



Designation: **ISO/ASTM 51401–2013(E) 51401 – 21**

Standard Practice for Use of a Dichromate Dosimetry System¹

This standard is issued under the fixed designation ISO/ASTM 51401; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice covers the preparation, testing, and procedure for using the acidic aqueous silver dichromate dosimetry system to measure absorbed dose 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 dichromate system. The dichromate dosimeter is classified as a type I dosimeter on the basis of the effect of influence quantities. The dichromate system may be used as either a reference standard dosimetry system or a routine dosimetry system.

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 dichromate dosimetry system. It is intended to be read in conjunction with ISO/ASTM Practice 52628.

1.3 This practice describes the spectrophotometric analysis procedures for the dichromate 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 from 2×10^3 to 5×10^4 Gy.

1.5.2 The absorbed dose rate does not exceed 600 Gy/pulse (12.5 pulses per second), or does not exceed an equivalent dose rate of 7.5 kGy/s from continuous sources (1).²

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

NOTE 1—The lower energy limits given are appropriate for a cylindrical dosimeter ampoule of 12 mm diameter. Corrections for displacement effects and dose gradient across the ampoule may be required for electron beams (2). The dichromate system may be used at lower energies by employing thinner (in the beam direction) dosimeter containers (see ICRU Report 35).

1.5.4 The irradiation temperature of the dosimeter shall be above $\theta^\circ\text{C}$ – 0°C and should be below 80°C – 80°C .

¹ This practice 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 . Originally developed as a joint ASTM/ISO standard in conjunction with ISO/TC 85/WG 3.

Current edition approved Sept. 14, 2013Oct. 1, 2021. Published November 2013May 2024. Originally published as ASTM E 1401 – 91. ASTM E 1401 – 96 approved ^{E1} was adopted by ISO in 1998 with the intermediate designation ISO 15561:1998(E). The present International Standard ISO/ASTM 51401:2013(E) replaces ISO 15561 and is a major revision of the last previous edition ISO/ASTM 51401:2003(E) in 1991. Last previous edition approved in 2013 as ISO/ASTM 51401:2013(E). DOI: 10.1520/51401-21.

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

NOTE 2—The temperature coefficient of dosimeter response is known only in the range of 5 to 50°C–50 °C (see 5.2). Use outside this range requires determination of the temperature coefficient.

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, safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 9.3.

1.7 *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

~~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

~~E3083 Terminology Relating to Radiation Processing: Dosimetry and Applications~~

2.2 ISO/ASTM Standards:³

51261 Practice for Calibration of Routine Dosimetry Systems for Radiation Processing

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

52628 Practice for Dosimetry in Radiation Processing

52701 Guide for Performance Characterization of Dosimeters and Dosimetry Systems for Use in Radiation Processing

2.3 ISO/IEC Standards:⁴

17025 General Requirements for the Competence of Testing and Calibration Laboratories

~~12749-4 Nuclear energy — Vocabulary — Part 4: Dosimetry for radiation processing~~

2.4 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, VIM International Vocabulary of Metrology - Basic and General Concepts and Associated Terms⁶~~

2.5 International Commission on Radiation Units and Measurements (ICRU) Reports:⁷

ICRU Report 35 Radiation Dosimetry: Electrons With Initial Energies Between 1 and 50 MeV

ICRU Report 80 Dosimetry Systems for Use in Radiation Processing

ICRU Report 85a Fundamental Quantities and Units for Ionizing Radiation

3. Terminology

3.1 Definitions:

~~3.1.1 approved laboratory—laboratory 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.~~

~~3.1.1.1 Discussion—~~

~~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 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 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), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, <http://www.iso.org>.

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

⁶ Document produced by Working Group 2 of the Joint Committee for Guides in Metrology (JCGM/WG 1). Available free of charge at the BIPM website (<http://www.bipm.org>).

⁷ Available from the International Commission on Radiation Units and Measurements (ICRU), 7910 Woodmont Ave., Bethesda, MD 20814, U.S.A.

3.1.1 *reference standard dosimetry system*—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.

3.1.2 *type I dosimeter*—dosimeter of high metrological quality, the response of which is affected by individual influence quantities in a well-defined way that can be expressed way that is well-defined and capable of expression in terms of independent correction factors.

3.2 Definitions of other terms used in this practice standard that pertain to radiation measurement and dosimetry may be found in ISO/ASTM Practice 52628. Other terms that pertain to radiation measurement and dosimetry may be found in ASTM Terminology E170E3083, Definitions and ISO Terminology 12749-4. Where appropriate, definitions used in E170 are compatible with ICRU Report 85a; that document, therefore, may be used as an alternative reference; these standards have been derived from, and are consistent with definitions in ICRU Report 85a, and general metrological definitions given in the VIM.

4. Significance and use

4.1 The dichromate system provides a reliable means for measuring absorbed dose to water. It is based on a process of reduction of dichromate ions to chromic ions in acidic aqueous solution by ionizing radiation.

4.2 The dosimeter is a solution containing silver and dichromate ions in perchloric acid in an appropriate container such as a sealed glass ampoule. The solution indicates absorbed dose by a change (decrease) in optical absorbance at a specified wavelength(s) ((3), ICRU Report 80). A calibrated spectrophotometer is used to measure the absorbance.

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 influence quantities that need to be considered when using the dichromate dosimetry system are given below.

5.2 The dosimeter response has a temperature dependence during irradiation that is approximately equal to -0.2 % per degree Celsius between 25 and 50°C. At temperatures below 25°C, the dependence is smaller. The dosimeter response between 5 and 50°C is shown in Table 1, where the response at a given temperature is tabulated relative to the response at 25°C (4, 5).

5.2.1 The data in Table 1 may be fitted with an appropriate formula for convenience of interpolation as follows:

$$R_t = b_0 + b_1 t^{b_2} \tag{1}$$

where:

R_t = dosimeter response at temperature t relative to that at 25°C.

R_t ≡ dosimeter response at temperature t relative to that at 25 °C.

The curve generated from the fitted data is shown in Fig. 1.

5.3 No effect of ambient light (even direct sunlight) has been observed on dichromate solutions in glass ampoules (6).

5.4 The dosimeter response is dependent on the type and energy of the radiation employed. For example, the response in high energy (10 MeV) electron beams is reported to be approximately 3 % lower than the response in cobalt-60 radiation (2).

TABLE 1 Effect of irradiation temperature on dosimeter response

Temperature, °C	Relative Response	Temperature, °C	Relative Response
5	1.020	30	0.992
10	1.017	35	0.983
15	1.013	40	0.972
20	1.007	45	0.960
25	1.000	50	0.948

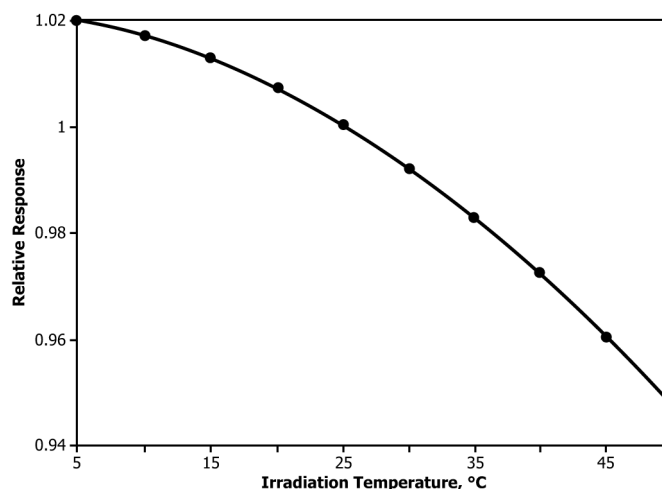


FIG. 1 Relative response of dichromate dosimeter as a function of irradiation temperature. A fit of the data using Eq 1 yields fit parameters as follows: $b_0 = 1.021$; $b_1 = -6.259 \times 10^{-5}$; $b_2 = 1.806$.

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

6. Interferences

6.1 The dichromate dosimetric solution response is sensitive to impurities, particularly organic impurities. Even in trace quantities, impurities can cause a detectable change in the observed response (6). For high accuracy results, organic materials shall not be used for any component in contact with the solution, unless it has been demonstrated that the materials do not affect dosimeter response. The effect of trace impurities may be minimized by pre-irradiation of the bulk dichromate solution (see Ref (6) and 9.4).

6.2 Undesirable chemical changes in the dosimetric solution can occur if care is not taken during sealing of ampoules (see 9.6).

7. Apparatus

7.1 *High-Precision Spectrophotometer*—For the analysis of the dosimetric solution, use a high-precision spectrophotometer capable of measuring absorbance values up to 2 with an uncertainty of no more than $\pm 1\%$ in the region of 350 to 440 nm. Use a quartz cuvette with 5 or 10 mm path length for spectrophotometric measurements of the solution. The cuvette capacity must be small enough to allow it to be thoroughly rinsed by the dosimeter solution and still leave an adequate amount of that solution to fill the cuvette to the appropriate level for the absorbance measurement. For dosimeter ampoules of less than 2 mL, this may require the use of micro-capacity cuvettes. Other solution handling techniques, such as the use of micro-capacity flow cells, may be employed provided precautions are taken to avoid cross-contamination. Either control the temperature of the dosimetric solution during measurement at $25 \pm 1^\circ\text{C}$, or determine the solution temperature during the spectrophotometric analysis and correct the measured absorbance to 25°C . The temperature coefficient during measurement is -0.1% per degree Celsius within the range of 20 to 30°C (6).

NOTE 3—The dosimetric ampoule commonly used has a capacity of about 2 mL.

7.2 *Glassware*—Use borosilicate glass or equivalent chemically resistant glass to store the reagents and the prepared dosimetric solution. Clean all apparatus used in the preparation of the solution, as well as the glass ampoules or other irradiation containers using chromic acid solution or an equivalent cleaning agent. Rinse at least three times with double-distilled water. Dry thoroughly and store in a dust-free environment.

NOTE 4—A validated process involving neutral liquid laboratory detergent in an ultrasonic bath, might be an alternative to chromic acid for cleaning glassware.

8. Reagents

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

8.2 Use of double-distilled water from coupled all-glass and silica stills is recommended. Alternatively, water from a high quality commercial purification unit capable of achieving Total Oxidisable Carbon (T.O.C.) content below 5 ppb may be used. Water purity is very important since it is the major constituent of the dosimetric solutions, and therefore may be the prime source of contamination. Use of deionized water is not recommended.

NOTE 5—Double-distilled water distilled from an alkaline permanganate (KMnO_4) solution (2 g KMnO_4 plus 5 g sodium hydroxide (NaOH) pellets in 2 dm^3 of distilled water) has been found to be adequate for preparation of the dichromate dosimetric solution. High purity water is commercially available from some suppliers. Such water labelled HPLC (high pressure liquid chromatography) grade is usually sufficiently free of organic impurities to be used in this practice.

9. Preparation of dosimeters

9.1 The recommended concentrations for the dichromate dosimeter to measure absorbed doses from about 2 to 10 kGy (hereafter called the low-range dosimeter) are $0.5 \times 10^{-3} \text{ mol dm}^{-3}$ silver dichromate ($\text{Ag}_2\text{Cr}_2\text{O}_7$) in 0.1 mol dm^{-3} aqueous perchloric acid (7). For measurement of absorbed doses from about 5 to 50 kGy (hereafter called the high-range dosimeter), the recommended concentrations are $0.5 \times 10^{-3} \text{ mol dm}^{-3}$ silver dichromate and $2.0 \times 10^{-3} \text{ mol dm}^{-3}$ potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in 0.1 mol dm^{-3} aqueous perchloric acid (6).

9.2 Air saturate both solutions before use. Shaking of the solution is normally sufficient to achieve this.

9.3 Silver dichromate dissolves slowly and normally requires at least 18 h to dissolve completely. For the high-range dosimeter, it is preferable to dissolve the silver dichromate before adding the potassium dichromate. (**Warning**—Concentrated perchloric acid is a strong oxidizer and dichromate salts are skin irritants. Appropriate precautions should be exercised in handling these materials.)

NOTE 6—Dichromate dosimeters of other formulations have been described (8, 9).

9.4 If appropriate, irradiate the bulk solution to minimize the effects of impurities.

9.4.1 The exact dose is not critical, but a dose of approximately 1.0 kGy is recommended (6). The size of the container for this bulk solution irradiation should be such that the dose variation to the solution is less than $\pm 10 \%$. Mix the solution thoroughly after irradiation.

9.5 Rinse the dosimeter ampoules or other containers as prepared in 7.2 at least once with the dosimeter solution before filling them for irradiation.

9.6 Exercise care in filling ampoules to avoid depositing solution in the ampoule neck, or remove traces of liquid by centrifuging the filled ampoules. Subsequent heating during sealing may cause an undesirable chemical change in the dosimetric solution remaining inside the ampoule neck. For the same reason, exercise care to avoid heating the body of the ampoule during sealing.

10. Calibration of the dosimetry system

10.1 Prior to use, the dosimetry system (consisting of a specific batch of dosimeters and specific measurement instruments) shall be calibrated in accordance with the user's documented procedure that specifies details of the calibration and quality assurance requirements. This calibration shall be repeated at regular intervals to ensure that the accuracy of the absorbed dose measurement is maintained within required limits. Calibration methods are described in ISO/ASTM Practice 51261.

10.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system.

10.2.1 When the dichromate dosimeter is used in a reference standard dosimetry system, calibration irradiations shall be performed at an approved laboratory, as defined in a laboratory having accreditation to ISO/IEC 3:1-1:17025.

10.2.2 When the dichromate dosimeter is used in a routine dosimetry system, the calibration irradiation may be performed in accordance with 10.2.1, or at a production or research irradiation facility together with reference- or transfer-standard dosimeters from a system that has measurement traceability to nationally or internationally recognized standards.

10.2.3 Specify the calibration dose in terms of absorbed dose to water.

10.2.4 For calibration with photons, the dichromate dosimeter shall be irradiated under conditions that approximate electron equilibrium.

10.2.5 The dosimeter shall be calibrated in a radiation field of the same type and energy as that in which it is to be used, unless evidence is available to demonstrate equivalence of response.

10.2.6 Calibrate each batch of dosimeters prior to use.

10.2.7 Separate five dosimeters from the remainder of the batch and do not irradiate them. Use them in determining A_0 , the absorbance of unirradiated dosimeters (see 10.5.1).

10.2.8 Control (or monitor) the temperature of the dosimeters during irradiation. Calculate or measure the mean irradiation temperature of each dosimeter to an accuracy of $\pm 2^\circ\text{C}$, $\pm 2^\circ\text{C}$, or better.

10.2.9 Use a set of at least three dosimeters for each absorbed dose value.

10.2.10 Irradiate these sets of dosimeters to at least five known dose values covering the range of utilization in order to determine the calibration curve for the dosimetry system.

10.3 *Measurement Instrument Calibration and Performance Verification*—For the calibration of the instruments, and for the verification of instrument performance between calibrations, see ISO/ASTM Practice 51261 or instrument-specific operating manuals, or both.

10.3.1 Check the wavelength scale of the spectrophotometer and establish its accuracy. The emission spectrum from a low-pressure mercury arc lamp can be used for this purpose. Such a lamp may be obtained from the spectrophotometer manufacturer or other scientific laboratory instrument suppliers. Other appropriate wavelength standards are holmium-oxide filters or solutions. For more details, see ASTM Practices E275, E925, and E958.

NOTE 7—For example, holmium-oxide solutions in sealed cuvettes are available as certified wavelength standards (SRM 2034) for use in the wavelength region of 240 to 650 nm.⁸

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

NOTE 8—Examples of absorbance standards are solutions of various concentrations such as SRM 931f and SRM 935 (10) and metal-on-quartz filters such as SRM 2031.⁸

10.4 *Measurement:*

10.4.1 For the low-range dosimeter, set the wavelength of the spectrophotometer at 350 nm, and use a spectral bandwidth of no more than 1 nm. For the high-range dosimeter, set the wavelength at 440 nm, and use a spectral bandwidth of no more than 1 nm.

10.4.2 Set the balance of the spectrophotometer to zero with air only (no cuvette) in the light path(s).

10.4.3 Fill a clean cuvette (or flow cell) of 5 or 10 mm pathlength with double-distilled water and measure the absorbance. Record this value.

NOTE 9—Choice of pathlength depends on the maximum absorbance that can be accurately measured by the spectrophotometer. For example, a pathlength of 10 mm will result in an absorbance of about 1.3 (or 0.65 for a pathlength of 5 mm) for the unirradiated dosimetric solution. The absorbance of irradiated solutions will be less than 1.3, that is, the absorbance decreases with increasing dose.

10.4.4 Empty the water from the cuvette (or flow cell) and rinse it at least once with the dosimetric solution from an ampoule.

⁸ Available from National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899, U.S.A.