

Calibrations of Amber Perspex-PMMA Dosimeter in the CDTN Gamma Irradiator Operational Conditions

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Abstract: This paper presents results of PMMA (polymethylmethacrylate) dosimeters calibrations that are used as routine dosimeters in CDTN (Nuclear Technology Development Center) Gamma Irradiation Laboratory. The study was carried out within the framework of the Facility Quality Assurance Program with the purpose of to determine the calibration curve and accuracy of the Harwell Amber Perspex 3042 W dosimeters. The irradiations of the dosimeters were made in operational conditions of a Gamma Irradiator at CDTN, an R & D (research and development) institute connected to the CNEN (National Nuclear Energy Commission) (Brazil). The values for estimate of the expanded uncertainties achieved are typical of a routine dosimetry system.

Key words: Accuracy, PMMA dosimeter, gamma processing, absorbed dose, irradiator.

1. Introduction

Any application that uses ionizing radiation, in order to achieve specific biological, chemical or physical effects is classified in a general way as an irradiation process. In such process, the material is intentionally irradiated, during a certain time interval to maintain, modify or improve its characteristics. In this time interval, the product absorbs only a fraction of the energy carried by the radiation that strikes it. This fraction, which is called radiation absorbed dose, depends on the mass of the product, its composition and the exposure time. The dose unit is the Gy (gray), which is defined as the absorption of 1 joule of energy per kg of product.

In order to determine the absorbed dose, irradiation facilities have used different measurement systems that characterize a dosimetry system. Dosimetry represents an important function in radiation processing, where large absorbed doses and dose rates from photon and electron sources have to be measred with reasonable accuracy.

Proven dosimetry systems are widely used to perform radiation measurements in development of processes, validation, qualification and new verification (quality control) of established processes and archival documentation of day-to-day and plant-to-plant processing uniformity. Proper calibration and traceability of routine dosimetry systems to standards are crucial to the success of many large-volume radiation processes.

It is essential that, users perform their own separate calibration for their own instrumentation and conditions of use [1].

This paper presents the calibrations results of Amber Perspex 3042 W PMMA (polymethylmethacrylate) dosimeters, produced by Harwell that are used as routine dosimeters in the CDTN (Nuclear Technology Development Center) Gamma Irradiation Facility [1]. The study was carried out within the framework of the Facility Quality Assurance Program with the purpose of to determine the calibration curve and uncertainties of the dosimeter in the facility operational conditions.

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2. Experiments

2.1 Dosimetry System

The following components of PMMA routine dosimetry systems are used in CDTN Gamma Irradiation Laboratory.

2.1.1 PMMA Dosimeter

The dosimeters studied are named Amber Perspex (4034, batch W), that cover the range which from 1 kGy to 30 kGy. They are presented in the form of small rectangular pieces (Fig. 1), optically transparent and individually sealed in aluminum sachets.

The pieces are kept in their protective cases during and after irradiation, only being open at the time of their reading. This procedure is justified by the need to preserve the radiosensitive material to dirt, abrasion and environmental conditions (light and humidity) factors that may influence the dosimeter quality. When exposed to radiation, they exhibit a change in its specific absorbance, which is characterized as a function of absorbed dose. This absorbance change is measured by UV-Vis (ultraviolet-visible) spectrophotometry.

2.1.2 Spectrophotometer

For the measurement of the dosimeters optical absorbance, the spectrophotometer shown in Fig. 2 was used. It was manufactured by Micronal and its model is B572 [2]. Its main characteristics are shown in Table 1.



Fig. 1 Harwell Amber Perspex 4034.

2.1.3 Thicknessgauge

For the dosimeters thickness measurements, a calibrated electronic outside micrometer shown in Fig. 3 was used. It was manufactured by digimess instruments, has a measurement range of 0-25 mm and accuracy of ± 0.002 mm [3].

2.2 The Irradiation Facility

The CDTN irradiator is a GB-127 (gamma beam-127) type, manufactured by Nordion [4]. The GB-127 is a dry storage gamma panoramic irradiator that uses Nordion's F-127 shipping container for source storage and operation. Products to be irradiated are placed inside the biological shield, either on turntables or directly on the floor. The source is raised by a



Fig. 2 Spectrophotometer UV-Vis.



Fig. 3 Electronic outside micrometer.

 Table 1
 Spectrophotometer characteristics.

Wavelength range	340 nm to 1,000 nm
Bandwidth	10 nm
Wavelength accuracy	±2 nm
Absorbance band	-0.300 to 3.000
Absorbance accuracy*	±0.012

*Verified through a set of standards filter.

Maximum source activity	60 kCi (2.220 TBq)
Dose rate at 1 m	600 Gy/h
Product configurations	12 turntables (50 cm diameter, 2 rpm)
Maximum product weight (on turntables)	700 lbs (320 kg)
Source to product distance	Variable up to 300 cm
Floor area	33 ft × 39 ft (10.1 m × 11.9 m)
Operation mode	Manual batch

Table 2Irradiator specifications.

Fig. 4 Irradiator cut away view.

pneumatic hoist. Table 2 shows the irradiator specifications and Fig. 4 presents a cut away view of the irradiation facility.

2.3 Dosimeters Positioning

The experimental setup for calibration consisted of a PVC (polyvinylchloride) pipe divided into 11 irradiation points with three dosimeters per point. The wood holder shown in Fig. 5 was fabricated for the dosimeters positioning.

The assembly was placed in the irradiation room, with the horizontal dosimeters midline coinciding with the source center plane, as shown in Fig. 6.

3. Methodology

3.1 Absorbed Doses

The dosimeters were calibrated according to ISO (International Organization for Standardization)/ASTM (American Society for Testing and Materials) standard [5]. Establishing the calibration curve was used as reference Fricke dosimeter, which is the secondary

Fig. 5 Support with three dosimeters.

Fig. 6 Dosimeters positioning.

standard used in the CDTN Gamma Irradiation Laboratory. This dosimeter was calibrated against a primary standard [6].

The dosimeters irradiations were carried out under conditions similar to those in routine use, according manufacture's recommendations [1].

All influence quantities (e.g., shelf-life, temperature, relative humidity, among others) associated with pre-irradiation, irradiation and post-irradiation conditions were taken into account [5].

The dosimeters optical absorbance was measured with the spectrophotometer at wavelength $\lambda = 603$ nm (1-10 kGy) and $\lambda = 651$ nm (10-30 kGy).

For PMMA dosimeter, the specific absorbance A_{sp} is the dosimeter response [5]:

$$A_{sp} = \binom{A_{\lambda}}{d} \tag{1}$$

where, A_{λ} = optical absorbance at a selected wavelength (λ); d = optical path length.

3.2 Uncertainties

The result of a measurement is only an estimate of the measurement value. Thus, this result is only complete when accompanied by an uncertainty indication of the estimate.

Uncertainty is defined as the associated measure value with that characterizes the distribution of values that could reasonably be attributed to the measuring parameter. On the other hand, the error is defined as the difference between a measurement result and the actual quantity value.

In order to obtain a significant value of the measurement of the absorbed dose, the uncertainties associated with its measurement must be estimated and their magnitudes quantified.

Uncertainties can be classified according to the type of effect that causes (random or not random) or based on their evaluations method (Type A or Type B). The random type comes from unpredictable variations (stochastic) parameters of influence. It can be reduced by increasing the number of observations but not eliminated [7]. Furthermore, the systematic error arises from the known influence parameters effects. It will be reduced if the effect can be quantified.

The purpose of classification in Types A and B indicates the two different ways of evaluating uncertainty components.

In Type A, the evaluation method is based on statistical analysis of a series of observations, thus, the uncertainty is obtained from a probability density function derived from a frequency distribution observed. In the Type B, the evaluation method is based on measuring instruments information, calibration, certificates, chemical purity, etc. The uncertainty in this case is obtained from a density of pre-defined probability.

The uncertainties sources in dosimetry usually reside in factors such as the dose absorbed by the dosimeter during calibration of the system, the analysis of the response of the dosimeter, the adjustment of calibration curves for the dosimetric data, among others [8].

Each uncertainty source usually consists of several components of both Types A and B.

For sources of independent (uncorrelated) uncertainty, the combined uncertainty is obtained by combining all of the standard uncertainties—Types A and B. This combined standard uncertainty is defined by:

$$U_{C} = \sqrt{U_{A}^{2} + U_{B}^{2}}$$
(2)

For related uncertainty sources, the effects of these correlations should be taken into account in determining the combined standard uncertainty.

The expanded uncertainty is obtained by multiplying the combined standard uncertainty by a coverage factor k:

$$U = k U_C \tag{3}$$

For normal probability distributions if $U_A \le U_B$, then k = 1 corresponds to a 66% confidence level, k = 2 corresponds to 95% and k = 3corresponds to 99%.

The uncertainty for n points is obtained by calculating the expanded variance V:

$$V = (1/n) \sum_{i=1}^{n} (U_{c}^{2})_{i}$$
(4)

And the global combined standard uncertainty U_{GC} is obtained by:

$$U_{GC} = \sqrt{V} \tag{5}$$

4. Results and Discussion

4.1 Dosimeters Irradiations Results

Table 3 shows the results of dosimeters irradiations which were performed on December 19, 2013. The changes in absorbance of the dosimeters were determined at a wavelength of $\lambda = 603$ nm.

The means specific absorbance $\overline{(A_{sp})}$ considers the measurement of the three dosimeters positioned at each point.

Table 4 shows the results of the dosimeters irradiation whose absorbance was determined at a wavelength $\lambda = 651$ nm.

Point	Distance	$\overline{A_{sp}}$	Dose
	(m)	(mm ⁻¹)	(kGy)
1	0.1	0.475	16.70
2	0.15	0.351	11.55
3	0.20	0.263	8.35
4	0.25	0.190	6.30
5	0.30	0.161	4.92
6	0.35	0.130	3.95
7	0.40	0.106	3.24
8	0.45	0.087	2.70
9	0.50	0.072	2.29
10	0.60	0.053	1.70
11	0.80	0.030	1.04

Table 3 Results at $\lambda = 603$ nm.

Table 4 Results at $\lambda = 651$ nm.

Point	Distance (m)	$\overline{A_{sp}}$ (mm ⁻¹)	Dose (kGy)
1	0.1	0.013	1.04
2	0.15	0.025	1.70
3	0.20	0.035	2.29
4	0.25	0.042	2.70
5	0.30	0.052	3.24
6	0.35	0.063	3.95
7	0.40	0.079	4.92
8	0.45	0.130	8.35
9	0.50	0.172	11.55
10	0.60	0.230	16.70
11	0.80	0.343	29.66

The mean specific absorbance $\overline{(A_{sp})}$ considers, also the measurement of the three dosimeters positioned at each point.

4.2 Uncertainties Evaluation

The uncertainties sources identified in measurements were the dose determination (Fricke reference dosimeter), the source decay, the dosimeter thickness (optical path length) measuring, the spectrophotometer accuracy and the dosimeter repeatability.

The dose determination uncertainty found was $\pm 2.48\%$. The source decay uncertainty has a value of $\pm 0.01\%$ while the dosimeter thicknesses are ± 0.002 mm and ± 0.012 for spectrophotometer reading.

The dosimeter repeatability is found by calculating the average standard deviation between absorbance measurements at each point. Table 5 shows, as an example, the calculations for the uncertainty in the absorbed dose at point #1.

The same calculation was performed for all calibration points, in order to obtain the expanded variance, whose value is:

$$V = {\binom{1}{9}} \sum_{i=1}^{11} (U_C^2)_i$$
 (6)

Thus, the overall combined standard uncertainty is obtained by:

$$U_{GC} = \sqrt{V} \tag{7}$$

With this methodology, the values 3.34% (range 0-17 kGy) and 3.46% (range 0-30 kGy) for the overall combined standard uncertainties were found.

4.3 Calibration Curves

The relation between the dosimeter response and the absorbed dose is represented by the fourth degree adjusted polynomial, in the range of 0-17 kGy.

For this range, the calibration curve is:

 $A_{sp} = a + bD + cD^2 + dD^3 + eD^4$ (8) where, A_{sp} (mm⁻¹) = specific absorbance; D (kGy)= absorbed dose; a = -8.57724E-03; d =7.010120E-06; b = 3.71476E-02; e = -2.49855E-08; c = -6.00360E-04.

The Fig. 7 shows the calibration curve. Considering the curve fit factor, which is 0.93%, and the overall combined standard uncertainty of 3.34%, is obtained a value of 3.47% for the combined standard uncertainty, when using Eq. (8) for specific absorbance determinations.

Specific absorbance determinations with Eq. (8) are affected with an expanded uncertainty of $\pm 6.94\%$ at the 95% confidence level.

4.3.2 Range 0 kGy to 30 kGy

For this range, the calibration curve is:

 $A_{sp} = a + bD + cD^2 + dD^3 + eD^4$ (9) where, A_{sp} (mm⁻¹) = specific absorbance; *D* (kGy) = dose rate; a = -5.89497E-03; d = 2.38222E-07; b = 1.87171E-02; e = 6.23733E-08; c = -2.96574E-04.

Calibration			Uncertainty		
Dose (kGy) Dosimeter	Desimentes	$A_{sp} (\mathrm{mm}^{-1})$	Uncertainty sources	Туре	
	Dosimeter			A (%)	B (%)
	1	0.473	Dose determination		2.48
16.70	2	0.477	Source decay		0.01
	3	0.474	Dosimeter thickness		0.03
Mean specific absorbance 0.475		0.475	Spectrophotometer reading		1.4
		0.473	Repeatability	0.25	
Uncertainty (95.45%) 0.027			Combined separated uncertainty	0.25	2.85
		0.027	Combined standard uncertainty	2.86	
			Relative expanded uncertainty (95.45%)	5.72	

Table 5 Uncertainty calculation for the irradiation point number #1 ($\lambda = 603$ nm).

Fig. 7 Calibration curves.

The Fig. 7 shows the calibration curve. Considering the curve fit factor, which is 0.78%, and the overall combined standard uncertainty of 3.46%, is obtained a value of 3.55% for the combined standard uncertainty, when using Eq. (9) for specific absorbance determinations.

Specific absorbance determinations with Eq. (9) are affected with an expanded uncertainty of $\pm 7.10\%$ at the 95% confidence level.

5. Conclusions

The values of $\pm 6.94\%$ and 7.10% for estimate of the expanded uncertainties achieved with the carried out measurements are typical of a routine dosimetry

system, that is of the order of $\pm 6\%$ (k = 2) [5].

This result makes clear that the use of PMMA Amber Perspex dosimeters in the routine dosimetry of the CDTN's Gamma Irradiation Laboratory is appropriate and contributes to the facility's irradiation processes quality assurance. That is true because a good routine dosimetry contributes to obtain reliable results in irradiation protocols.

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