Designation: E60 – 11 (Reapproved 2022)ε1

Standard Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry[1]

This standard is issued under the fixed designation E60; 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.

ε1 NOTE—Editorial changes were made throughout in July 2022.

1. Scope

1.1 This practice covers general recommendations for photoelectric photometers and spectrophotometers and for photometric practice prescribed in ASTM methods for chemical analysis of metals, sufficient to supplement adequately the ASTM methods. A summary of the fundamental theory and practice of photometry is given. No attempt has been made, however, to include in this practice a description of every apparatus or to present recommendations on every detail of practice in ASTM photometric or spectrophotometric methods of chemical analysis of metals.[2]

1.2 These recommendations are intended to apply to the ASTM photometric and spectrophotometric methods for chemical analysis of metals when such standards make definite reference to this practice, as covered in Section 4.

1.3 In this practice, the terms "photometric" and "photometry" encompass both filter photometers and spectrophotometers, while "spectrophotometry" is reserved for spectrophotometers alone.

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

1.5 *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:*[3]

E131 Terminology Relating to Molecular Spectroscopy

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E168 Practices for General Techniques of Infrared Quantitative Analysis

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Definitions and Symbols

3.1 For definitions of terms relating to this practice, refer to Terminology E135.

3.2 For definitions of terms relating to absorption spectroscopy, refer to Terminology E131.

3.3 *Definitions of Terms Specific to this Practice:*

3.3.1 *background absorption*—any absorption in the solution due to the presence of absorbing ions, molecules, or complexes of elements other than that being determined is called background absorption.

3.3.2 *concentration range*—the recommended concentration range shall be designated on the basis of the optical path of the cell, in centimetres, and the final volume of solution as recommended in a procedure. In general, the concentration range and path length shall be specified as that which will produce transmittance readings in the optimum range of the instrument being used as covered in Section 14.

3.3.3 *initial setting*—the initial setting is the photometric reading (usually 100 on the percentage scale or zero on the logarithmic (absorbance) scale) to which the instrument is adjusted with the reference solution in the absorption cell. The scale will then read directly in percentage transmittance or in absorbance.

[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 additional information on the theory and photoelectric photometry, see the list of references at the end of this practice.

[3] 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.

3.3.4 *photometric reading*—the term "photometric reading" refers to the scale reading of the instrument being used. Available instruments have scales calibrated in transmittance, *T*, **(1)**[4] or absorbance, *A*, **(2)** (see 5.2), or even arbitrary units proportional to transmittance or absorbance.

3.3.5 *reagent blank*—the reagent blank determination yields a value for the apparent concentration of the element sought, which is due only to the reagents used. It reflects both the amount of the element sought present as an impurity in the reagents, and the effect of interfering species.

3.3.6 *reference solution*—photometric readings consist of a comparison of the intensities of the radiant energy transmitted by the absorbing solution and the radiant energy transmitted by the solvent. Any solution to which the transmittance of the absorbing solution of the substance being measured is compared shall be known as the reference solution.

4. Reference to This Practice in Standards

4.1 The inclusion of the following paragraph, or a suitable equivalent, in any ASTM test method (preferably after the section on scope) shall constitute due notification that the photometers, spectrophotometers, and photometric practice prescribed in that test method are subject to the recommendations set forth in this practice.

"Photometers, Spectrophotometers, and Photometric Practice—Photometers, spectrophotometers, and photometric practice prescribed in this test method shall conform to ASTM Practice E60, Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry.

5. Theory

5.1 Photoelectric photometry is based on Bouguer's and Beer's (or the Lambert-Beer) laws which are combined in the following expression:

$$P = P_o 10^{-abc}$$

where:

P = transmitted radiant power,
P_o = incident radiant power, or a quantity proportional to it, as measured with pure solvent in the beam,
a = absorptivity, a constant characteristic of the solution and the frequency of the incident radiant energy,
b = internal cell length (usually in centimetres) of the column of absorbing material, and
c = concentration of the absorbing substance, g/L.

5.2 Transmittance, *T*, and absorbance, *A*, have the following values:

$$T = P/P_o$$
$$A = \log_{10} (1/T) = \log_{10} (P_o/P)$$

where P and P_o have the values given in 5.1.

5.3 From the transposed form of the Bouguer-Beer equation, $A = abc$, it is evident that at constant *b*, a plot of *A versus c* gives a straight line if Beer's law is followed. This line will pass through the origin if the practice of cancelling out solvent reflections and absorption and other blanks is employed.

5.4 In photometry it is customary to make indirect comparison with solutions of known concentration by means of calibration curves or charts. When Beer's law is obeyed and when a satisfactory instrument is employed, it is possible to dispense with the curve or chart. Thus, from the transposed form of the Bouguer-Beer law, $c = A/ab$, it is evident that once *a* has been determined for any system, *c* can be obtained, since *b* is known and *A* can be measured.

5.5 The value for *a* can be obtained from the equation $a = A/cb$ by substituting the measured value of *A* for a given *b* and *c*. Theoretically, in the determination of *a* for an absorbing system, a single measurement at a given wavelength on a solution of known concentration will suffice. However, it is better to use the average value obtained with three or more concentrations, covering the range over which the determinations are likely to be made and making several readings at each concentration. The validity of the Bouguer-Beer law for a particular system can be tested by showing that *a* remains constant when *b* and *c* are changed.

APPARATUS

6. General Requirements for Photometers and Spectrophotometers

6.1 A photoelectric photometer consists essentially of the following:

NOTE 1—The choice of an instrument may naturally be based on price considerations, since there is no point in using a more elaborate (and, incidentally, more expensive) instrument than is necessary. In addition to satisfactory performance from the purely physical standpoint, the instrument should be compact, rugged enough to stand routine use, and not require too much manipulation. The scales should be easily read, and the absorption cells should be easily removed and replaced, as the clearing, refilling, and placing of the cells in the instrument consume a major portion of the time required. It is advantageous to have an instrument that permits the use of cells of different depth (see Practice E275).

6.1.1 An illuminant (radiant energy source),

6.1.2 A device for selecting relatively monochromatic radiant energy (consisting of a diffraction grating or a prism with selection slit, or a filter),

6.1.3 One or more absorption cells to hold the sample, calibration, reagent blank, or reference solutions, and

6.1.4 An arrangement for photometric measurement of the intensity of the transmitted radiant energy, consisting of one or more photocells or photosensitive tubes, and suitable devices for measuring current or potential.

6.2 Precision instruments that employ monochromators capable of supplying radiant energy of high purity at any chosen wavelength within their range are usually referred to as spectrophotometers. Instruments employing filters are known as filter photometers or abridged spectrophotometers, and usually isolate relatively broad bands of radiant energy. Frequently the absorption peak of the compound being measured is relatively broad, and sufficient accuracy can be obtained using a fairly broad band (10 nm to 75 nm) of radiant energy for the measurement (Note 2). Other times the absorption

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.