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Standard Practice for Flame Atomic Absorption Analysis¹

This standard is issued under the fixed designation E 663; 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.

⁶¹ NOTE—Section 12 was added editorially in July 1991.

1. Scope

1.1 This practice covers the use of an atomic absorption spectrophotometer (AAS) for determining the concentration of elements in solution by flame atomization. Recommendations are provided for preparing the instrument for making measurements, measuring the sensitivity, establishing criteria which should result in satisfactory instrument performance, determining the useful calibration concentration range, and measuring and calculating the test solution analyte concentration.

1.2 *This standard does not purport to address all of the safety problems, 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.* Specific precautionary statements are given in Section 6 and specific warning statements are given in Note 4.

2. Referenced Documents

2.1 ASTM Standards:

- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²
- E 131 Terminology Relating to Molecular Spectroscopy³
- E 135 Terminology Relating to Analytical Atomic Spectroscopy⁴
- E 863 Practice for Describing Flame Atomic Absorption Spectroscopy Equipment⁴

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology E 131 and E 135.

3.2 Description of Term Specific to This Standard:

3.2.1 *useful concentration range*—the useful concentration range is the range in analyte concentrations through which the precision of the method or the AAS, or both, are acceptable.

4. Significance and Use

4.1 This practice describes how to prepare and calibrate an AAS for the quantitative determination of elements in

solution by flame atomization. It includes the preparation of curves (Figs. 1 and 2) to establish the useful calibration range for the instrument and analytical method to be used. With these curves the operator can objectively determine the useful concentration range of a particular instrument. Once the useful concentration range is established for a specific atomic absorption procedure and instrument, repetition of the work is not required unless the performance of the instrument changes. These operations are common to and are a part of some basic atomic absorption methods.

5. Atomic Absorption Theory and Practice

5.1 In atomic absorption, a test solution is aspirated into a flame through which passes radiation from a line emission source of the element sought. The radiation of the element sought is absorbed in proportion to the concentration of its neutral atoms present in the flame. The concentration of the analyte is obtained by comparison to calibration solutions.

5.2 A theoretical basis for using atomic absorption to determine analyte concentration is described by Walsh (1).⁵ A broader more practical background can be found in a modern text on instrumental analysis such as that by Willard et al (2). A detailed discussion of atomic absorption theory and practice can be found in Dean and Rains (3).

6. Safety Precautions

6.1 Operating personnel should adhere to the manufacturer's recommended practice for igniting and extinguishing the burner on the atomic absorption spectrophotometer, to avoid an explosion which could cause physical injury. See Note 4. Also see Practices E 50 for further general safety precautions.

7. Preparation of Reference, Blank, and Calibration Solutions

7.1 *Reference Solution*—Combine all acids, reagents, and other additions present in the test solution and dilute to the same concentrations. This solution is used to set the zero absorbance or 100 % transmittance of the AAS.

7.2 *Blank Solution*—Incorporate in the reference solution all the specimen matrix elements of significant concentration, except the analyte, in the same concentrations as in the test solution.

NOTE 1—In practice it may not always be possible to obtain matrix elements containing nonmeasurable quantities of analyte. In such situations a correction must be applied as directed in 7.3 or 8.2.

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 14.01.

⁴ Annual Book of ASTM Standards, Vol 03.06.

⁵ The boldface numbers in parentheses refer to the list of references at the end of this practice.

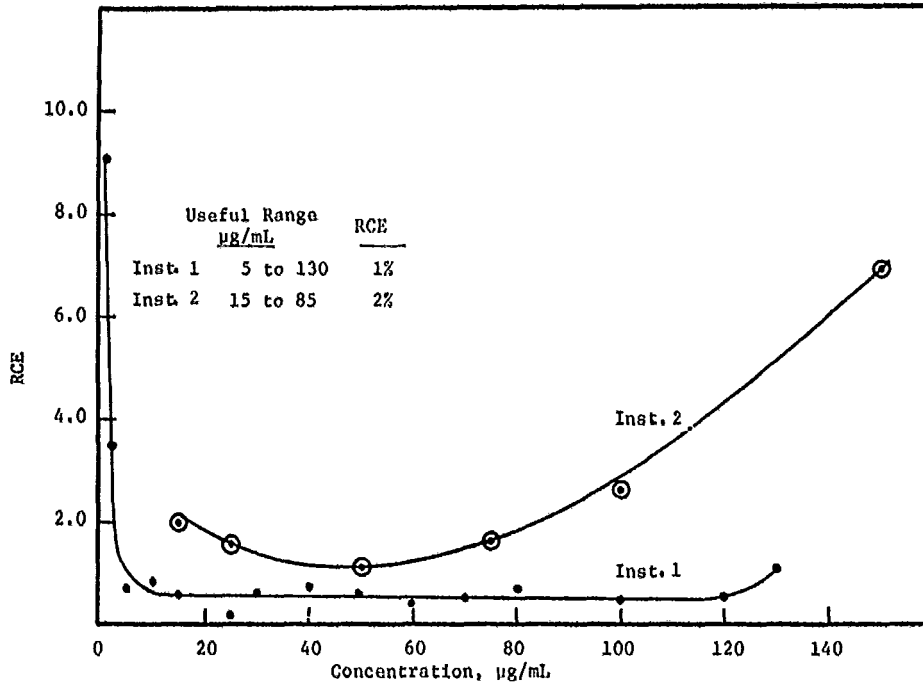


FIG. 1 RCE Versus Concentration (Lead in 2 % Nitric Acid, 283.3-nm Line)

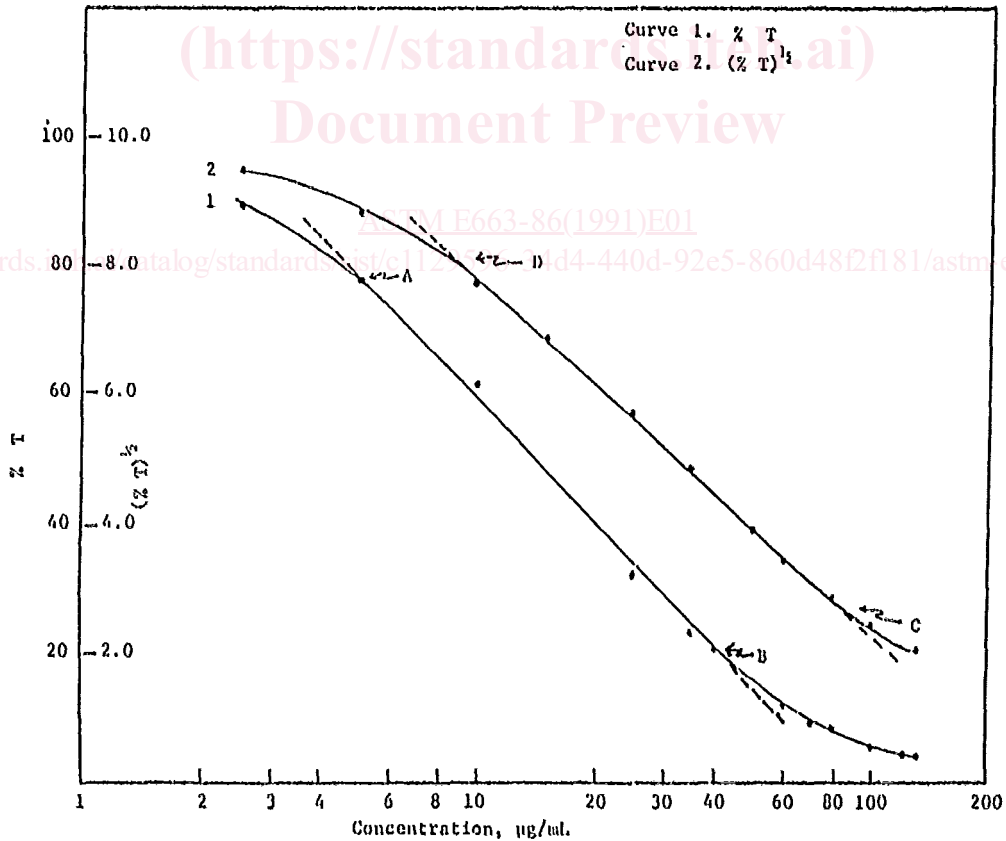


FIG. 2 Typical Curves (Lead in 2 % Nitric Acid, 283.3-nm Line)

7.3 Calibration Solutions—Using the manufacturer’s data as a guide and some trial and error, prepare a minimum of twelve calibration solutions to cover the absorbance range

0.005 to 1.5, or greater if needed. At least three of these solutions should be in the range 0.005 to 0.1 absorbance and three in the range 0.9 to 1.5. The remaining six solutions should be approximately equally spaced in terms of concentration between 0.1 and 0.9 absorbance. These calibration solutions should, as closely as possible, match the composition of the test solution. If the composition of the test solution is unknown to the extent that matching calibration solutions cannot be prepared, for example, other matrix impurities are concentrated along with the analyte, refer to the method of standard additions described in 10.4. If a measurable amount of the analyte is present in the blank solution, its absorbance reading must be converted to concentration and this amount must be added to the concentration of the analyte in these calibration solutions, when these solutions are used to establish the useful calibration range. When the calibration solutions are used to construct a working curve to measure the analyte in the test solution, the blank concentration may be subtracted from the analyte concentration or added to the calibration concentrations as directed by the basic method.

NOTE 2—The twelve solutions described here are used for preparing a precalibration curve to establish the useful range of the AAS. The working or calibration curve used to measure the test solution will usually contain fewer calibration solutions, but may include some of these twelve solutions.

NOTE 3—Matching the chemical composition of calibration and test solutions to eliminate matrix effects may not be necessary if the weight of the sample in the solution is less than 0.1 % on a weight per unit volume basis. For test specimens of this type the analysis of standard reference materials, or of test solutions to which further additions of the analyte have been made, will reveal the presence of a matrix effect as evidenced by a significant difference in results. If a matrix effect is absent the calibration solutions need contain nothing more than known amounts of the analyte in the same solvent as the sample.

8. Preparation of Apparatus

8.1 *Atomic Absorption Spectrophotometer*—Select the instrument operating parameters including wavelength of the analyte element line emission source, support and fuel gases, and slit width for the element being determined by referring to the instrument manufacturer's instructions or by referring to the basic analytical method that is being used (see Practice E 863). Turn on the appropriate support and fuel gases, and ignite the burner. Aspirate water until a stable signal is obtained (that is, until the absorbance or transmittance reaches a plateau value). Adjust the readout system to obtain zero absorbance of 100 % transmittance while aspirating the reference solution prepared as directed in 7.1. Continue the aspiration until a stable signal is obtained. Aspirate a calibration solution, prepared as described in 7.3, containing the analyte at a concentration that will yield an absorbance of 0.2 to 0.4, and at a concentration that is expected to be within the linear absorbance range of the method. Refer to the instrument manufacturer's instruction manual for guidance to determine an approximation of the linear concentration range for the analyte, and for the following mechanical adjustments: Adjust iteratively the zero absorbance using the reference solution, the burner height and its horizontal position, the flow rate of the fuel gases, and the nebulizer position for maximum absorbance of the selected calibration solution. Obtain three measurements of the absorbance of the calibration solution, washing with solvent or reference

solution between each measurement. In the same way, make three measurements of the blank solution. Average each of these measurement sets and use the average to calculate the blank and the sensitivity as directed in 8.2 and 8.3, respectively.

NOTE 4: Warning—Follow the manufacturer's recommended practice for igniting and extinguishing the burner to avoid an explosion which will damage the burner chamber or cause physical harm to operating personnel.

8.2 *Blank*—Calculate an analyte concentration for the blank as follows:

$$C_B = A_B(C_1/A_1) \quad (1)$$

where:

C_B = concentration of analyte in the blank,
 C_1 = concentration of analyte in the calibration solution,
 A_B = average absorbance of C_B , and
 A_1 = average absorbance of C_1 .

The concentration of analyte in the blank is added to the concentration of analyte in the calibration solutions as directed in 7.3.

NOTE 5—The blank solution may be substituted for the reference solution when the respective absorbances are essentially the same, analyte concentration is not measurable with an AAS, and errors in operation cancel.

8.3 *Sensitivity*—The sensitivity (S) is the concentration of analyte that gives an absorption of 1 % or an absorbance of 0.0044. The smaller this sensitivity value the more sensitive is the AAS. To determine if the AAS has adequate response, calculate from the data obtained in 8.1 the sensitivity as follows:

$$S = 0.0044 (C_1/A_1) \quad (2)$$

where C_1 and A_1 have the same meaning as in Eq 1. Determine if sensitivity is adequate by reference to the manufacturer's handbook or to the basic analytical method. Sensitivity should not be less than 75 % of the required value.

8.4 *Precision of Measurements*—Use the following procedure to determine if the instrument precision is acceptable: Set the absorbance to zero while aspirating a reference solution prepared as specified in 7.1. Obtain an absorbance or concentration measurement on the calibration solution used in 8.1. Repeat the measurement sequence for the reference and the calibration solutions, alternating with solvent flushing between measurements to obtain six readings of absorbance for the calibration solution. Calculate the *standard deviation* of the readings made on this calibration solution by applying acceptable statistical methods. If the *standard deviation* is greater than 1 % of the average absorbance of the calibration solution, determine the cause of the variability (for example, deposits in the burner or clogged capillary), and make the appropriate changes. If unacceptable precision is caused by a large amount of dissolved solids in the calibration and test solutions, make an appropriate dilution to prevent an erratic flame or clogging of the nebulizer.

NOTE 6—An approximation of the *standard deviation* (s) can be obtained with less calculation as follows:

$$s = (A - B) \times 0.40 \quad (3)$$