
Practice for use of a dichromate dosimetry system

Utilisation d'un système de mesure dosimétrique au dichromate



Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 15561 was prepared by the American Society for Testing and Materials (ASTM) Subcommittee E10.01 (as E 1401-96) and was adopted, under a special "fast-track procedure", by Technical Committee ISO/TC 85, *Nuclear energy*, in parallel with its approval by the ISO member bodies.

A new ISO/TC 85 Working Group WG 3, *High-level dosimetry for radiation processing*, was formed to review the voting comments from the ISO "Fast-track procedure" and to maintain these standards. The USA holds the convenership of this working group.

International Standard ISO 15561 is one of 20 standards developed and published by ASTM. The 20 fast-tracked standards and their associated ASTM designations are listed below:

ISO Designation	ASTM Designation	Title
15554	E 1204-93	<i>Practice for dosimetry in gamma irradiation facilities for food processing</i>
15555	E 1205-93	<i>Practice for use of a ceric-cerous sulfate dosimetry system</i>
15556	E 1261-94	<i>Guide for selection and calibration of dosimetry systems for radiation processing</i>
15557	E 1275-93	<i>Practice for use of a radiochromic film dosimetry system</i>
15558	E 1276-96	<i>Practice for use of a polymethylmethacrylate dosimetry system</i>
15559	E 1310-94	<i>Practice for use of a radiochromic optical waveguide dosimetry system</i>
15560	E 1400-95a	<i>Practice for characterization and performance of a high-dose radiation dosimetry calibration laboratory</i>
15561	E 1401-96	<i>Practice for use of a dichromate dosimetry system</i>

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Printed in Switzerland

15562	E 1431-91	<i>Practice for dosimetry in electron and bremsstrahlung irradiation facilities for food processing</i>
15563	E 1538-93	<i>Practice for use of the ethanol-chlorobenzene dosimetry system</i>
15564	E 1539-93	<i>Guide for use of radiation-sensitive indicators</i>
15565	E 1540-93	<i>Practice for use of a radiochromic liquid dosimetry system</i>
15566	E 1607-94	<i>Practice for use of the alanine-EPR dosimetry system</i>
15567	E 1608-94	<i>Practice for dosimetry in an X-ray (bremsstrahlung) facility for radiation processing</i>
15568	E 1631-96	<i>Practice for use of calorimetric dosimetry systems for electron beam dose measurements and dosimeter calibrations</i>
15569	E 1649-94	<i>Practice for dosimetry in an electron-beam facility for radiation processing at energies between 300 keV and 25 MeV</i>
15570	E 1650-94	<i>Practice for use of cellulose acetate dosimetry system</i>
15571	E 1702-95	<i>Practice for dosimetry in a gamma irradiation facility for radiation processing</i>
15572	E 1707-95	<i>Guide for estimating uncertainties in dosimetry for radiation processing</i>
15573	E 1818-96	<i>Practice for dosimetry in an electron-beam facility for radiation processing at energies between 80 keV and 300 keV</i>

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E 1401

ICRU Report 37 Stopping Powers for Electrons and Positrons

3. Terminology

3.1 Definitions:

3.1.1 *absorbed dose (D)*, *n*—quantity of ionizing radiation energy imparted per unit mass of a specified material. The SI unit of absorbed dose is the gray (Gy), where 1 gray is equivalent to the absorption of 1 joule per kilogram of the specified material (1 Gy = 1 J/kg). The mathematical relationship is the quotient of $d\bar{\epsilon}$ by dm , where $d\bar{\epsilon}$ is the mean incremental energy imparted by ionizing radiation to matter of incremental mass dm (see ICRU Report 33).

$$D = \frac{d\bar{\epsilon}}{dm} \quad (1)$$

3.1.1.1 *Discussion*—The discontinued unit for absorbed dose is the rad (1 rad = 100 erg/g = 0.01 Gy). Absorbed dose is sometimes referred to simply as dose.

3.1.2 *calibration facility*, *n*—combination of an ionizing radiation source and its associated instrumentation that provides a uniform and reproducible absorbed dose, or absorbed-dose rate traceable to national or international standards at a specified location and within a specific material, and that may be used to derive the dosimetry system's response function or calibration curve.

3.1.3 *dosimetry system*, *n*—a system used for determining absorbed dose, consisting of dosimeters, measurement instruments, and their associated reference standards, and procedures for the system's use.

3.1.4 *measurement quality assurance plan*, *n*—a documented program for the measurement process that ensures on a continuing basis that the overall uncertainty meets the requirements of the specific application. This plan requires traceability to, and consistency with, nationally or internationally recognized standards.

3.1.5 *net absorbance*, (ΔA), *n*—change in measured optical absorbance at a selected wavelength determined as the absolute difference between the pre-irradiation absorbance, A_0 , and the post-irradiation absorbance, A , as follows:

$$\Delta A = |A - A_0| \quad (2)$$

3.1.6 *reference standard dosimeter*, *n*—a dosimeter of high metrological quality, used as a standard to provide measurements traceable to and consistent with measurements made using primary standard dosimeters.

NOTE 3—For other terms, see Terminology E 170.

4. Significance and Use

4.1 The dichromate system provides a reliable means for measuring absorbed dose in 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 flame-sealed glass ampoule. The solution indicates absorbed dose by a change (decrease) in optical absorbance at a specified wavelength(s) (3). A calibrated spectrophotometer is used to measure the absorbance.

4.3 Effect of Irradiation Temperature:

4.3.1 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).

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

$$R_T = b_0 + b_1 T^{b_2} \quad (3)$$

where:

R_T = dosimeter response at temperature T relative to that at 25°C.

The fitted data is shown in Fig. 1.

4.4 No effect of ambient light (even direct sunlight) has been observed on dichromate solutions in glass ampoules (5).

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

4.6 The absorbed dose in other materials irradiated under equivalent conditions may be calculated. Procedures for making such calculations are given in Practices E 666 and E 668 and Guide E 1261.

4.7 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). The dosimeter shall be calibrated in a radiation field of the same type and energy as that in which it is to be used.

5. Interferences

5.1 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 (5). For high accuracy results, organic materials shall not be used for any component in contact with the solution. The effect of trace impurities may be minimized by pre-irradiation of the bulk dichromate solution (see Ref (5) and 8.2).

5.2 Undesirable chemical changes in the dosimetric solution can occur if care is not taken during flame-sealing of the ampoules (see 8.4).

6. Apparatus

6.1 *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 ± 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

TABLE 1 Effect of Irradiation Temperature on Dosimeter Response

Temperature, °C	Response	Temperature, °C	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

E 1401

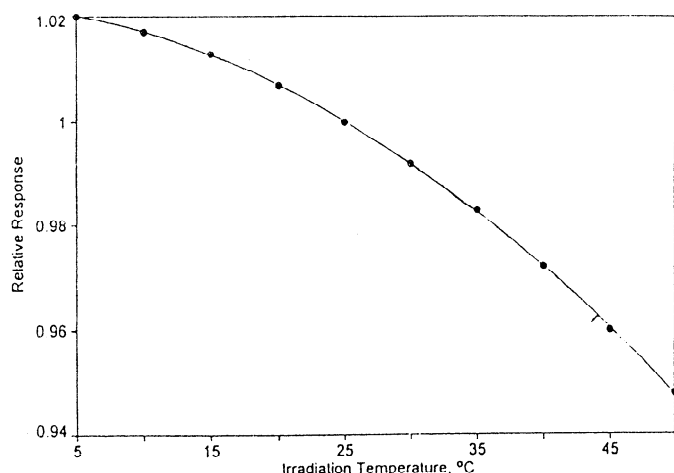


FIG. 1 Relative Response of Dichromate Dosimeter as a Function of Irradiation Temperature. A Fit of the Data Using Eq 3 Yields Fit Parameters as Follows: $b_0 = 1.021$; $b_1 = -6.259 \times 10^{-5}$; $b_2 = 1.806$.

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 2 mL or less, 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. Control the temperature of the dosimetric solution during measurement at $25 \pm 1^\circ\text{C}$. If this is not possible, 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 (5).

NOTE 4—The dosimetric ampoule commonly used has a capacity of about 2 mL.

6.2 *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 (see Practice C 912). Dry thoroughly and store in a dust-free environment (see Practice E 1026).

7. Reagents

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

7.2 Use of double-distilled water from coupled all-glass and silica stills is recommended. 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 dm³ of distilled water) has been found to be adequate for routine 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.

8. Preparation of Dosimeters

8.1 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 (6). For measurement of absorbed doses from about 5 to 50 kGy (using the dosimeter 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 (5). Air saturate both solutions before use. 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.

NOTE 6: **Precaution**—Concentrated perchloric acid is a strong oxidizer and dichromate salts are skin irritants. Appropriate precautions should be exercised in handling these materials.

NOTE 7—Dichromate dosimeters of other formulations have been described (7,8).

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

8.2.1 The exact dose is not critical, but a minimum dose of 1.0 kGy is recommended (5).

8.2.2 Choose the size of the container for this bulk solution so that the dose delivered to any part of the solution is $1.1 \pm 0.1 \text{ kGy}$.

8.2.3 Mix the solution thoroughly after irradiation.

8.3 Rinse the dosimeter ampoules or other containers as prepared in 6.2 at least once with the dosimeter solution before filling them for irradiation.

8.4 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. For the same reason, exercise care to avoid heating the body of the ampoule during sealing.

9. Spectrophotometer Calibration

9.1 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 Practices E 275, E 925, and E 958.

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

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

* Available from National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.

E 1401

NOTE 13—Computer software is available commercially for performing least-squares fits of data with polynomials or other analytical forms.

10.3.3 Graphs of the data in Table 2 are shown in Figs. 2 and 3. The curves should tend towards $\Delta A = 0$ at zero dose. An appreciable ΔA intercept value is indicative of contamination of the dosimetric solution with impurities.

10.3.4 Compare the net absorbances determined by a given calibration with the examples given in Table 2. For cobalt-60 radiation, agreement should be within $\pm 3\%$ if the dosimetric solutions were properly prepared and all associated analysis equipment was properly calibrated. Values for high energy electron beam irradiation should be approximately 3 % lower. Agreement of the dosimetric response values from batch to batch over the useful range of the system should be within $\pm 1\%$.

10.3.5 Estimate the precision (random uncertainty) of the individual dosimeter results either from the results of replicate measurements or from the statistics of the least-squares fit to the data. The precision provides a measure of acceptable performance of the dosimetry system. The precision, expressed as one standard deviation, should not exceed 0.002 absorbance units for the high-range dosimeter or 0.003 absorbance units for the low range dosimeter for an optical pathlength of 10 mm. Suspected data outliers should be tested using statistical procedures such as those found in Practice E 178.

11. Application of Dosimetry System

11.1 For most applications, use a minimum of three dosimeters for each dose measurement. The number of dosimeters required for the measurement of absorbed dose on or within a material is determined by the precision of the dosimetry system and the required precision associated with the application. Appendix X3 of Practice E 668 describes a statistical method for determining this number.

11.2 Use the irradiation and measurement procedures in accordance with 10.1.1, 10.1.3, 10.1.8, 10.2.1 through 10.2.5, and 10.3.1.

11.3 Determine the absorbed dose from the net absor-

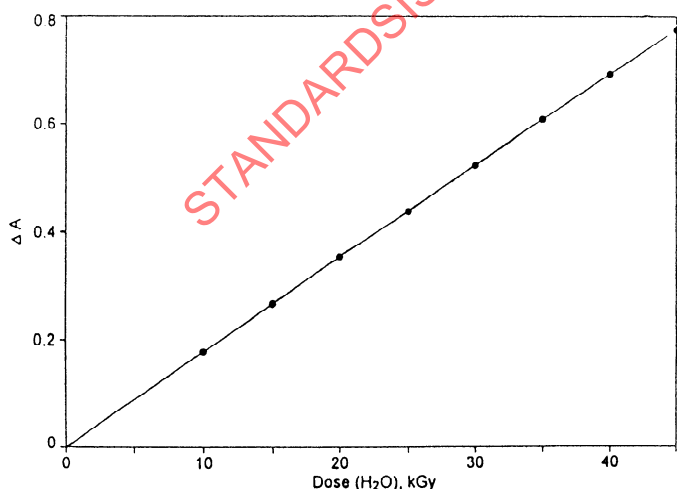


FIG. 2 Response of High-Range Dosimeter in Terms of ΔA as a Function of Absorbed Dose in Water. A Least-Squares Second Order Polynomial Fit (see Eq 6) of the Data Yields Fit Parameters as Follows: $b_0 = 1.338 \times 10^{-3}$; $b_1 = 1.799 \times 10^{-2}$; $b_2 = 1.648 \times 10^{-5}$.

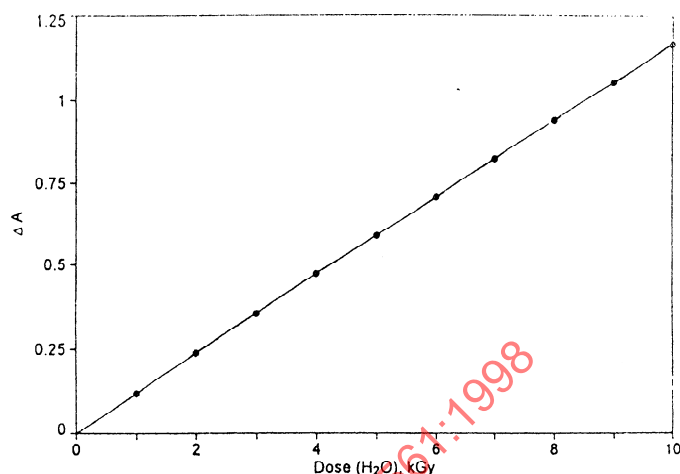


FIG. 3 Response of the Low-Range Dosimeter in Terms of ΔA as a Function of Absorbed Dose in Water. A Least-Squares Second Order Polynomial Fit (see Eq 6) of the Data Yields Fit Parameters as Follows: $b_0 = 1.162 \times 10^{-3}$; $b_1 = 1.200 \times 10^{-1}$; $b_2 = 3.398 \times 10^{-4}$.

bance values and the calibration curve.

11.4 Record the calculated absorbed dose values and all other relevant data as outlined in Section 12.

12. Minimum Documentation Requirements

12.1 Calibration:

12.1.1 Record the dosimeter type and batch number (code).

12.1.2 Record or reference the date, irradiation temperature, temperature variation (if any), dose range, radiation source, and associated instrumentation used to calibrate and analyze the dosimeters.

12.2 Application:

12.2.1 Record the date and temperature of irradiation, temperature variation (if any), and the date and temperature of absorbance measurement, for each dosimeter.

12.2.2 Record or reference the radiation source type and characteristics.

12.2.3 Record the absorbance, net absorbance value, temperature correction (if applicable), and resulting absorbed dose for each dosimeter. Reference the calibration curve used to obtain the absorbed dose values.

12.2.4 Record or reference the uncertainty in the value of the absorbed dose.

12.2.5 Record or reference the measurement quality assurance plan used for the dosimetry system application.

13. Precision and Bias

NOTE 14—Measurement uncertainty is a more accurate description of the contents of this section.

13.1 To be meaningful, a measurement shall be accompanied by an estimate of uncertainty. Components of uncertainty shall be identified as either Type A or Type B according to their method of evaluation. Type A evaluation of standard uncertainty is based on the statistical analysis of a series of observations, and Type B is based on all other methods of analysis. Additional information is given in Guide E 1707 and Refs (10) and (11).