Designation: E 1812 – 96

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

This standard is issued under the fixed designation E 1812; 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 flame 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.* For specific hazards associated with the use of this practice see Practices E 50 and warning statement included in this practice.

2. Referenced Documents

2.1 *ASTM Standards:*

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[2]

E 863 Practice for Describing Flame Atomic Absorption Spectroscopy Equipment[3]

E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and Spectroscopic Atomic Analysis[3]

3. Significance and Use

3.1 This practice is used for optimizing the parameters in the analysis of metals and alloys by flame atomic absorption spectrometry and also describes the practice of checking the spectrometer performance. The work is expected to be performed in a properly equipped laboratory, and appropriate disposal procedures are to be followed.

4. Apparatus

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

4.2 *Radiation Source*—Hollow cathode lamp or electrodeless discharge lamp for the analyte(s).

4.3 For a general discussion of the instrumental requirements for flame atomic absorption see Practice E 863.

4.4 For a general discussion of apparatus requirements see Practices E 50.

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 (<0.1 µg/g) of the analyte of interest.

5.2 *Calibration Solutions*—Refer to the preparation of calibration solutions in the relevant analytical method and 7.1.1 of this practice. Also refer to Practice E 1452.

6. Instrument Adjustments

6.1 Determine the flame type (air-acetylene or nitrous oxide/acetylene) to be used by the relevant analytical procedure. Set up the atomic absorption spectrometer to operate with the appropriate single slot laminar flow burner head in accordance with the manufacturer's instructions.

NOTE 1—**Warning:** A "flashback" may occur if a nitrous oxide/acetylene flame is used with burners not specifically designed for a nitrous oxide/acetylene flame, particularly on older or more basic instruments.

6.2 Use a single-element radiation source (hollow cathode or electrodeless discharge lamp) as the light source under test. Operate the lamp as directed by the manufacturer.

NOTE 2—The use of multielement lamps is not generally recommended, especially if one of the elements contained in them is iron. However, some binary alloy lamps give a more stable emission than single-element lamps.

6.3 Light the burner and aspirate water until a thermal equilibrium is reached. Pass a cleaning wire through the nebulizer. Check the burner slot for any buildup which may clog the burner.

6.4 Aspirate a mid-range calibration solution of the element being tested and adjust the instrument to give optimum absorption. Use the wavelength setting specified in the relevant analytical method. Use the slit setting or bandpass recommended by the instrument manufacturer for the element being tested and the wavelength specified. Adjust the burner heights and alignment for optimum absorption. The use of scale expansion may be necessary.

6.5 Adjust the nebulizer for maximum absorption.

6.6 Flush the burner system with HCl (1 + 19), HNO_3 (1 + 19), or deionized water and zero the instrument. Proceed

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[1] 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.

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[2] *Annual Book of ASTM Standards*, Vol 03.05.

[3] *Annual Book of ASTM Standards*, Vol 03.06.

with the instrument performance checks.

7. Instrument Performance Checks

7.1 The calibration solutions are prepared as described in the relevant analytical method. Normally five calibration solutions are used, including the one containing no analyte (S_0).

7.1.1 Two pairs of calibration solutions are required for the instrument performance check. One pair of calibration solutions is at the low end of the calibration graph, where the lower one is the "blank" solution containing no analyte (S_0), and the other one is the calibration solution containing the lowest amount of analyte (S_1). For the other pair, the two calibration solutions containing the two highest amounts of analyte are used (S_3 and S_4, or S_4 and S_5 if there are six calibration solutions in the set). The difference in the analyte contents between S_1 and S_0 has to be identical to the difference in the analyte contents between S_4 and S_3(or S_4 and S_5).

7.2 *Readability*:

7.2.1 Aspirate the two calibration solutions having the highest concentrations of the analyte under test. Record the instrument readings and calculate the difference.

7.2.2 Divide the difference between the readings by 20. The readability of the instrument is acceptable for the procedure if this result is not less than the smallest effective interval which can be read or estimated on the instrument readout.

7.3 *Linearity of Instrument Response*:

7.3.1 Aspirate the two calibration solutions at the low end of the calibration graph (S_0 and S_1). Record the readings and calculate the difference.

7.3.2 Divide the difference in the readings for the two calibration solutions of the highest concentration, as determined in 7.2.1, by the difference in the readings obtained between the two low concentration calibration solutions (S_0 and S_1).

7.3.3 The linearity of the instrument response for the procedure is acceptable if this ratio is 0.70 or greater.

7.3.4 If the ratio is less than 0.70, further adjustments to the instrument may give acceptable results. Otherwise the operating range of the method shall be reduced by lowering the concentration of the calibration solution of the highest concentration.

7.4 *Stability*:

7.4.1 Aspirate HCl (1 + 19) and zero the instrument.

7.4.2 Aspirate the calibration solution with the highest analyte concentration (S_4) and record the absorbance reading.

7.4.3 Aspirate HCl (1 + 19), HNO_3(1 + 19), or deionized water. Observe the absorbance reading on this solution. The absorbance reading should return to zero. If it does not return to zero, re-zero the instrument.

7.4.4 Repeat the measurement of the calibration solution with the highest analyte concentration six times, aspirating HCl (1 + 19), HNO_3(1 + 19), or deionized water between the readings but not adjusting any of the instrument settings.

7.4.5 The variability (*VA*), expressed as a percentage of the readings of the calibration solution with the highest analyte concentration is given by the following formula:

$$VA = 100\,[0.40(A_h - A_l)]/A \quad (1)$$

where:

A = average instrument reading for the calibration solution with the highest matrix concentration, calculated from the six readings,
A_h = highest of the six instrument readings, and
A_l = lowest of the six instrument readings.

NOTE 3—0.04 ($A_h - A_l$) is an estimation of the standard deviation.

7.4.6 The instrument meets the stability requirements if the variability is less than 1.5 %.

NOTE 4—This test can also be applied to other points on the calibration graph. It may also be applied to the evaluation of the stability of the instrument zero.

8. Keywords

8.1 atomic absorption spectrometry; flame atomic absorption spectrometry; instrument adjustment; instrument performance checks; readability; stability