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Practicefor use of a dichromate dosimetry system

Pratique de l'utilisation d'un système dosimétrique au dichromate

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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.

ASTM International is one of the world's largest voluntary standards development organizations with global participation from affected stakeholders. ASTM technical committees follow rigorous due process balloting procedures.

A project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this project, ASTM Subcommittee E10.01, Dosimetry for Radiation Processing, is responsible for the development and maintenance of these dosimetry standards with unrestricted participation and input from appropriate ISO member bodies.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. Neither ISO nor ASTM International shall be held responsible for identifying any or all such patent rights.

International Standard ISO/ASTM 51401 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear Energy.

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

ICRU Report 60 Fundamental Quantities and Units for IonizingRadiation

3. Terminology

3.1 *Definitions:*

3.1.1 *net absorbance, (ΔA)*—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:

$$\Delta A = |A - A_0| \quad (1)$$

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

3.2 Definitions of other terms used in this practice that pertain to radiation measurement and dosimetry may be found in ASTM Terminology E 170. Definitions in E 170 are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

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 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 **(4,5)**.

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

$$R_t = b_0 + b_1 t^{b_2} \quad (2)$$

where:

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.

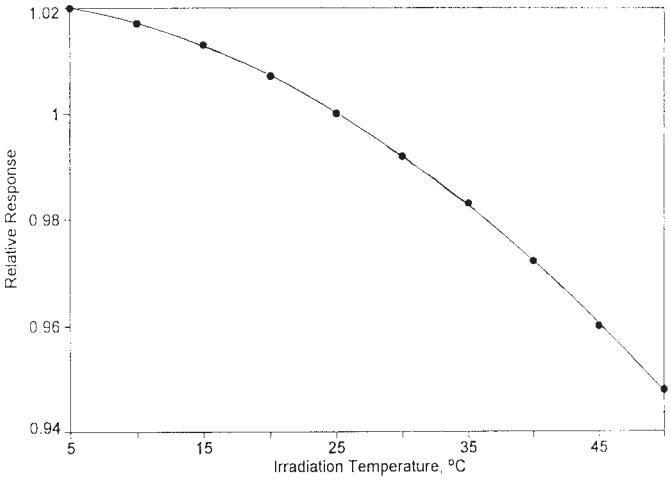

FIG. 1 Relative response of dichromate dosimeter as a function of irradiation temperature. A fitof the data using Eq 2 yields fit parameters as follows: b_0 $b_1 = -6.259 \times 10^{-5}$; $b_2 = 1.806$.

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

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

4.6 The absorbed dose in materials other than water irradiated under equivalent conditions may be calculated using the procedures given in ASTM Practices E 666, E 668 and ISO/ASTM Guide 51261.

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.

4.8 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 before and after irradiation.

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 **(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 8.4).

5.2 Undesirable chemical changes in the dosimetric solution can occur if care is not taken during sealing of ampoules (see 8.6).

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 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. Control the temperature of the dosimetric solution during measurement at 25 ± 1°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 **(6)**.

NOTE 3—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 ASTM Practice C 912). Dry thoroughly and store in a dust-free environment (see ASTM 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. 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 4—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 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 0.5×10^{-3} mol dm^{-3} silver dichromate ($Ag_2Cr_2O_7$) in 0.1 mol dm^{-3} aqueous perchloric acid **(7)**. For measurement of absorbed doses from about 5 to 50 kGy (using the dosimeter hereafter called the high-range dosimeter), the recommended concentrations are 0.5×10^{-3} mol dm^{-3} silver dichromate and 2.0×10^{-3} mol dm^{-3} potassium dichromate ($K_2Cr_2O_7$) in 0.1 mol dm^{-3} aqueous perchloric acid **(6)**.

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

8.3 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 5—Dichromate dosimeters of other formulations have been described **(8, 9)**.

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

8.4.1 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 ±10 %. Mix the solution thoroughly after irradiation.

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

8.6 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. Calibration of the dosimetry system

9.1 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 process and quality assurance requirements. This calibration process 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 Guide 51261.

9.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed at an accredited calibration laboratory, or at an in-house calibration facility meeting the requirements in ISO/ASTM Practice 51400, that provides an absorbed dose (or absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards.

9.2.1 When the dichromate dosimeter is used as a routine dosimeter, the calibration irradiation may be performed in accordance with 9.2, or at a production or research irradiation facility together with reference- or transfer-standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

9.2.2 Specify the calibration dose in terms of absorbed dose in water.

9.2.3 Calibrate each batch of dosimeters prior to use.

9.2.4 Separate five dosimeters from the remainder of the batch and do not irradiate them. Use them in determining A_0 (see 9.5.1).

9.2.5 Control (or monitor) the temperature of the dosimeters during irradiation. Calculate or measure the mean irradiation temperature of each dosimeter to an accuracy of ±2°C, or better.

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

9.2.7 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.

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

9.3.1 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 E 275, E 925, and E 958.

NOTE 6—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.[8]

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

NOTE 7—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]

9.4 *Measurement*:

9.4.1 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.

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

9.4.3 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 8—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.

9.4.4 Empty the water from the cuvette (or flow cell) and rinse it at least once with the solution from an ampoule. Discard the rinse solution and fill to the appropriate level with more solution from the same ampoule. Carefully wipe off any solution on the exterior surfaces of the cuvette and measure the absorbance. Repeat this procedure for all unirradiated and irradiated solutions.

NOTE 9—Inadequate rinsing of the cuvette (or flow cell) between dosimeter solutions can lead to errors due to solution carryover (cross-contamination). Techniques for minimizing this effect are discussed in Ref **(10)**.

9.4.5 Check the zero reading after each sample with air only in the light beam(s). Periodically during the measurement process, remeasure the absorbance of distilled water to detect any contamination of the cuvette (or flow cell) and take appropriate corrective actions to remove any contamination, if required.

9.5 *Analysis*:

9.5.1 Calculate the mean absorbance of the unirradiated dosimeters, A_0 (see 9.2.4). Calculate the net absorbance, ΔA, for each irradiated dosimeter by subtracting its absorbance, A_i, from A_0 as follows:

$$\Delta A = A_0 - A_i \tag{3}$$

9.5.2 Using the data in Table 1 and Eq 2, correct the measured net absorbance ΔA to the net absorbance expected for an irradiation temperature of 25°C using the formula:

$$\Delta A_{25} = \Delta A_t / R_t \tag{4}$$

9.5.3 Prepare a calibration curve by plotting the ΔA values versus absorbed dose, D. Fit the data by means of a least-squares method with an appropriate analytical form that provides a best fit to the data. The data for these dichromate dosimeters should fit a second (or third) order polynomial of the form:

$$\Delta A = b_0 + b_1 D + b_2 D^2 \, (+ \, b_3 D^3) \tag{5}$$

9.5.4 Examples of calibration test data of solutions known to produce good dosimetric results are given in Table 2.

NOTE 10—Computer software is available commercially for performing least-squares fits of data with polynomials or other analytical forms.

TABLE 2 Typical dichromate calibration data[A]

High-Range Dosimeter Approximate A_0 = 1.1		Low-Range Dosimeter Approximate A_0 = 1.3	
Dose, kGy	ΔA	Dose, kGy	ΔA
10.0	0.1752	1.0	0.1185
15.0	0.2625	2.0	0.2374
20.0	0.3490	3.0	0.3557
25.0	0.4348	4.0	0.4733
30.0	0.5198	5.0	0.5902
35.0	0.6038	6.0	0.7065
40.0	0.6866	7.0	0.8220
45.0	0.7679	8.0	0.9369
50.0	0.8475	9.0	1.0511
55.0	0.9249	10.0	1.1646

[A] The conditions during irradiation and measurement for these data were as follows:
Radiation type: ^{60}Co
Irradiation and measurement temperature: 25°C
Optical path length during analysis: 10 mm
Wavelength for analysis of high-range dosimeter: 440 nm
Wavelength for analysis of low-range dosimeter: 350 nm

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

Further information on mathematical methods for handling calibration datais given in ISO/ASTM Guide 51707.