Designation: ~~E1770 – 14~~ E1770 – 19

Standard Practice for Optimization of ~~Electrothermal~~ Instrumentation for Graphite Furnace Atomic Absorption ~~Spectrometric Equipment~~Spectrometry[1]

This standard is issued under the fixed designation E1770; 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~~ graphite furnace atomic absorption spectrometers and the checking of spectrometer performance criteria.

1.2 The values stated in SI units are to be regarded as the standard.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

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]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data (Withdrawn 2003)[3]
E1184 Practice for Determination of Elements by Graphite Furnace Atomic Absorption Spectrometry
E1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis (Withdrawn 2005)[3]

3. Terminology

3.1 For definitions of terms used in this ~~test method,~~ practice, refer to Terminology E135.

4. Significance and Use

4.1 This practice is for optimizing the parameters used in the determination of trace elements in metals and alloys by the ~~electrothermal~~ graphite furnace 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.

5. Apparatus

5.1 *Atomic Absorption Spectrometer with ~~Electrothermal~~ Graphite Furnace Atomizer,* equipped with an appropriate background corrector, a signal output device such as a video display screen (VDS), a digital computer, a printer, and an autosampler.

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

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

5.3 *Pyrolytic Graphite Platforms,* L'vov design, fitted to the tubes specified in 5.2.

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

5.5 *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.~~

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

5.6 For general discussion of the theory and instrumental requirements of ~~electrothermal~~ graphite furnace atomic absorption spectrometric analysis, see Practice E1184.

6. Reagents

6.1 *Purity and Concentration of Reagents*—The purity and concentration of common chemical reagents shall conform to Practices E50. The reagents should be free of or contain minimal amounts (<0.01 µg/g) of the analyte of interest.

6.2 *Magnesium Nitrate Solution* ~~[2 g/L Mg(NO~~(2 g/L)~~$_3$)$_2$]~~—Dissolve 0.36 g ± 0.01 g high-purity $Mg(NO_3)_2$ ~~·6H~~•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. Alternatively, this solution may be purchased from a commercial supplier.

6.3 *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~~ the calibration solution with the next highest concentration; through S_k, the most concentrated calibration solution. Also refer to Practice E1452.

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

7. Initial Checks and Adjustments

7.1 Turn on power, cooling water, gas supplies, and fume exhaust system as required for the instrument being used.

7.2 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.

7.2.1 New graphite contacts or new tubes should be conditioned prior to use, ~~in accordance with~~as directed in the heating program recommended by the manufacturer.

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

8. Radiation Source

8.1 Install and operate hollow cathode lamps or electrodeless discharge lamps ~~in accordance with~~as directed in the manufacturer's instructions.

8.2 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.

9. Spectrometer Parameters

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

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

9.3 *Background Correction:*

9.3.1 *Zeeman Background Correction System:*

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

9.3.1.2 If necessary, set the optical temperature sensor ~~in accordance with~~as directed in the instrument manufacturer's instructions.

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

9.3.2 *Continuum Background System:*

9.3.2.1 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.

9.3.2.2 If necessary, set the optical temperature sensor ~~in accordance with~~as directed in the instrument manufacturer's recommendation.

9.3.3 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 nm to 250 nm region (for example, Bi 223.1 nm) using a dry temperature of ~~120°C,~~120 °C, a pyrolysis temperature of ~~950°C,~~950 °C, and an atomization temperature of ~~1800°C.~~1800 °C. A large background signal should be observed with no over-or under-correction of the atomic signal.

NOTE 1—In general, Zeeman systems should compensate for background levels as high as 1.0 absorbance unit to 1.5 absorbance units. A continuum correction system should be able to correct for the broad-band background absorbance up to 0.5 absorbance unit to 0.6 absorbance ~~units.~~unit.

9.4 *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~~as directed in the manufacturer's instructions.

NOTE 2—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.

10. Optimization of the Furnace Heating Program

10.1 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:~~as follows:

Furnace Step	Section
Drying	10.2
Pyrolysis	10.3
Atomization	10.4
Clean-out	10.5

10.2 *Drying Step:*

10.2.1 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 9.3.3. Optimize the drying parameters using any of the calibration solutions (see 6.3) and the procedure given in either 10.2.2 or 10.2.3.

10.2.2 *Samples Deposited on the Tube Wall*—For wall-deposited samples, a drying temperature of ~~120°C~~120 °C is satisfactory. To avoid spattering, a 20 s ramping time should be used to reach the ~~120°C~~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

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

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

10.2.3.2 In the first stage, heat the sample rapidly to ~~80°C,~~80 °C, using a 1 s ramp and then hold the temperature at ~~80°C~~80 °C for a short time. The holding time depends upon the volume of the solution injected. Typical holding times are shown in 10.2.2.

10.2.3.3 For the second stage, the temperature is ramped over a period of 20 s to 30 s, to a value ~~20~~20 °C to ~~40°C~~40 °C above the boiling point of the solvent. The holding times should be the same as given in 10.2.3.2.

10.2.4 In both cases, select a preliminary set of drying conditions and monitor the drying process visually with the aid of a dental mirror, to ensure that it proceeds without spattering. Hold the mirror directly above the sample introduction port (avoid touching the magnet), or near the end windows of the graphite tube. Observe vapor formation on the mirror as drying proceeds. Vapor evolution should cease at approximately 10 s before the end of the drying step. Adjust the hold times accordingly to accomplish this.

10.2.4.1 ~~Warning:~~**Warning—**To prevent serious eye injury, do not view the tube directly during the atomization or clean-out steps.

10.3 *Pyrolysis Step:*

10.3.1 During this step, volatile components of the matrix are driven off and precursory reactions occur (for example, reduction of the analyte oxide to the elemental state and the formation of matrix refractory oxides and carbides).

NOTE 3—Because of the low volatility of most metal alloy matrices, most of the matrix will remain in the furnace after the pyrolysis.

10.3.2 Use the optimum drying conditions as determined in 10.2.

~~NOTE 5—At this stage, both the optimum pyrolysis and atomization temperatures are unknown. An estimate of the suitable atomization temperature shall be made and entered into the instrument prior to optimization of the pyrolysis temperature.~~