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ANALYSIS OF UNCERTAINTY SOURCES IN DOSE MEASUREMENT AT AN INDUSTRIAL ELECTRON ACCELERATOR

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The procedure for in-situ/in-plant calibration of routine dosimeters used to support the product processing at the LU-10 industrial electron linac of NSC KIPT is described. The standard polystyrene calorimeters are used as reference dosimeters. The uncertainty budget for dose measurements with the Harwell Red Perspex 4034 routine dosimeters has been compiled. Based on the analysis of numerous measurements of absorbed dose using polystyrene calorimeters, the changes in their metrological characteristics have been observed related both to the duration of their use and the amount of absorbed dose accumulated over that period. The data obtained confirm the need for recalibrating reference dosimeters by an accredited calibration lab, as well as the importance of participating in the dosimetry intercomparisons, including those conducted under the IAEA projects.

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INTRODUCTION

The development, validation and monitoring of industrial radiation processing parameters involve a number of activities, the results of which depend on the accuracy of measuring the radiation dose delivered to an irradiated object. Depending on the accuracy and purpose of application, the dosimetry systems are divided into reference and routine ones.

A reference standard dosimetry system of the highest level is a primary standard dosimeter that is defined or widely recognized as one having the highest metrological quality and whose value is accepted without reference to other standards of the same value.

A reference dosimeter provide the highest metrological quality available at a given location or organization. Such measuring devices are used to calibrate dosimetry systems used for routine measurements (routine dosimeters). The expanded uncertainty for a reference standard dosimeter is typically about ±3% (k=2).

Routine dosimetry systems are used for routine (everyday) absorbed dose measurements, including process monitoring and dose mapping. The expanded uncertainty achievable in measurements with a routine dosimeter is typically about ±6% (k=2). To ensure a controlled uncertainty in absorbed dose measurement during product processing, a dosimetry system whose calibration can be traced to a national and/or international primary standard should be used.

Typically, a dosimetry system consists of a number of components, including a dose detector, as well as equipment and procedures for reading it and processing the measurement result. The equipment includes not only the instruments used to readout the dosimeter response, but also auxiliary instruments such as thickness gauges, reference samples and others. A documented procedure to ensure traceability to the appropriate national or international standard is also a necessary component of establishing absorbed dose measurements.

The "Accelerator" Science and Research Establishment of NSC KIPT develops and operates specialized linear electron accelerators, validates and conducts radiation sterilization processes for medical products, and also conducts the radiation tests. The basic installation for this work is the LU-10 electron linac (10 MeV, 10 kW), which annually processes over 10 thousand m^3 of products and carries out several dozen tests. These activities are organized by the Radiation Technology Laboratory (RTL), accredited against ISO/IEC 17025:2017 and ISO 14385:2016 standards.

This work focuses on the components, characteristics, and calibration procedures of dosimetry systems used in radiation processing, along with an analysis of the uncertainty associated with the dose measurements.

1. STANDARD DOSIMETRY SYSTEMS

Dosimetric support of the radiation treatment at the "Accelerator" Sc.&R Est. is based on the use of both standard dosimetry systems and those developed at RTL. The Harwell Red Perspex 4034 PMMA dosimeters (Harwell Dosimeters, UK) are used as the routine dosimetry systems for dose control, while the B3 dosimetric film based on cellulose triacetate (GEX Corp., USA) is used for dose mapping of an object in the transport boxes [1, 2].

A dosimetry system based on a polystyrene calorimeter (Polystyrene calorimeter, Technical University of Denmark, Roskilde, Denmark) is used as a reference standard for the calibration of routine dosimeters.

The cathodoluminescent and "Radiation Shadow" techniques, developed at RTL, are employed for online absorbed dose monitoring [3, 4]. A spectrophotometer, photo-scanner and digital ohmmeter are used as the readout devices in the dosimetry systems (Fig. 1).

The calorimetric dosimeter consists of a disk (calorimeter body) made of high-density polystyrene (ρ=1.05 g/cm^3), which is placed inside a heat-insulating box made of expanded polystyrene (ρ=0.03 g/cm^3), see Fig. 2 [5]. The temperature of the calorimeter is measured with a calibrated thermistor located inside the disk. The range of the dose measurements is 3…50 kGy, the uncertainty of dose measurement usually does not exceed 3.6% (k=2). The application of the reference dosimeters, as well as participation in dosimetry intercomparisons ensures metrological traceability of routine dosimeters to the UK National Standard of Absorbed Dose (Fig. 3).

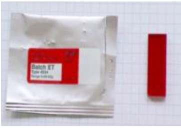
Harwell Red4034	B3 films	Polystyrene calorimeter
		
		
Spectrophotometer	Photo Scanner	Digital Ohmmeter

Fig. 1. Standard dosimetry systems

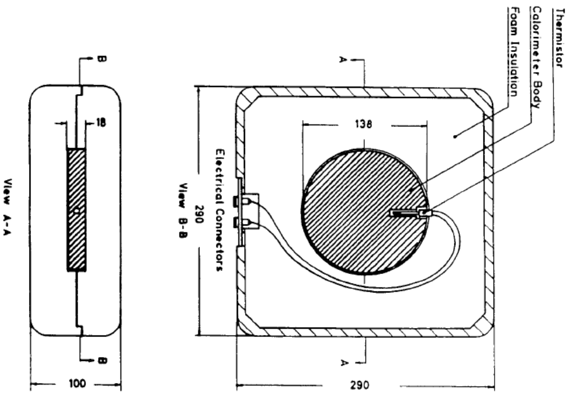


Fig. 2. Polystyrene calorimeter design [6]

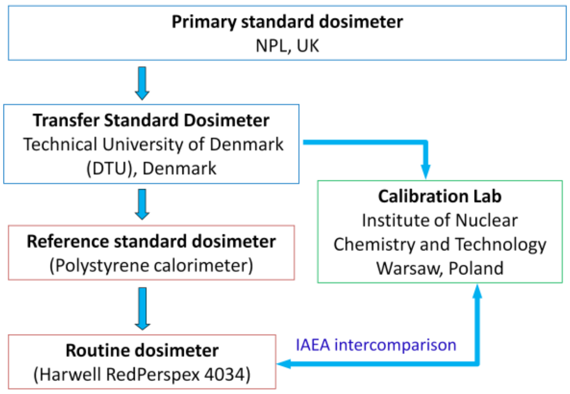


Fig. 3. Chart of traceability of dosimetry systems to relevant international standards

2. IN-SITU/IN-PLANT CALIBRATION OF ROUTINE DOSIMETERS

As Ukraine lacks a national standard for dosimeter calibration, the polystyrene calorimeters traceable to the UK National Standard are used as the reference ones. The procedure of in-plant calibration of routine dosimeters using the polystyrene calorimeters is carried out for each new batch of Red Perspex detectors and B3 film, but not less than once a year to check the stability of their calibration curves.

A typical procedure of the routine dosimeter calibration includes the following steps:

- combined irradiation with given set of doses in the range of 5…30 kGy of a polystyrene calorimeter and a set of calibrated routine dosimeters placed in a phantom having the same absorption characteristics of radiant energy as the calorimeter;
- determination of the dosimeter response corresponding to each dose value using a spectrophotometer;
- determination of the dose measured with the polystyrene calorimeter;
- obtaining the calibration curve corresponding to the measured response readings of the routine dosimeters against the absorbed dose measured with the polystyrene calorimeter;
- preparation of uncertainty budget table;
- documentation of the calibration procedure and results.

Let us consider in more detail the procedure for determining the absorbed dose using a polystyrene calorimeter. The calorimeter and the phantom, which contains 4 routine dosimeters, are placed on the transport container of the LU-10 facility conveyor and moved through the irradiation zone at a given speed that provides the required dose value. The calorimeter dose measurement was conducted in accordance with the ISO/ASTM 51631:2013 standard. For Red Perspex and B3 dosimeters, measurements were carried out using the ISO/ASTM 51276:2019 and ISO/ASTM 51275:2013 standards, respectively.

The data of the calorimeter temperature measurements before and after irradiation of them are extrapolated to the moment of irradiation. The two values T_1 and T_2 obtained as the result of the extrapolation are used as the temperature before irradiation (T_1) and after irradiation (T_2). Their difference (T_2-T_1) being proportional to the absorbed radiation energy, determines the average absorbed dose in the calorimeter body. To find the temperatures T_1 and T_2 by the extrapolation technique (Fig. 4), the two fitting straight lines are drawn: the first – according to the values measured before irradiation, and the second according to the values measured after irradiation. In this case, T_1 and T_2 are determined by the temperature values on the corresponding straight line at the moment of irradiation.

The data obtained were processed with a specially developed software. The calculation of the calorimeter temperature T ºC was carried out using the next expression:

$$T=\frac{B}{\ln(R)+A}-T_k\ , \qquad (1)$$

where R – is the resistance of the calorimeter thermistor (Ohm), measured with a digital ohmmeter; A, B, T_k – are the coefficients specified for each calorimeter thermistor in the calibration laboratory.

The following expression was used to calculate the calorimeter absorbed dose D (kGy):

$$D=(T_2-T_1-T_a)\cdot\left(k_1+k_2\cdot\frac{T_1+T_2}{2}\right)\cdot k, \qquad (2)$$

where T_a – is the heating of the calorimeter from the conveyor and accelerator (°C); k_1, k_2, k – are the calibration constants corresponding the temperature dependence of the heat capacity of the calorimeter body.

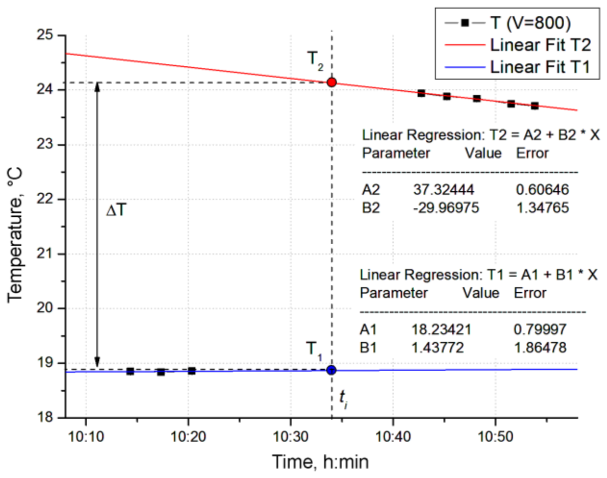


Fig. 4. The temperature data of the polystyrene calorimeter extrapolated to T_1 and T_2 (t_i – is the irradiation time). $\Delta T = T_2 – T_1$ is used as an input for the dose calculation

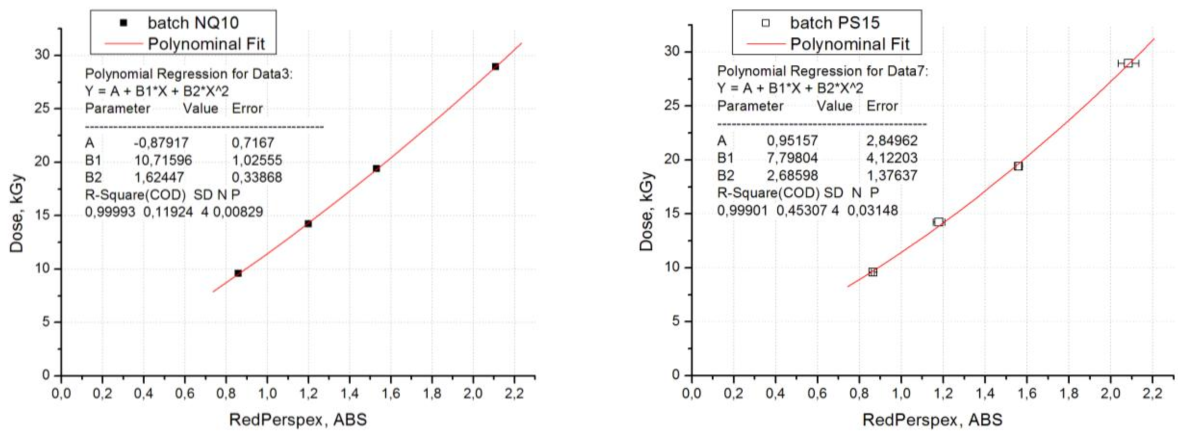
Table 1 shows the calorimeter temperature values before and after irradiation, as well as the doses that were determined by the method described above.

Table 1

Results of absorbed dose measurements by calorimeters

Calo-rimeter Num.	Con-veyor speed, cm/s	Temperature before irradiation T_1, °C	Temperature after irradiation T_2, °C	Temperature differen. ΔT, °C	Dose, kGy
2141	3.72	17.45	25.10	7.65	9.57
2142	2.48	18.96	30.02	11.06	14.23
2143	1.86	17.75	32.73	14.98	19.39
2141	1.24	18.21	39.88	21.67	28.95

As an example, Fig. 5 shows the calibration curves obtained in this way for the dependence of the response of Red Perspex dosimeters on the absorbed dose.



a *b*

Fig. 5. Dependence of the absorbed dose measured by the calorimeter on the optical density of Red Perspex dosimeters, and its 2nd degree polynomial extrapolation (a – batch NQ10; b – batch PS15)

Fig. 6 shows the residuals between the calculated and measured dose values by the calorimeter, to verify the calibration results of Red Perspex dosimeters.

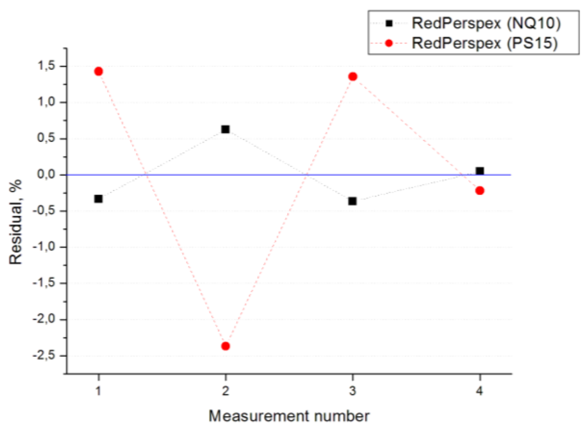


Fig. 6. The ratio of Red Perspex dosimeter doses (determined from the calibration curve in Fig. 5) to the polystyrene calorimeter dose

The calibration progress and results are documented in the form of reports, tables, etc. based on processing data.

3. ESTIMATION OF UNCERTAINTY IN ABSORBED DOSE MEASUREMENT

The precision of the reference dosimetry system directly affects the uncertainty of a calibrated system. Therefore, it is important that the reference system has low uncertainty and traceability to a national or international primary standard. The estimation of the uncertainty of dose measurements with a routine dosimeter should include all components, including those arising from calibration, reproducibility of the measurement result, stability of the reading device, and the influence of external factors (temperature, humidity, etc.) [7]. The list of the individual uncertainty components together with their values and methods of estimation is called the uncertainty budget. For example, consider the uncertainty budget of the dose measurements using the Red Perspex routine dosimeters (Table 2). This budget includes:

- uncertainty due to instability of measuring instruments (spectrophotometer, thickness gauge);
- uncertainty due to extrapolation by the calibration function;
- reproducibility of optical density measurements for dosimeters from the same batch;
- uncertainty due to temperature variations between calibration and measurement conditions.
- uncertainty of the reference dosimeter.

Table 2

Uncertainty budget for dose measurement with Red Perspex dosimeters

Component of uncertainty	Value	Probability distribution	Divisor	Relative standard uncertainty	
				Type A, %	Type B, %
Calibration (from laboratory Certificate)	3.6% (k=2)	Gaussian	2		1.8
Fit of calibration function	1.6% RMSE	Gaussian	1	1.6	
Temperature difference between calibration and usage	8 °C		1		1.3
Dosimeter to dosimeter scatter (reproducibility)	2.0%	Gaussian	1	2.0	
Difference in dose to reference and calibration dosimeters	0.5%	Rectangular	√3		0.3
Instability of instrumentation	1.8%	Gaussian	1		1.8
Combined uncertainty	–			**3.85**	
Expanded uncertainty (k=2)	–			**7.7**	

Dose measurement using Red Perspex routine dosimeters and its uncertainty estimation follow this procedure. The absorbance A of the dosimeter is measured using a spectrophotometer at a wavelength of λ=640 nm:

$$A = log_{10}\left(\frac{I_0}{I_t}\right), \quad (3)$$

where I_0 – is the intensity of the incident luminous flux, I_t – is the intensity of the transmitted luminous flux. The uncertainty of the absorbance measurement is about 1%.

The optical density *OD* (the absorbance per unit length, cm^{-1}) of the PMMA dosimeters is:

$$OD = \frac{A}{t}, \quad (4)$$

where t – is dosimeter thickness (cm) measured with an uncertainty of approximately 1.5%.

The combined uncertainty of *OD* for the Red Perspex dosimeters with thickness of t=0.3 cm is σ=1.8% (k=1).

The extrapolation errors in the calibration of Red Perspex dosimeters (see Fig. 6) do not show a significant tendency to change it with dose. In this case, the root mean square error (the square root of the average squared differences between predicted and observed outcomes, RMSE=1.6%) can be used as an approximation of the standard uncertainty of the fitting function [8].

The difference in the ambient temperature during calibration of dosimeters (23 ºC – June) and (15 ºC – February) is ~8 ºC. The temperature coefficient of Red Perspex dosimeters, in the temperature range of 0…25 ºC, is about ~0.1…0.16 %/°C, which leads to the change in the dose value within 1.3% [9, 10].

The B3 dosimetric film calibration and uncertainty estimation employs a similar procedure.

4. DOSE INTERLABORATORY COMPARISONS

According to the ISO/IEC 17025:2017 standard, a necessary component for monitoring the accuracy of calibration of measuring instruments in accredited testing laboratory is its participation in the interlaboratory comparisons. Within the framework of the regional IAEA projects, the intercomparisons are regularly carried out in the industrial dose range of the electron and gamma radiation, in which the "Accelerator" Sc.&R Est. takes part. In particular, such intercomparisons were carried out in 2022–2023 under the RER1021 project in two stages. At the first stage, the coordinator was the Danish Technical University, Denmark, at the second, the Institute of Nuclear Chemistry and Technology (Warsaw, Poland). The alanine dosimeters, delivered by the coordinator, were applied as the reference ones. The "Accelerator" Sc.&R Est. used Red Perspex 4034 as the routine dosimeters under study.

The procedure for preparing the dosimeters for comparisons was as follows. A phantom containing 4 reference alanine dosimeters, 2 Red Perspex dosimeters, and a calorimeter was installed in the transport container of the LU-10 conveyor (Fig. 7). The calorimeter was involved in the measurements to evaluate the dependence of its sensitivity on the integral dose received over the entire period of its operation. The phantom with the dosimeters and the calorimeter were irradiated at conveyor speeds that provided a set of absorbed dose values specified by the coordinator. The accelerator parameters and the irradiation time were recorded. After irradiation, the Red Perspex dosimeters and the calorimeter were removed for measurements. Dosimeter readings were taken approximately 20 min after irradiation. The absorbed dose of the calorimeter was determined by the method described earlier.

a *b* *c*

Fig. 7. Dosimeters located in the phantom (a). The phantom with dosimeters on the transport container (b). Cathodoluminescent glow of the irradiated phantom (c)

5. ANALYSIS OF INTERCOMPARISON RESULTS

The data of the intercomparisons are presented in Table 3. Fig. 8 shows the ratio of the dose measured with the alanine dosimeter to that measured with the calorimeter. The uncertainty of dose measurements was no more than 4.6% for the alanine dosimeter and 3.6% for the calorimeter (k=2). It appears that the calorimeter systematically underestimates the dose reading by about -6.6%.

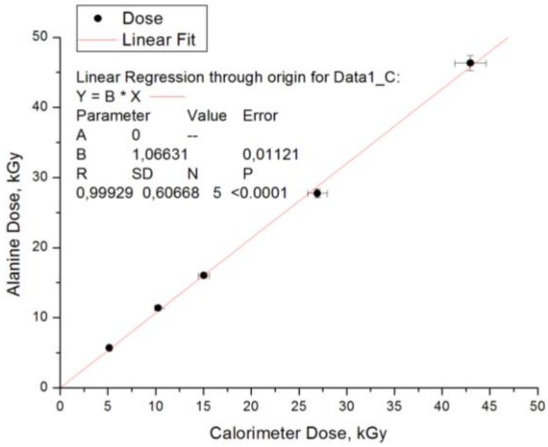


Fig. 8. Ratio of the dose measured with alanine dosimeters to the dose measured with calorimeter during the RER1021 interlaboratory comparisons

The deviation of the dose value measured with the calorimeters (corrected by factor +6.6%) from the dose measured with the alanine dosimeters is shown in Fig. *9*. After correction of the calorimeter readings, the difference in the dose value does not exceed ±4% (Table 3), which corresponds to the uncertainty of dose measurements with the calorimeter (k=2).

Table 3

Results of interlaboratory comparisons

Target Dose, kGy	Calorimeter Dose, kGy	Alanine Dose, kGy	Residual Calor. /Alanine, %	Calorimeter Dose (corr. +6.6%), kGy	Residual Calor., corr. /Alanine, %
5	5.15	**5.67**	-9.18	5.49	-3.15
10	10.27	**11.38**	-9.79	10.95	-3.77
15	15.05	**16.02**	-6.03	16.05	0.17
25	26.95	**27.74**	-2.86	28.74	3.59
40	42.96	**46.32**	-7.25	45.81	-1.10

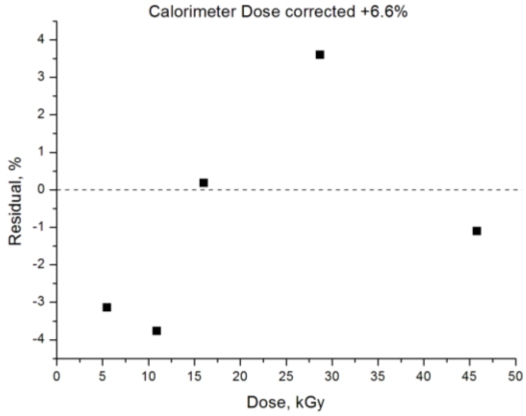


Fig. 9. Deviation of the dose measured with calorimeter (after correction by factor +6.6%) from the dose measured with alanine

6. RADIATION AGING OF POLYSTYRENE CALORIMETER

In the dose measurement with the polystyrene calorimeters, conducted at the "Accelerator" Sc.&R Est. from 2012 to the present, the several software packages were used. So, the Cern ROOT package (an open source C++ data analysis system) was applied during 2012–2018 [11]. The graphical package (a software for plotting and analyzing data) and the programs in the Python programming language are used from 2019 until the present. The software written in Python and Cern ROOT calculate the dose automatically according to a given algorithm (only the input data are required), and a graphical package is used interactively for a more detailed analysis of the measurement data. Table 4 shows the calorimeter dose values obtained with the use of these packages under the RER1021 interlaboratory comparisons.

Table 4

Dose values calculated with the different software packages

Target Dose, kGy	Graphing software Dose, kGy	Python Dose, kGy	Cern ROOT Dose, kGy
5	5.15	5.15	5.15
10	10.27	10.27	10.27
15	15.05	15.05	15.05
25	26.95	26.95	26.95
40	42.96	42.97	42.97

As it can be seen from the data in Table 4, the dose values determined using three different packages are completely consistent. This can be used as an additional approach to validating the software for dose determination with the polystyrene calorimeters.

The dependence of the dose value on the temperature difference of the calorimeter before and after irradiation (ΔT) measured in 2012–2014 and in 2023 is presented in Fig. 10. The slope coefficient in 2023 is roughly 4% lower than the average slope coefficient observed between 2012 and 2014. That coefficient in 2023 is roughly 4% lower than its average value observed between 2012 and 2014. The alteration in the calorimeter characteristics can be explained by the change in the thermal properties of the calorimeter body, its insulation and/or thermistor.

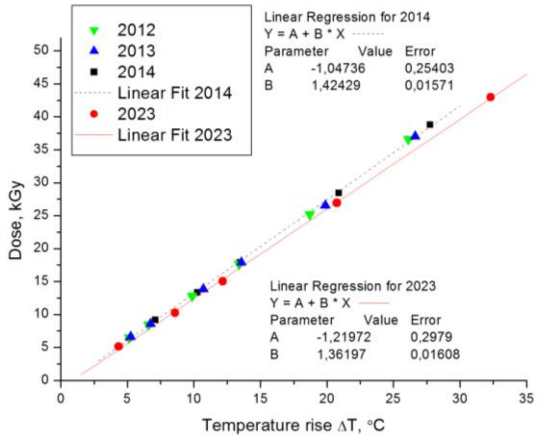


Fig. 10. Dependence of dose value on differential of calorimeter temperature before and after irradiation (ΔT) measured in 2012–2014 and in 2023

A more detailed comparison of the measurement data related to 2023 and 2012-2014 period is shown in Fig. 11.

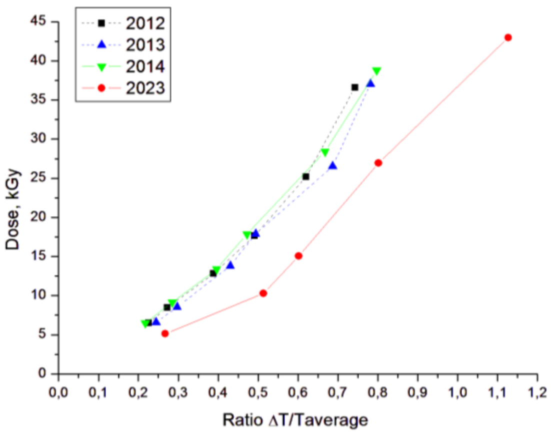


Fig. 11. Dose value dependence on the ratio of the differential of calorimeter temperature before and after irradiation (ΔT) to the average temperature $0.5{\cdot}(T_1{+}T_2)$

The calorimeter absorbed dose estimates were derived from the measurements conducted between 2012 and 2023. The data on the yearly absorbed doses obtained by the calorimeters are shown in Fig. 12. Table 5 shows the average doses per year and the total doses for that period for each calorimeter.

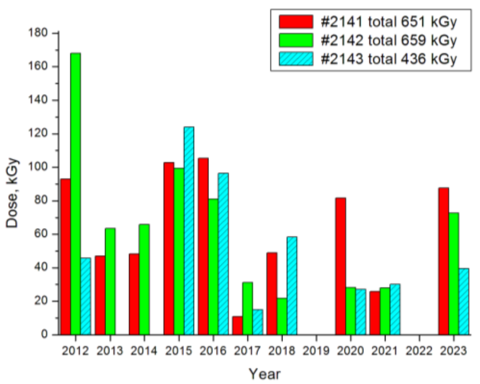


Fig. 12. Absorbed dose obtained by calorimeters for the period 2012–2023

Table 5

Average and total accumulated dose obtained by calorimeters for the period 2012–2023

Serial Number of calorimeter	Average dose obtained by calorimeter, kGy	Total accumulated dose, kGy
2141	65 ±33	651
2142	66 ±45	659
2143	55 ±38	436

Measurement data from 2007 to 2011 has not been retained, but based on the average dose value of about 62 kGy, it can be assumed that during that period each calorimeter received about 300 kGy. So, for the period of operation from 2007 to 2023, the estimated accumulated dose for each calorimeter was approximately 1000 kGy. That value does not exceed the manufacturer's established maximum accumulated dose of 2000 kGy.

Fig. 13 illustrates the relationship between specific heat capacity and irradiation dose in High Impact Polystyrene samples (HIPS), based on the data taken from [12]. HIPS exhibits a 2% increase in specific heat capacity with total accumulated dose of approximately 1 MGy. An increase in the specific heat capacity of the polystyrene leads to both a decrease in the temperature T_2 after irradiation and an increase in the cooling time constant of the calorimeter, which in turn also affects the determination of the temperature T_2 by the extrapolation technique described above.

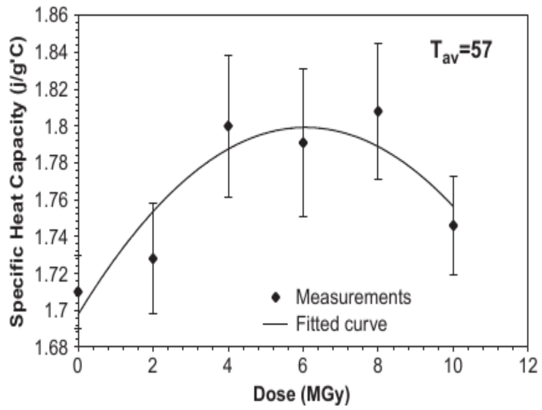


Fig. 13. Specific heat capacity of HIPS depending on irradiation dose [12]

CONCLUSIONS

The uncertainty of the absorbed dose measurement with the routine dosimeters used for radiation treatment monitoring is largely determined by the accuracy of their calibration with the reference dosimeters. When using the polystyrene calorimeters as reference ones, the systematic uncertainty associated with radiation aging of such devices should be taken into account. So, the specific heat capacity of some materials used in the calorimetric dosimeters as sensitive components may vary depending on the accumulated absorbed dose, thereby affecting the metrological characteristics of such devices. This is especially true for polymers, specifically for polystyrene, which is used to make reference dosimeters in the field of industrial dose measurements. Therefore, it's essential to recalibrate those systems periodically, depending on the total dose they have accumulated. Thus, taking part in interlaboratory comparisons coordinated by the accredited calibration laboratories, particularly under the IAEA projects, is an important element in ensuring the accuracy of dosimetric maintenance of the radiation technology processes.

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АНАЛІЗ ДЖЕРЕЛ НЕВИЗНАЧЕНОСТІ ПРИ ВИМІРЮВАННІ ДОЗИ НА ПРОМИСЛОВОМУ ПРИСКОРЮВАЧІ ЕЛЕКТРОНІВ

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Описано процедуру on-site калібрування робочих дозиметрів, що використовуються для супроводу обробки продукції на прискорювачі електронів ЛУ-10 ННЦ ХФТІ. Як референтні дозиметри використовуються стандартні полістирольні калориметри. Складено бюджет невизначеності вимірювань дози робочими дозиметрами Harwell Red Perspex 4034. На підставі аналізу численних вимірювань поглинутої дози за допомогою полістирольних калориметрів спостережено зміни в їх метрологічних характеристиках, пов'язані як з тривалістю їх використання, так і з кількістю накопиченої за цей час дози. Отримані дані підтверджують необхідність повторного калібрування референтних дозиметрів акредитованими калібрувальними лабораторіями, а також важливість участі у міжлабораторних порівняннях робочих дозиметрів, у тому числі, що проводяться у рамках проектів МАГАТЕ.