

**Designation: E 1770 – 95**

## **Standard Practice for Optimization of Electrothermal Atomic Absorption Spectrometric Equipment<sup>1</sup>**

This standard is issued under the fixed designation E 1770; 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 the optimization of electrothermal atomic absorption spectrometers and the checking of spectrometer performance criteria.

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.*

### **2. Referenced Documents**

#### **2.1 ASTM Standards:**

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals<sup>2</sup>

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data<sup>3</sup>

E 1184 Practice for Electrothermal Atomic Absorption Analysis<sup>3</sup>

E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric Atomic Analysis<sup>3</sup>

### **3. Significance and Use**

3.1 This practice is for optimizing the parameters used in the determination of trace elements in metals and alloys by the electrothermal atomic absorption spectrometric method. It also describes the practice for checking the spectrometer performance. The work is expected to be performed in a properly equipped laboratory by trained operators and appropriate disposal procedures are to be followed.

### **4. Apparatus**

4.1 *Atomic Absorption Spectrometer with Electrothermal Atomizer*, equipped with an appropriate background corrector, a signal output device such as a video display screen (VDS), a digital computer, a printer or strip chart recorder, and an autosampler.

4.2 *Grooved Pyrolytic Graphite-Coated Graphite Tubes*, conforming to the instrument manufacturer's specifications.

4.3 *Pyrolytic Graphite Platforms*, L'vov design, fitted to the tubes specified in 4.2.

4.4 *Pyrolytic Graphite-Coated Graphite Tubes*, platformless, conforming to the instrument manufacturer's specifications.

4.5 *Radiation Source for the Analyte*—A hollow cathode lamp or electrodeless discharge lamp is suitable.

NOTE 1—The use of multi-element lamps is not generally recommended, since they may be subject to spectral line overlaps.

4.6 For general discussion of the theory and instrumental requirements of electrothermal atomic absorption spectrometric analysis, see Practice E 1184.

### **5. Reagents**

5.1 *Purity and Concentration of Reagents*—The purity and concentration of common chemical reagents shall conform to Practices E 50. The reagents should be free of or contain minimal amounts ( $<0.01 \mu\text{g/g}$ ) of the analyte of interest.

5.2 *Magnesium Nitrate Solution* [ $2 \text{ g/L Mg(NO}_3)_2$ ]—Dissolve  $0.36 \pm 0.01 \text{ g}$  high-purity  $\text{Mg(NO}_3)_2 \cdot 6\text{H}_2\text{O}$  in about 50 mL of water, in a 100-mL beaker, and transfer the solution into a 100-mL volumetric flask. Dilute to mark with water and mix. Store in polypropylene or high-density polyethylene bottle.

5.3 *Calibration Solutions*—Refer to the preparation of calibration solutions in the relevant analytical method for the determination of trace elements in the specific matrix. Calibration solution  $S_0$  represents the calibration solution containing no analyte;  $S_1$  the least concentrated calibration solution;  $S_2$  the calibration solution with the next highest concentration; through  $S_k$ , the most concentrated calibration solution. Also refer to Practice E 1452.

5.4 *Matrix Modifiers*—Refer to the relevant analytical method for the determination of trace elements in the specific matrix.

### **6. Initial Checks and Adjustments**

6.1 Turn on power, cooling water, gas supplies, and fume exhaust system.

6.2 Open the furnace to inspect the tube and contacts. Replace graphite components, if wear or contamination is evident. Inspect windows and clean or replace as required.

6.2.1 New graphite contacts or new tubes should be conditioned prior to use, in accordance with the heating program

<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 Fundamental Practices.

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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.