge lamp (EDL) is also an excellent source of sharp line spectra. It provides more intensity than the hollow cathode lamp, but is not as universally applicable. EDLs require a special power supply.

5.2.1 The hollow cathode lamp shall emit stable, low-drift radiation of usable intensity, free of interfering spectral lines due to filler gas or cathode impurities.

5.2.2 *Stability*—Intensity drift shall not exceed 3 % per 15 min after a 30-min warm up. Many lamps available now are better than 1 % per 15 min after warm up.

5.2.3 *Lamp Life*—The hollow cathode lamp or EDL shall maintain the above characteristics over the manufacturer's warranted life. The expected life of hollow cathode lamps is approximately 5 Ah.

5.3 *Flames*—Flames are classified as diffusion or premixed. The characteristics of the diffusion flame are determined by the rate at which the fuel diffuses into the ambient oxygen or the oxidant supplied through a separate orifice.<sup>4</sup> In the premixed

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.20 on Fundamental Practices and Measurement Traceability.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.06.

<sup>4</sup> "Guidelines for the Purity and Handling of Gases Used in Atomic Absorption Spectroscopy", *A12.1a*, 1978, Scientific Glass Makers Association, 1140 Connecticut Ave., N.W., Washington, DC 20036.

flame, the characteristics are determined by the kinetics of the chemical combustion producing the flame. In general, diffusion flames are more turbulent than pre-mixed flames and the flame reactions are not spatially resolved. Premixed acetylene and air or acetylene and nitrous oxide flames are the most commonly used atomic absorption flames. Acetylene is the most common fuel. Hydrogen, natural gas, propane, and similar fuels are sometimes used to produce cooler flames. Air and nitrous oxide are the normal oxidants. Oxygen has sometimes been used, but due to higher flame propagation velocities, it is not used anymore.

**NOTE 1—Warning:** Never use copper tubing for acetylene because of the possible formation of explosive copper acetylide.

**5.4 Burners**—Historically two types of burners were used in atomic absorption spectroscopy, the direct injection or total consumption burner and the pre-mix nebulizer burner. The total consumption burner ensures freedom from flashbacks and was once popular for flame spectroscopy. However, it has serious disadvantages such as high noise levels, lack of distinct flame zones, high background emission, and difficulties in obtaining flame shape suitable for absorption measurement,<sup>5</sup> and, therefore is no longer being used. The most widely used burner now is the pre-mix nebulizer burner. In this burner a small fraction of the aspirated solution reaches the flame. Only the very finest droplets are used, which makes this an energy-efficient system. The burner produces a high-temperature flame with relatively low emission background or noise. The flame can be shaped to provide for a long light path through it. A serious disadvantage is that the pre-mix chamber contains an explosive gas mixture during operation. An explosive flashback is possible when the burning velocity exceeds the velocity of the burning gases passing through the burner orifice. Equipment shall be designed to minimize the probability of a flashback, protect the instrument operator and anyone nearby from injury if a flashback does occur, and minimize damage to the burner, chamber, and instrument.

**5.4.1 Nebulizers**—The nebulizer is used to aspirate the sample and convert it to a fine mist. The most common nebulizers employ oxidant gas as the source of energy for nebulization. The mist consists of a wide distribution of particle sizes but only the smallest (less than 1  $\mu\text{m}$  in diameter) reach the flame and contribute to the atomic absorption signal. Some pre-mix chambers employ an impingement bead to enhance the generation of smaller particles, and mix the gases and vapors before passing through the burner slots. Other pre-mix chambers enhance particle size separation by baffles that allow only the finest particles to reach the flame. The result is a smaller but more stable absorption signal. Ultrasonic nebulizers have been used experimentally for fog generation. They provide an efficient generation of a uniform mist. In practice, however, such problems as feeding the sample uniformly, conducting the vapor to the burner, and sample memory have prevented this nebulization system from becoming popular. The nebulizer efficiency, or the fraction of the

aspirated solution that actually reaches the flame, shall be specified for pre-mix nebulizer burner systems. The efficiency is a function of the rates of solution uptake and of the discharge through the drain tube.

**5.4.2 Gas Flow Controls**—Stable flame operation depends on reproducible and stable flow of fuel and oxidant gases. A minimal system shall have a two-stage regulating device at the fuel source and at the oxidant source. There shall be a pressure meter for the nebulizing gas, a flow meter for each gas, and a flow control system for each gas line at the burner.

**5.4.2.1** The manufacturer shall specify suitable gas flows. Flow meters should be calibrated in litres per minute or some comparable unit. If the burner system is to be used with a nitrous oxide acetylene flame, special precautions to avoid flashback when the flame is ignited and extinguished, shall be provided. The gas control system shall allow the oxidant to be changed from air to nitrous oxide and back again without interruption of flow to the burner. The flame shall be ignited using air and acetylene, then switched to nitrous oxide. The reverse procedure shall be followed when the flame is extinguished. Some instruments have systems which automatically provide the startup-shutdown sequence, monitor gas flows, and shut down automatically if the gas flows are outside a prescribed range.

**5.5 Electrothermal Atomizers**—Most flame atomic absorption spectrometers manufactured currently can be easily adapted for electrothermal analysis, while some atomic absorption spectrometers are dedicated to electrothermal analysis.

**5.5.1** 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 graphic 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.

**5.6 Automatic Background Correction**—Automatic background correction is recommended for all atomic absorption units.

**5.6.1** Automatic background correction is a necessity 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 particulate matter in the furnace contribute to the background signal. Therefore, it is essential to correct or compensate for this background.

**5.6.1.1** Magnetic (Zeeman type) background correction gives wider range of correction than hydrogen or deuterium

<sup>5</sup> Kirkbright, G. F., and Sargent, M., *Atomic Absorption and Fluorescence Spectroscopy*, Academic Press, New York, NY, 1974, p. 201.