Designation: E 1770 – 95 (Reapproved 2001)

Standard Practice for
Optimization of Electrothermal Atomic Absorption Spectrometric Equipment[1]

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 (ϵ) 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:* [2]
E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals
E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data
E 1184 Practice for Electrothermal (Graphite Furnance) Atomic Absorption Analysis
E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric Atomic Analysis

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 µg/g) of the analyte of interest.

5.2 *Magnesium Nitrate Solution* [2 g/L $Mg(NO_3)_2$]— Dissolve 0.36 ± 0.01 g high-purity $Mg(NO_3)_2 \cdot 6H_2O$ 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.

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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 recommended by the manufacturer.

6.2.1.1 In the absence of manufacturer's recommendations, a conditioning program for a graphite furnace is shown in Table 1.

7. Radiation Source

7.1 Install and operate hollow cathode lamps or electrodeless discharge lamps in accordance with the manufacturer's instructions.

7.2 After the manufacturer's prescribed warm-up time, the signal from the radiation source should not deviate by more than 0.5 % from the maximum value (that is, by not more than 0.002 absorbance units) over a period of 15 min. Significantly greater fluctuations are usually indicative of a faulty lamp or power supply.

8. Spectrometer Parameters

8.1 *Wavelength*, as specified by the appropriate procedure.

8.2 *Slit Width*, as recommended by the manufacturer. Where two slit height settings are available, select the shorter height.

8.3 *Background Correction*:

8.3.1 *Zeeman Background Correction System*:

8.3.1.1 Ensure that the poles of the magnet are clean and securely tightened.

8.3.1.2 If necessary, set the optical temperature sensor in accordance with the instrument manufacturer's instructions.

8.3.2 *Continuum Background System*:

8.3.2.1 Select the background correction option and allow lamps to stabilize for 30 min. Verify that the energies of the analyte lamp and the deuterium lamp are balanced within tolerances recommended by the manufacturer.

8.3.2.2 If necessary, set the optical temperature sensor in accordance with the instrument manufacturer's recommendation.

8.3.3 To check the performance of the background correction system, measure the atomic background absorbance of 20 μL of 2 g/L magnesium nitrate solution at a wavelength in the 200 to 250 nm region (for example, Bi 223.1 nm) using a dry temperature of 120°C, a pyrolysis temperature of 950°C, and

TABLE 1 Program for Graphite Furnace Conditioning

Step	Temperature, °C	Ramp, s	Hold, s	Gas flow, mL/min
1	1500	60	20	300
2	20	1	10	300
3	2000	60	20	300
4	20	1	10	300
5	2600	60	10	300
6	20	1	10	300
7	2650	2	5	0

an atomization temperature of 1800°C. A large background signal should be observed with no over-or under-correction of the atomic signal.

NOTE 2—In general, Zeeman systems should compensate for background levels as high as 1.0 to 1.5 absorbance units. A continuum correction system should be able to correct for the broad-band background absorbance up to 0.5 to 0.6 absorbance units.

8.4 *Autosampler*—Check operation of the autosampler. Pay particular attention to the condition of the pipette tip and position of the tip during sample deposition. Clean the pipette tip with methanol. Adjust in accordance with the manufacturer's instructions.

NOTE 3—Use of an appropriate surfactant in the rinse water may enhance operation. If a surfactant is used, it should be checked for the presence of all the analytes to be determined.

9. Optimization of the Furnace Heating Program

9.1 Optimization of the furnace heating program is essential. Furnace programs recommended by the manufacturers are often designed for samples of a completely unrelated matrix. The analyst shall optimize the furnace program for a particular sample matrix (for example, steel, nickel alloys, etc.) and modifier system in accordance with the following procedure:

Furnace Step	Section
Drying	9.2
Pyrolysis	9.3
Atomization	9.4
Clean-out	9.5

9.2 *Drying Step*:

9.2.1 Select the graphite tube type (L'vov or platformless) and measurement mode (peak height or integrated peak area). Then select the same heating parameters used in 8.3.3. Optimize the drying parameters using any of the calibration solutions (see 5.3) and the procedure given in either 9.2.2 or 9.2.3.

9.2.2 *Samples Deposited on the Tube Wall*—For wall-deposited samples, a drying temperature of 120°C is satisfactory. To avoid spattering, a 20 s ramping time should be used to reach the 120°C temperature and then held at that temperature. The holding time will depend on the volume of the sample introduced. Typical holding times are as follows:

Injected Volume, μL	Holding Time, s
10	15
40	30

9.2.3 *Samples Deposited on the L'vov Platform*:

9.2.3.1 When using a L'vov platform, a two-stage drying process is beneficial to prevent spattering.

9.2.3.2 In the first stage, heat the sample rapidly to 80°C, using a 1 s ramp and then hold the temperature at 80°C for a short time. The holding time depends upon the volume of the solution injected. Typical holding times are shown in 9.2.2.

9.2.3.3 For the second stage, the temperature is ramped over a period of 20 to 30 s, to a value 20 to 40°C above the boiling point of the solvent. The holding times should be the same as given in 9.2.3.2.

9.2.4 In both cases, select a preliminary set of drying conditions and monitor the drying process visually with the aid