



# Standard Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry<sup>1</sup>

This standard is issued under the fixed designation E 1024; 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 guide covers general recommendations for the use of atomic absorption spectrophotometers in the flame mode for the chemical analysis of Metals and metal bearing ores by ASTM methods.

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

E 863 Practice for Describing Flame Atomic Absorption Spectroscopy Equipment<sup>3</sup>

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

E 1812 Practice for Optimization of Flame Atomic Absorption Spectrometric Equipment<sup>3</sup>

## 3. Significance and Use

3.1 The precision and accuracy of atomic absorption analyses are influenced by many factors. Most important of these factors are the proper preparation of the test solutions, the proper selection and preparation of calibration solutions, and instrument-related factors, such as proper setting of instrument parameters, the proper sequence of calibration and test solution measurements, and correct interpretation of the instrument's response. An ASTM method that refers to this guide specifies the procedures for preparation of test and calibration solutions. This guide gives the instrument-related procedures for using the atomic absorption spectrophotometer in the flame mode and calculating the results.

3.2 Because atomic absorption spectrophotometers vary greatly in their sensitivity for any specific element, the con-

centration ranges for test and calibration solutions given in each method must be considered only a guide for analysts who are not familiar with the response of their instruments for the element to be determined. Those who know the optimum concentration range of their instrument should prepare calibration solutions based upon that knowledge.

3.3 This guide is based upon simple graphical or mathematical ratio interpolations of instrument response between calibration points. For this reason, the criterion for "curve linearity" is conservative in order to minimize the error introduced by a linear approximation to, or manual graphing of, a markedly curvilinear response. Other methods of relating concentration to instrument response (such as, microprocessor curve-fitting techniques) may be valid for concentrations higher than the limit posed by the "curve linearity" test in this guide. None of these methods have been universally adopted and each method that has been proposed achieves its highest precision only in dealing with certain cases. The methods given in this guide are universally applicable. If employed carefully, they will provide satisfactory precision and accuracy.

## 4. Interferences

4.1 Chemical and spectral interferences from sample matrix elements and solvent medium anions are common. The standard method that references this guide will have provided the appropriate remedy: prechemical isolation of the analyte species, the addition of matrix-modifying reagents, or the specification of special calibration procedures.

4.2 Ionization of the analyte element in the flame is a frequent problem since only ground-state atoms are measured. If this effect is significant, the standard method that references this guide will have specified the addition of ionization suppressant or other means to minimize it.

## 5. Concentration Range

5.1 Prepare the reference and calibration solution as directed in 7.1 and 7.2 and in accordance with Practice E 1452, using as a guide the concentrations suggested in the standard method.

5.2 Prepare the instrument and perform the instrument performance checks according to Practice E 1812.

5.3 If the criteria for readability and linearity are met, the initial concentration range may be considered acceptable. Proceed as directed in 6.1.5.

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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.

5.4 If adequate instrument response is not obtained, prepare a calibration solution to provide a higher concentration and repeat 6.1 and 6.2. If the linearity criterion is not met, prepare dilute standard solutions to provide a concentration range lower than that of the original standard solutions and repeat 6.1 and 6.2. If a concentration range cannot be found for which both criteria are met, performance of the instrument must be improved before it can be used.

5.5 Perform the stability test as directed in Practice E 1812. If the minimum requirements are not met with the selected calibration solutions, do not use the instrument with this method until the desired stability is obtained.

## 6. Apparatus

6.1 *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. Also refer to Practice E 863.

NOTE 1—If an autosampler is used that also makes dilutions automatically, the user shall initially, and frequently thereafter, check the accuracy and precision of the dilutions. If required, appropriate adjustment shall be made in accordance with the manufacturer's instructions, or by the manufacturer's service department to bring the unit within acceptable tolerances.

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

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

6.4 Optimum settings for the operating parameters of the atomic absorption spectrometer vary with the instrument used. Use the wavelength and instrument parameters recommended in the relevant standard method. Initially, parameters should be set as the instrument manufacturer recommends.

6.5 For general discussion of apparatus requirements see Practices E 50.

## 7. Calibration

7.1 *Reference Solution*—This solution contains all the acids and other additions made in the preparation of the sample solution. The ASTM method will specify if sample matrix matching additives, ionization suppressants, or other additions must be included.

7.2 *Calibration Solutions*—Five solutions, equally spaced in analyte concentration, are prepared in the same manner as the reference solution. A calibration curve that extends to about 0.5 absorbance without serious curvature is common. The method that references this guide will specify the addition of sample matrix matching additives, ionization suppressants, or other additions, if needed.

## 8. Measurements

### 8.1 Instrument Adjustments:

8.1.1 Set the parameters to the values obtained in 6.1, light the burner, and aspirate water until the instrument comes to thermal equilibrium.

8.1.2 Aspirate a high-calibration solution and adjust param-

eters to obtain optimum absorption.

8.1.3 Aspirate the reference solution and adjust the instrument to zero. Aspirate the calibration solutions and make a preliminary record of the readings.

8.2 Aspirate the test solution, which has been prepared in accordance with the relevant ASTM method, to determine its place in the order of increasing concentration of the calibration solutions. Proceed as specified in the method in accordance with either 8.3 or 8.4.

### 8.3 Graphical Procedure:

8.3.1 Aspirate the reference solution until a steady signal is obtained and adjust the instrument to zero. Aspirate the calibration solutions and test solution in order of increasing absorbance and record the reading for each.

8.3.2 Aspirate water to flush the system and proceed as directed in 8.3.1 at least twice more.

8.3.3 Prepare a calibration curve by plotting the averages of the values obtained for the calibration solutions against the concentrations of analyte.

8.3.4 Determine the concentration of analyte in the test solution from the calibration curve.

### 8.4 Ratio Procedure:

8.4.1 Prepare two more calibration solutions (one only, if the absorbance reading for the test solution falls close to one of the earlier calibration solutions) such that they closely bracket the test solution. The portion of the analytical graph between the two calibration solutions should effectively be a straight line.

8.4.2 With the instrument adjusted as in 8.1, aspirate the test solution and the closely bracketing calibration solutions in order of increasing absorbance without intervening water aspirations. Repeat at least twice and calculate the average absorbance values.

8.4.3 The concentration of the test solution may now be calculated by ratio:

$$C_t = C_1 + \left[ \frac{(A_t - A_1)}{(A_h - A_1)} \times (C_h - C_1) \right]$$

where:

$C_t$  = concentration of analyte in the test solution,

$C_h$  = concentration of analyte in the higher calibration solution,

$C_l$  = concentration of analyte in the lower calibration solution,

$A_t$  = absorbance reading of the test solution,

$A_h$  = absorbance reading of the higher calibration solution, and

$A_l$  = absorbance reading of the lower calibration solution.

### 8.5 Computerized Procedure:

8.5.1 If the instrument is provided with a microprocessor or a computer to calculate results, follow the instrument manufacturers' instructions.

## 9. Keywords

9.1 atomic absorption; flame

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