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Standard Practice for Use of a Ceric-Cerous Sulfate Dosimetry System¹

This standard is issued under the fixed designation ISO/ASTM 51205; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision.

1. Scope

1.1 This practice covers the procedures for preparation, testing, and procedure for using the ceric-cerous sulfate dosimetry system to determinemeasure absorbed dose (in terms of absorbed dose to water) in materials irradiated by photons (gamma radiation or X-radiation/bremsstrahlung) or high-energy electrons. to water when exposed to ionizing radiation. The system consists of a dosimeter and appropriate analytical instrumentation. For simplicity, the system will be referred to as the ceric-cerous system. It The ceric-cerous dosimeter is classified as a reference-standard dosimetry system (see ISO/ASTM Guidetype 1 dosimeter on the basis 51261). Cerie-cerous dosimeters are also used as transfer-standard dosimeters or routine dosimeters of the effect of influence quantities. The ceric-cerous system may be used as a reference standard dosimetry system or as a routine dosimetry system.

<u>1.2 This document is one of a set of standards that provides recommendations for properly implementing dosimetry in radiation processing, and describes a means of achieving compliance with the requirements of ISO/ASTM Practice 52628 for the ceric-cerous system. It is intended to be read in conjunction with ISO/ASTM Practice 52628.</u>

1.3 This practice describes both the spectrophotometric and the potentiometric readout procedures for the ceric-cerous system.

1.4 This practice applies only to gamma radiation, X-radiation/bremsstrahlung, and high energy electrons.

1.5 This practice applies provided the following conditions are satisfied:

1.5.1 The absorbed-dose range is between 0.5 and 50 from 5×10^2 kGy to 5×10^4 Gy (1).²

1.5.2 The absorbed-dose rate is less than does not exceed 10^6 Gy s⁻¹ (1).

1.5.3 For radionuclide gamma-ray sources, the initial photon energy is greater than 0.6 MeV. For bremsstrahlung photons, the initial energy of the electrons used to produce the bremsstrahlung photons is equal to or greater than 2 MeV. For electron beams, the initial electron energy is greater than 8 MeV.

NOTE 1—The lower energy limits are appropriate for a cylindrical dosimeter ampoule of 12-mm diameter. Corrections for dose <u>gradientsgradient</u> across an ampoule of that diameter or less are not required for photons, but <u>the ampoule</u> may be required for electron beams (2). The ceric-cerous system may be used at lower energies by employing thinner (in the beam direction) dosimeters.<u>dosimeters</u> (see ICRU Report 35).

1.5.4 The irradiation temperature of the dosimeter is above 0° C and below 62° C (3).

Note 2—The temperature $\frac{\text{dependence}_{\text{coefficient}}}{\text{dependence}_{\text{coefficient}}}$ of dosimeter response is known only in this range (see $\frac{4.35.2}{1.2}$). Use outside this range requires determination of the temperature $\frac{\text{dependence}_{\text{coefficient}}}{1.2}$.

1.6 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.

<u>1.7 This international standard was developed in accordance with internationally recognized principles on standardization</u> 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.

¹ This <u>guidepractice</u> is under the jurisdiction of ASTM Committee E61 on Radiation Processing and is the direct responsibility of Subcommittee E61.02 on Dosimetry Systems, and is also under the jurisdiction of ISO/TC 85/WG 3.

Current edition approved June 18, 2008. Published June 2009. March 8, 2017. Published May 2017. Originally published as ASTM E1205–88. Last previous ASTM edition E1205–99. ASTM E1205–93 was adopted by ISO in 1998 with the intermediate designation ISO 15555:1998(E). The present International Standard ISO/ASTM 51205:2009(E)51205:2017(E) is a major revision of ISO/ASTM 51205-2002(E) which replaced ISO 15555.51205-2009(E). DOI:10.1520/ISOASTM51205-17.

² The boldface numbers in parentheses refer to the bibliography at the end of this standard.



2. Referenced documents

2.1 ASTM Standards:³ C912 Practice for Designing a Process for Cleaning Technical Glasses E170 Terminology Relating to Radiation Measurements and Dosimetry E178 Practice for Dealing With Outlying Observations E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers E666 Practice for Calculating Absorbed Dose From Gamma or X Radiation E668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices E925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Bandwidth does not Exceed 2 nm E958 Practice for Estimation of the Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers 2.2 ISO/ASTM Standards:³ 51261 GuidePractice for Selection and Calibration of Routine Dosimetry Systems for Radiation Processing 51707 Guide for Estimation of Measurement Uncertainty in Dosimetry for Radiation Processing 5140052628 Practice for Characterization and Performance of a High-Dose Radiation Dosimetry Calibration LaboratoryDosimetry in Radiation Processing 5170752701 Guide for Estimating Uncertainties in Dosimetry for Performance Characterization of Dosimeters and Dosimetry Systems for Use in Radiation Processing 2.3 ISO Standards:⁴ 12749-4 Nuclear energy - Vocabulary - Part 4: Dosimetry for radiation processing 2.4 ISO/IEC Standards:⁴ 17025 General Requirements for the Competence of Testing and Calibration Laboratories 2.5 Joint Committee for Guides in Metrology (JCGM) Reports: JCGM 100:2008, GUM 1995, with minor corrections, Evaluation of measurement data - Guide to the Expression of Uncertainty in Measurement⁵ JCGM 200:2012 (JCGM 200:2008 with minor revisions), VIM, International Vocabulary of Metrology - Basis and General Concepts and Associated Terms⁶ 2.6 International Commission on Radiation Units and Measurements (ICRU) Reports:⁷ ICRU Report 1410b (NBS Handbook 85) Radiation Dosimetry: X-Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 60 MeVPhysical Aspects of Irradiation ICRU Report 34 The Dosimetry of Pulsed Radiation ICRU Report 35 Radiation Dosimetry: Electrons Electron Beams with Initial Energies Between 1 and 50 MeV ICRU Report 3780 Stopping Powers for Electrons and Positrons Dosimetry Systems for Use in Radiation Processing

ICRU Report 6085a RadiationFundamental Quantities and Units for Ionizing Radiation ee2ad //astm-iso-astm51205-17

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration*<u>approved laboratory</u><u>set of operations under specified conditions, which establishes the relationship between values indicated by a measuring instrument or measuring system, and the corresponding values realized by standards traceable to a nationally or internationally recognized laboratory.]aboratory that is a recognized national metrology institute, or has been formally accredited to ISO/IEC 17025, or has a quality system consistent with the requirements of ISO/IEC 17025.</u>

3.1.1.1 Discussion—

Calibration conditions include environmental and irradiation conditions present during irradiation, storage and measurement of the dosimeters that are used for the generation of a calibration curve. To achieve stable environmental conditions, it may be necessary to condition the dosimeters before performing the calibration procedure. A recognized national metrology institute or other calibration laboratory accredited to ISO/IEC 17025 should be used in order to ensure traceability to a national or international

³ For referenced ASTM and ISO/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.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

⁵Document produced by Working Group 1 of the Joint Committee for Guides in Metrology (JCGM WG1), Available free of charge at the BIPM website (http://www.bipm.org).

⁶Document produced by Working Group 2 of the Joint Committee for Guides in Metrology (JCGM WG2), Available free of charge at the BIPM website (http://www.bipm.org).

⁷ Available from International Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814, USA.



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standard. A calibration certificate provided by a laboratory not having formal recognition or accreditation will not necessarily be proof of traceability to a national or international standard.

3.1.2 calibration curve—graphical representation of the dosimetry system's response function.

3.1.2 *ceric-cerous dosimeter*—specially prepared solution of ceric sulfate and cerous sulfate in sulfuric acid, individually sealed in an appropriate container such as a glass ampoule, where the radiation-induced changes in electropotential or optical absorbance of the solution are related to absorbed dose to water.

3.1.4 *measurement quality assurance plan*—documented program for the measurement process that ensures that the expanded uncertainty consistently meets the requirements of the specific application. This plan requires traceability to nationally or internationally recognized standards.

3.1.3 molar linear absorption coefficient, $\varepsilon_{\rm m}$ —constant relating the spectrophotometric absorbance, A_{λ} , of an optically absorbing molecular species at a given wavelength, λ , per unit pathlength, d, to the molar concentration, c, of that species in solution:

$$\varepsilon_{\rm m} = \frac{A_{\lambda}}{d \cdot c} \tag{1}$$

SI unit: m² mol⁻¹

3.1.3.1 Discussion-

The measurement is sometimes expressed in units of $L \text{ mol}^{-1} \text{ cm}^{-1}$.

3.1.6 net absorbance, ΔA —change in measured optical absorbance at a selected wavelength determined as the absolute difference between the pre-irradiation absorbance, A_o , and the post-irradiation absorbance, A, as follows:

$$\Delta A = \left| A - A_o \right| \tag{2}$$

3.1.4 radiation chemical yield, G(x)—quotient of n(x) by \bar{c} , where n(x) is the mean amount of a specified entity, x, produced, destroyed, or changed by the mean energy, \bar{c} , imparted to the matter.

(https://stag(x) =
$$\frac{n(x)}{\overline{\epsilon}}$$
 of s.iteh.ai) (2)

SI unit: mol J^{-1}

<u>3.1.5 reference standard dosimetry system</u>—dosimetry system, generally having the highest metrological quality available at a given location or in a given organization, from which measurements made there are derived.

3.1.6 *reference-standard* <u>type 1</u> dosimeter—dosimeter of high metrological quality used as a standard to provide measurements traceable to measurements made using primary-standard dosimeters.quality, the response of which is affected by individual influence quantities in a well-defined way that can be expressed in terms of independent correction factors.

3.1.9 response function—mathematical representation of the relationship between dosimeter response and absorbed dose, for a given dosimetry system.

3.1.10 routine dosimeter-dosimeter calibrated against a primary-, reference-, or transfer-standard dosimeter and used for routine absorbed-dose measurements.

3.1.11 *transfer_standard dosimeter*—a dosimeter, often a reference-standard dosimeter suitable for transport between different locations, used to compare absorbed-dose measurements.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *electropotential, E*—difference in potential between the solutions in the two compartments of an electrochemical cell, measured in millivolts.

3.3 For definitions Definitions of other terms used in this practice that pertain to radiation measurement and dosimetry, refer to ASTM Terminologydosimetry may be found in E170. Definitions in ISO 12749-4, ASTM Terminology E170 are compatible with ICRU 60; that document, ICRU 85a, and VIM; these documents, therefore, may be used as an alternative reference.references.

4. Significance and use

4.1 The ceric-cerous system provides a reliable means for determining absorbed dose to water. It is based on a process of reduction of ceric ions to cerous ions in acidic aqueous solution by ionizing radiation (1, 4)-, ICRU Report 80).

NOTE 3—The ceric-cerous system described in the practice has cerous sulfate added to the initial solution to reduce the effect of organic impurities and to allow the potentiometric method of measurement. Other systems used for dosimetry include solutions of ceric sulfate or ceric ammonium sulfate in sulfuric acid without the initial addition of cerous sulfate. These other systems are based on the same process of reduction of ceric ions to cerous ions but are not included in this practice.

4.2 The dosimeter is a solution of ceric sulfate and cerous sulfate in sulfuric acid in an appropriate container such as a flame-sealed glass ampoule. The solution indicates a level of absorbed dose by a change (decrease) in optical absorbance at a

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specified wavelength in the ultraviolet region, or a change (increase) in electropotential. A calibrated spectrophotometer is used to determine the absorbance and a potentiometer, with a specially designed cell, is used to determine the electropotential in millivolts.

4.3 The dosimeter response has an irradiation temperature dependence since the radiation chemical yield ($G(Ce^{3+})$) depends on temperature. The dependence of $G(Ce^{3+})$ is approximately equal to -0.2 % per degree Celsius between 0 and 62°C (3, 5, 6). This irradiation temperature dependence has a slight dependence on the initial cerous ion concentration (see 10.6.3).

4.4 The absorbed dose to materials other than water when irradiated under equivalent conditions may be calculated. Procedures for making such calculations are given in ASTM Practices E666 and E668 and ISO/ASTM Guide 51261.

Note 4—For a comprehensive discussion of various dosimetry methods applicable to the radiation types and energies discussed in this practice, see ICRU Reports 14, 34, 35, and 37.

5. Effect of influence quantities

5.1 Guidance on the determination of the performance characteristics of dosimeters and dosimetry systems can be found in ASTM Guide 52701. The relevant quantities that need to be considered when using the ceric-cerous dosimetry system are given below.

<u>5.2 The dosimeter response has a temperature dependence during irradiation that is approximately equal to -0.2 % per degree Celsius between 0 and 62°C (**3**, **5**, **6**). This irradiation temperature dependence has a slight dependence on the initial cerous ion concentration (see 10.6.2).</u>

<u>5.3</u> The electropotential, E, within the electrochemical cell, has a positive temperature coefficient of 0.33 % per °C between 25°C and 30°C and corrections are required for differences between measurement temperatures and the reference temperature used during calibration (see 10.5.8)

5.4 No effect of ambient light (even direct sunlight) has been observed on dosimetric solutions in glass ampoules.

5.5 The dosimeter response is dependent on the type and energy of the radiation employed. Since cerium is a heavy element from the viewpoint of absorption characteristics of gamma radiation, the response of the dosimetric solution for lower energy degraded radiation during use my be greater than the response in the cobalt-60 radiation during calibration (7). However, studies in an industrial gamma irradiator indicate that this effect is small (8).

5.6 If the dosimetric solution is prepared as described in this document, and steps are taken to avoid contamination, the dosimetric solution stored, or sealed, in glass vessels (for example, ampoules) is stable for several years before and after irradiation.

6. Interferences

6.1 The ceric-cerous dosimetric solution response is sensitive to impurities, particularly organic impurities. Even in trace quantities, impurities can cause a detectable change in the observed response (79). Organic materials shallshould not be used for any component in contact with the solution unless they have been tested and shown to have no effect. The effect of trace impurities is minimized by the addition of cerous ions to the solution (810, 911). Water purification methods found to be adequate for use in preparing ceric-cerous dosimeters are decribed in 7.28.2.

6.2 Undesirable chemical changes in the dosimetric solution can occur if care is not taken during sealing of the ampoules (see 8.79.7).

7. Apparatus

7.1 Spectrophotometric Method—For the analysis of the dosimetric solution, use a high-precision spectrophotometer capable of measuring absorbance values up to two with an uncertainty of no more than 1 % in the <u>analysis wavelength</u> region from 254 to 320 nm. Use quartz_nm should be used. Quartz_cuvettes with 10-mm path length should be used for spectrophotometric measurements of absorbance of the solution.

7.2 *Potentiometric Method*—Use an <u>An</u> electrochemical cell, similar to that described in <u>Annex A1</u>, <u>should be used</u> (see Fig. A1.1). <u>Measure the <u>The</u> electropotential across the cell <u>should be measured</u> with a high-precision digital <u>potentiometervoltmeter</u> that is capable of measuring dc potentials in the range from 1 to 100 mV within an uncertainty of 1 %.</u>

NOTE 4—As shown in Fig. A1.1, the electrochemical cell has two compartments separated by a porous junction, such as a glass frit, a ceramic or kaolin junction, or a fibreglass wick. The inner compartment is <u>always</u> filled with unirradiated solution. The lower compartment is filled with solution transferred whose response is to be measured (transferred from an irradiated or unirradiated ampoule: ampoule). The electropotential, *E*, generated between the platinum electrodes in the two compartments is measured by a digital potentiometer.voltmeter.

7.3 *Glassware*—Use borosilicate Borosilicate glass or equivalent chemically resistant glass <u>should be used</u> to store the reagents and the prepared dosimetric solution. <u>Clean all All glassware</u>, except ampoules, <u>should be cleaned</u> using chromic acid cleaning solution or an equivalent cleaning agent (see ASTM Practice C912). <u>Rinse Glassware should be rinsed</u> at least three times with <u>double-distilled water</u>. <u>Drypurified water</u>, <u>dried</u> thoroughly and <u>storestored</u> under conditions that will minimize exposure to dust.



7.4 *Glass Ampoules*—If required, elean glass ampoules <u>should be cleaned</u> in boiling <u>double-distilled water</u>. <u>Rinsepurified water</u>, <u>rinsed</u> twice with <u>double-distilled water</u> and oven <u>dry.dried</u>.

NOTE 5—The dosimetric ampoule normally used has a capacity of approximately 2 mL. Quick-break glass ampoules, or Type 1 glass colorbreak ampoules or equivalent containers, are commonly used. Commercially available ampoules have been found to give reproducible results without requiring additional cleaning.

8. Reagents

8.1 Analytical reagent grade (or better) chemicals shall be used for preparing all solutions.⁸

8.2 Water quality is very important since it is the major component of the dosimetric solutions, and therefore may be the prime source of contamination. The water quality is more important for cerie-cerous dosimeters used for measurements in the lower absorbed-dose range than for those used in the upper absorbed-dose range. For high-range dosimeters double-distilled water Double-distilled water from coupled all-glass and silica stills ean be used. For low-range dosimeters, use triply-distilled water. Alternatively, use or water from a high-quality commercial purification unit capable of achieving Total Oxidizable Carbon (T.O.C.) content below 5 ppb. ppb should be used. Use of deionized water is not recommended.

Note 6—Double-distilled water distilled from an alkaline potassium permanganate ($KMnO_4$) solution (2 g KMnO_4 plus 5 g sodium hydroxide (NaOH) pellets in 2 L of distilled water) has been found to be adequate for routine preparation of the dosimetric solution. High-purity water is commercially available from some suppliers. Such water labeled HPLC (high-pressure liquid chromatographic) grade is usually sufficiently free from organics to be used in this practice.

8.3 Do not store purified Purified water used in this practice should not be stored in plastic containers or in containers with plastic caps or plastic cap liners.

9. Preparation of the dosimeters

9.1 The recommended Recommended concentrations for the ceric-cerous dosimeter to measure for measurement of absorbed doses from about 5 to 50 kGy (high-range dosimeter) are 15 mmol dm⁻³ceric sulfate [Ce(SO₄)₂ · 4H₂O] and 15 mmol dm⁻³ cerous sulfate [Ce₂(SO₄)₃ · 8H₂O]. For measurement of absorbed doses from about 0.5 to 10 kGy (low-range dosimeter), the recommended concentrations are 3 mmol dm⁻³ [Ce(SO₄)₂ · 4H₂O] and 3 mmol dm⁻³ [Ce₂(SO₄)₃ · 8H₂O].

9.2 The dosimetric solutions specified in 8.19.1 may be formulated from the following nominal stock solutions: (a) 0.4 mol dm⁻³ and 4 mol dm⁻³ sulfuric acid (H₂SO₄), (b) 0.1 mol dm⁻³ Ce(SO₄)₂ · 4H₂O, and (c) 0.1 mol dm⁻³ Ce₂(SO₄)₃ · 8H₂O. Procedures for preparing these solutions are given in Annex A2. (Warning—Concentrated sulfuric acid is corrosive and can cause serious burns. Ceric-cerous solutions are skin irritants. Appropriate precautions should be exercised in handling these materials.)

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https://standards.iteh.ai/catalog/standards/sist/d8c5d990-7c4d-47d2-bd54-4dac51ee2ad1/astm-iso-astm51205-17

⁸ Reagent specifications are available from American Chemical Society, 1115 16th St., Northwest, Washington, DC 20036, USA.

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9.3 Use the following equations to determine the volume in millilitres of each stock solution necessary to prepare 1 L of dosimetric solution:

High Range	Low Range
$\frac{V_1}{1000} = \frac{0.015}{c_1}$	$\frac{V_1}{1000} = \frac{0.003}{c_1} $ (3)
$\frac{V_2}{1000} = \frac{0.015}{c_2}$	$\frac{V_2}{1000} = \frac{0.003}{c_2} $ (4)
$\frac{V_3}{1000 - V_1} = \frac{0.4}{c_3}$	$\frac{V_3}{1000 - V_1} = \frac{0.4}{c_3} \tag{5}$
$V_4 = 1000 - V_1 - V_2 - V_3$	$V_4 = 1000 - V_1 - V_2 - V_3 \tag{6}$

where:

 V_1

- = volume of nominal 0.1 mol dm⁻³ ceric-sulfate stock solution, = volume of nominal 0.1 mol dm⁻³ cerous-sulfate stock solution, V_2
- = volume of nominal 4 mol dm^{-3} sulfuric-acid stock solution, V_3
- $\dot{V_4}$ = volume of distilled water,
- V_4 <u>= volume of purified water,</u>
- = actual concentration of the ceric-sulfate stock solution, c_1
- = actual concentration of the cerous-sulfate stock solution, and C_2
- = actual concentration of the nominal 4 mol dm^{-3} sulfuric-acid stock solution.

NOTE 7—If the nominal concentrations of $c_1 = c_2 = 0.1$ mol dm⁻³, and $c_3 = 4$ mol dm⁻³ are assumed, then $V_1 = V_2 = 150$ mL for the high range and $V_1 = V_2 = 30$ mL for the low range; $V_3 = 85$ mL for the high range and $V_3 = 97$ mL for the low range. If the concentrations of the various stock solutions are significantly different from the nominal values, then use Eq 4-6 to determine the exact volumes. To prepare a volume of the dosimetric solution other than 1000 mL, the result of these equations should be multiplied by the ratio of the desired volume in millilitres to 1000 mL.

9.4 Determine all of the volumes given in 8.39.3 using a calibrated graduated cylinder volumetric flask that can be read to within ±0.5 mL.

9.5 Transfer the volume of each component of the dosimetric solution into a 1-L or larger glass storage container. Rinse the graduated cylindervolumetric flask used for measuring V_1 , V_2 , and V_3 by using some portion of the distilled purified water of V_4 . Stopper the container and shake well. Before use, allow the dosimetric solution to stand for at least five days in the dark.dark (ICRU 10b).

9.6 Quality control testing of the dosimetric solution prior to ampouling is performed by comparing the measurement of dosimetric solution parameters, such as ceric-ion concentration, cerous-ion concentration, ceric-ion molar linear absorption coefficient, radiation chemical yield for the cerous ion, and density with acceptable values. Procedures for performing these measurements are given in Annex A3. Quality control testing following ampouling is performed by comparing calibration data for the new dosimeter batch with data obtained from previous batches (see $\frac{10.5.310.6.4}{10.5.310.6.4}$).

9.7 Prepare dosimeters by filling ampoules with approximately 2 mL of dosimetric solution. Take care not to contaminate the dosimetric solution with impurities. Exercise care in filling ampoules to avoid depositing solution in the ampoule neck. Subsequent heating during sealing may cause an undesirable chemical change in the dosimetric solution remaining inside the ampoule neck. Flame seal the ampoules, exercising care to avoid heating the body of the ampoule during sealing.

9.8 Store dosimeters in a dark place at room temperature $(23 \pm 5^{\circ}C)$. $(23 \pm 5^{\circ}C)$.

9. Analytical instrument performance

9.1 Spectrophotometer Performance:

9.1.1 Check the wavelength scale of the spectrophotometer. Appropriate wavelength standards are holmium-oxide filters and solutions. For more details see ASTM Practices E275, E925, and E958.

Note 9-For example, holmium-oxide solutions in sealed euvettes are available as certified wavelength standards (SRM 2034) for use in the wavelength region from 240 to 650 nm (10).

9.1.2 Check the accuracy of the photometric (absorbance) scale of the spectrophotometer. Certified absorbance standard filters or solutions are available for this purpose.

Note 10-Examples of absorbance standards are solutions of various concentrations, such as SRM 931d (11) and SRM 935 (12), and metal-on-quartz filters, such as SRM 2031 (13, 14).