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Advancements in accuracy of the alanine dosimetry system. Part 2. The influence of the irradiation temperature

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Abstract

Systematic measurements of the temperature coefficient for alanine electron paramagnetic resonance (EPR) response have been performed for irradiation in the temperature range (10–50)°C and in the absorbed dose range (1–100) kGy at the dose rate 9.5 kGy/h. During the ^{60}Co˙-ray irradiation, ˙-L-alanine dosimeters were kept in a sealed aluminum holder that provided an effective heat exchange with the temperature-controlled environment. The time between the irradiation and signal measurements was standardized, and a reference sample fixed in the resonant cavity was used to correct the signals for small variations in the spectrometer sensitivity. The temperature coefficient for each dose was determined from approximately 30 experimental points processed by the weighted least-squares technique after the necessary statistical tests were done. The temperature coefficients thus determined were considerably lower than previously reported. The dose dependence of the temperature coefficient features a minimum at (20–30) kGy (about 0.135%/K) with higher values at 1 kGy (0.17%/K) and at 100 kGy ((0.175–0.19) %/K). With the exception of very high doses, no significant distinction was found between the temperature coefficients of Bruker and NIST dosimeters, which differ in shape and binder content.

Keywords: Alanine; EPR dosimetry; Temperature coefficient

1. Introduction

Appreciable heating of dosimeters during irradiation is an inevitable effect of any high-dose irradiation at moderate and high dose rates. Ideally a dosimetric system would be insensitive to temperature; in practice, however, application of an appropriate temperature correction is necessary. The alanine–EPR system, currently in wide use as a reference class dosimeter in the industrial and, to a lesser extent, therapeutic dose ranges, is radiation temperature dependent. The effect of irradiation temperature on the EPR signal is not large, but quite noticeable. Irradiation temperature increases of 10°C, which are very common in practice, result in EPR signal differences and potential dose errors of more than 1%, which are significant at the precision level presently achieved by this method. To make an accurate correction for differences in the irradiation temperatures for test and calibration dosimeters, one needs to know (i) the precise temperature dependence of alanine response and (ii) the precise temperatures of the test and reference dosimeters during irradiation.

Early researchers in alanine dosimetry established that, at least in the (0–50)°C interval, the amplitude of the EPR signal of irradiated alanine grows linearly with temperature. However, reported values of the

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Table 1
Temperature coefficients for alanine response reported in the literature

Author (year)	Dosimeter characteristics	Dose (kGy)[a]	Temperature range (°C)	**Temperature coefficient (%/°C)**	Dose rate (kGy/h)	Uncertainty	Comments
Deffner and Regulla (1980)	?-α-Ala + paraffin (? + ?); $D = 5$ mm, $h = 7.5$ mm	≤ 10	0–80	**0.2**	NA[b]	NA	Polyethylene vials
Regulla and Deffner (1982)	L-Ala + paraffin (90 + 10); $D = 4.9$ mm, $h = 10$ mm	≤ 10	−10–+90	**0.18**	9	NA	
Regulla and Deffner (1985)	L-α-Ala + paraffin (90 + 10)	NA	< 80	**0.18**	NA	NA	
Schneider et al. (1985)	?-α-Ala powder encapsulated in wax; $D = 5$ mm, $h = 8$ mm	0.2, 0.42	20–47	**0.3**	0.12	NA	10 MeV electrons
Kojima and Tanaka (1989)	DL-α-Ala + polystyrene (70 + 30); $D = 3$ mm; $h = 30$ mm	1	−40–+50	**0.29**	0.04	NA	Polystyrene vials?
Wieser et al. (1989)	?-α-Ala + paraffin (80 + 20); D = 4.9 mm, $h = 10$ mm	1–50[c]	−60–+50	**0.185**	2.7	NA	Pellets in the air flow in Al holder; misprint in eq. (1)[c]
Janovsky (1991)	L-α-Ala + polyethylene vinylacetate (30 + 70); 0.2 mm films	12	10–75	**0.13**	2.5	± 0.06	^{60}Co; Fixed ruby standard
		10	−196–−25	**0.4**			^{60}Co; Fixed ruby standard
		4.5	20–70	**0.13**			4 MeV electrons; fixed ruby standard
Kojima et al. (1992)	DL-α-Ala + polystyrene (70 + 30); $D = 3$ mm, $h = 30$ mm	1.4–100	0–70	**0.24**	7	Plot available	Polystyrene vials
Chen et al. (1992)	DL-alanine	1	NA	**0.237**	NA		Polystyrene vials
		10	NA	**0.222**	NA		Polystyrene vials
		100	NA	**0.252**	NA		Polystyrene vials
Tateishi et al. (1992)	DL-α-Ala + polyethylene (70 + 30)	1.4	2–50	**0.24**			
Mehta (1996)	DL-α-Ala + polystyrene (70 + 30); $D = 3$ mm, $h = 30$ mm	15, 45	5–40	**0.23**	NA	Plot available	Polystyrene vials
Kojima et al. (1996)	DL-α-Ala + polystyrene (70 + 30); $D = 3$ mm, $h = 30$ mm	0.1–10	−15–+30	**0.24**	NA	Plot available	Polystyrene vial, 30 min thermostatting, 1-day measurements; Mn(2 +) adjacent standard
Coninckx et al. (1996)	Various binders and dimensions	< 1	−268–+67	**0.21**	0.9–1.2		
	Various binders and dimensions	100	−268–+67	**~1.0**	0.9–1.2	Plot available	
Kojima et al. (1997)	DL-α-Ala + polystyrene (50 + 50); $D = 3$ mm, $h = 20$ mm	1	5–45	**0.25**	10	Plot available	Irradiation in air-conditioned Al cubic box; no vials, 30 min thermostatting, measurements within 1 day

[a] Co-60 gamma rays are assumed unless specified otherwise in the "Comments" column.
[b] NA = no explicit information on this point is available in the publication.
[c] Dose dependence of the temperature coefficient is represented by an empirical formula. Note: Eq. (1) as published in Wieser et al. (1989) is incorrect. The correct equation for the temperature correction factor is: $S(22\ \mathrm{C})/S(T) = 1-[(1.85 \times 10^{-3}) + 1.05 \times 10^{-5}(D-1) \times \mathrm{Th}(D-1)] \times (T - 22\ \mathrm{C})$ (A. Wieser, private communication, 1998).

slope of this dependence vary considerably (Table 1). Very few authors provide estimates of the uncertainty in the reported values, but the scatter of published data points revealed very large uncertainties. The increase in the EPR signal with irradiation temperature over a temperature range of ~50°C is not much greater than the normal scatter of points from replicate dosimeters irradiated at the same temperature; this makes precise determinations of the slope fairly difficult. Also, in many temperature-coefficient studies the dosimeters were irradiated in plastic vials, which are effective thermal insulators, and the dosimeters were assigned irradiation temperatures measured outside the vials. Another potential source of errors arises from intensity changes in the EPR signal of irradiated alanine during the first days after irradiation (Nagy and Desrosiers, 1996). With these conditions in mind, we undertook a new experiment to measure the irradiation temperature coefficient for L-α-alanine. Our experimental approach includes measures to standardize the time between the irradiation and signal measurements, ensure thermal equilibrium between the dosimeters and the temperature-controlled environment, and considerably increase the number of the measurements.

2. Experimental[1]

2.1. Dosimeters

Both commercial alanine dosimeters (Bruker Instruments) and dosimeters manufactured at NIST were used. Bruker dosimeters (Batch No. 3) contain 80% L-α-alanine and 20% polyethylene, and are 5 mm in diameter and 5 mm in height. NIST dosimeters (5 mm diameter, 2.7 mm height, containing 90% (by mass) L-α-alanine (Aldrich, 99 + %) and 10% polyethylene (Polysciences, MW = 700, 60 μm) as a binder), are prepared by the following procedure. The bulk alanine crystals are ground with a centrifugal mill (Brinkmann) fixed with a 0.5 mm ring sieve. Alanine crystals in the 53–125 μm range are selected with a vibrating sieve (Brinkmann). Appropriate weights of sieved alanine and polyethylene were blended in a powder blender (Paterson–Kelly). The mixture is pressed into pellets with a Manesty hand-tabletting press to produce alanine dosimeters 4.9 mm in diameter with an average height of 2.7 mm. The dosimeters are placed in a 130°C oven for 30 min, followed by 5 min in an 85°C oven.

2.2. Irradiation

Irradiations were conducted in a Gammacell (Nordion, Canada) with a dose rate of about 9.5 kGy/h. The specially designed aluminum holder used to achieve thermal equilibrium is shown in Fig. 1. Six replicate pellets were irradiated at each dose–temperature combination. Temperature control during the pre-irradiation thermal equilibration of the system and dosimeter irradiation was performed with a controlled-temperature airflow from a Turbo-Jet system (FTS Systems, NY, USA) and a Type-T thermocouple inserted into the body of the aluminum holder. The flow rate was about 140 L/min. To pre-equilibrate the dosimeters to the desired temperature, the dosimeters were placed in a thermally equilibrated holder for at least 1 h prior to placing them in the irradiation chamber. At the conclusion of the irradiation they were immediately transferred to the room-temperature environment.

The dosimeters used in this study were preconditioned at the relative humidity of the measurement laboratory ($30 \pm 5\%$ R.H.). They were sealed in the holder during irradiation.

2.3. EPR measurement

The EPR signals of all the irradiated dosimeters were measured approximately 72 h after irradiation. This time interval was selected based on our earlier data on the short-term evolution of the EPR signal of irradiated alanine (Nagy and Desrosiers, 1996). On the third day after irradiation signal changes are slowest for most dose levels, which is favourable for high-precision measurements. Signal measurements were made using an ECS106 spectrometer (Bruker Instruments) with a 4103TM cavity at room temperature. The field modulation frequency was 50 kHz. The microwave power utilized, 0.25 mW, was the highest within the linear range of the signal amplitude dependence on the square root of the microwave power. The sweep rate was low enough to make the passage between the derivative extrema longer than 10 time constants. A 1.8 mT central portion of the alanine signal was recorded, and its peak-to-peak amplitude was measured. A special quartz holder (a high-precision tube with an inner diameter (5.0 mm) closely matched to the diameter of the dosimeters (4.9 mm), and an inner fused-quartz support) was permanently mounted in the cavity to provide highly reproducible dosimeter positioning. Dosimeters were inserted and removed with a pneumatic manipulator. The EPR signal of each dosimeter was recorded at two orientations with respect to

[1] The mention of commercial products throughout this paper does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that products identified are necessarily the best available for this purpose.

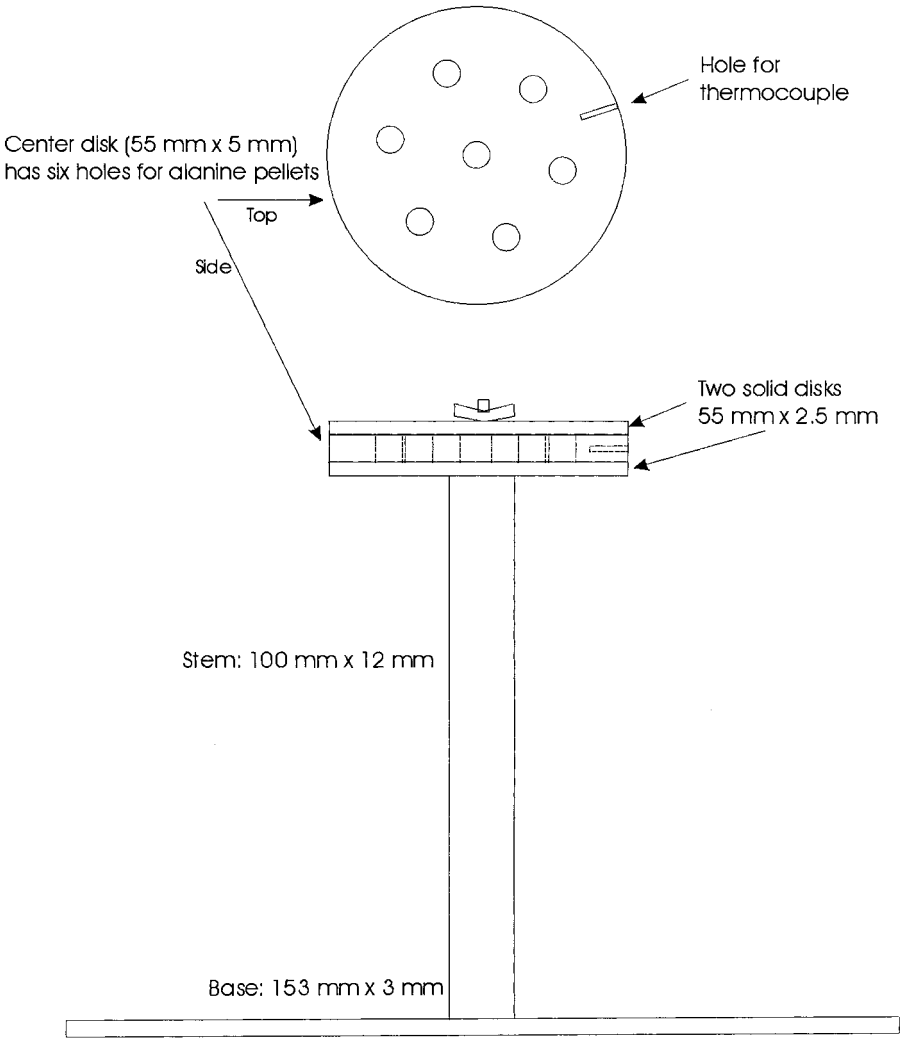

Fig. 1. Aluminum holder used for dosimeter irradiation at controlled temperatures.

the Zeeman field, differing from each other by approximately 90°. Prior to rotation and removal of the alanine dosimeter, a Cr(3+) signal from a ruby crystal fixed in the cavity was recorded without changing the settings of the critical parameters (microwave power, modulation amplitude). The ratios of the alanine signal amplitude to the ruby signal amplitude were used as alanine signal values in the data analysis.

2.4. Data processing

For each dose studied, five groups of pellets (six replicate pellets in each group) were irradiated at approximately 10, 20, 30, 40 and 50°C. The averaged ruby-normalized signals for the two orientations of each dosimeter were plotted vs the average irradiation temperature, thus giving 30 points in total. A typical plot is shown in Fig. 2. Normally, a within-group relative standard deviation (RSD) was 0.6–0.8% for Bruker dosimeters and 0.4–0.5% for NIST dosimeters. The hypothesis of linear regression was tested by comparing the average within-group scatter of the points to the scatter of points with respect to a least-squares linear fit using the *F*-test at the 95% confidence level (Draper and Smith, 1981). After the linearity hypothesis was confirmed to be reasonable, the data were treated by a weighted least-squares technique (with the statistical weights taken proportional to the inverse within-group variances) to generate the prediction band for *Y*-values. In some cases, one to three points had to be rejected as outliers on the basis of their positions outside of the prediction band. The refined data set was reprocessed by weighted least squares, and the characteristics of the resulting fit were used to calculate

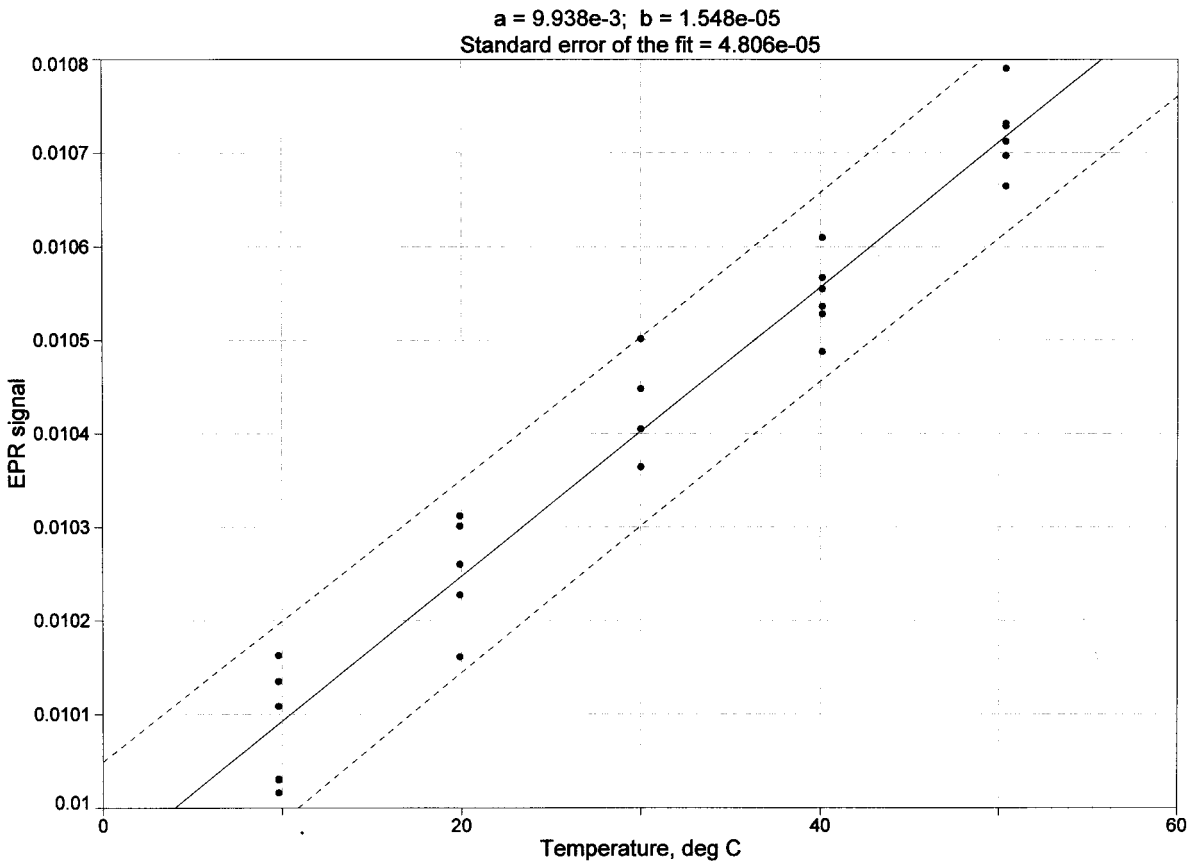

Fig. 2. A typical plot of the alanine dosimeter response on irradiation temperature using Bruker dosimeters (dose 13 kGy). Some of the closely positioned points are not resolved. The dotted lines show the prediction band; no outliers occur in this experiment.

the temperature coefficient. The temperature coefficients presented in this paper are percentages of the regression slope (signal units per °C) with respect to the predicted signal value in the middle of the temperature range (30°C) and, thus, are expressed in the units %/°C (%/K). The combined standard uncertainties in the temperature coefficients were calculated according to the law of propagation of uncertainty: the standard deviation of the slope was used as an uncertainty estimate for the numerator and the standard error of the fit served as an uncertainty estimate for the denominator.

3. Results and discussion

Our preliminary experiments have shown that irradiation of dosimeters in plastic vials (which were frequently used to determine the temperature coefficient in the past), does not provide the required thermal equilibrium between the pellets and the outside air whose temperature is measured. When Bruker pellets were irradiated at 50°C in a polystyrene vial with a thermocouple inserted through a small hole in the middle of the vial side wall (Fig. 3), the thermocouple reading was very close to the controlled air temperature outside the vial, but the EPR signals of the pellets exhibited a systematic trend: they increased from the peripheral areas to the vial center that hosted the thermocouple. When the same experiment was repeated at 10°C, the results were similar, but the signal trend was inverse: the signals decreased from the top and bottom of the vial to its center. A reasonable explanation of these observations was lack of thermal equilibrium in the system. The metallic thermocouple not only measured the temperature, but it also served as a thermal channel equilibrating the temperature of the air outside of the vial and in the area inside the vial where the thermocouple resides. The farther a dosimeter was located from this area of the thermocouple-affected temperature, the bigger was the deviation of its response from the responses of the center dosimeters. The direction of the deviation corresponded to the difference between the temperature outside of the vial

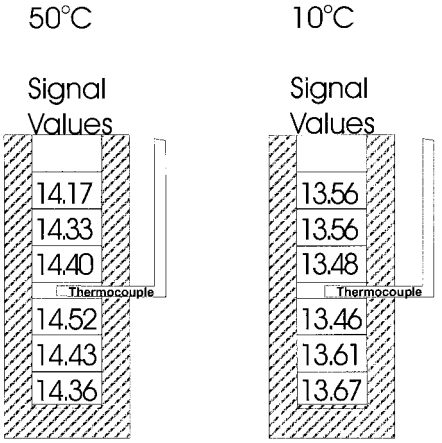

Fig. 3. Dependence of the EPR responses of alanine dosimeters on their position in a polystyrene vial with a thermocouple inserted in the vial center. The vials with the dosimeters were kept at the specified temperatures for 45 min, the subsequent irradiations to 10 kGy took about 1 h.

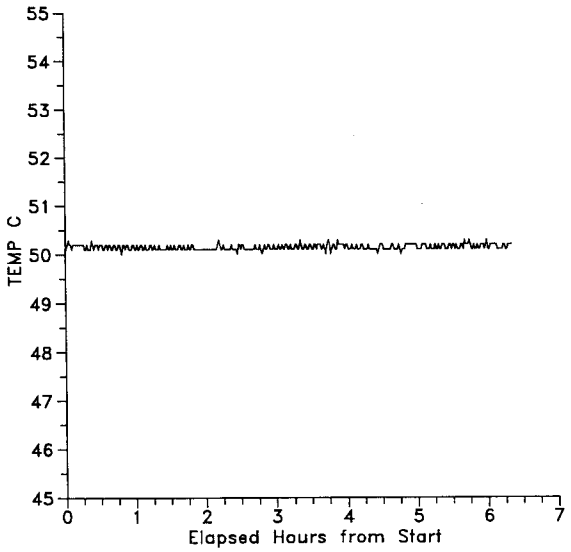

Fig. 4. Variations in the temperature of the thermostatted aluminum during the 1.25-h pre-irradiation period and 5-h irradiation at a dose rate of 9.5 kGy/h.

and the original room temperature of the pellet. An experimental design that would maintain the dosimeters at the measured temperature during irradiation was needed.

A holder made of aluminum (Fig. 1) proved to be suitable for this study. This material provided a good compromise: aluminum exhibits one of the highest thermal conductivities of all metals and alloys, while its atomic number is one of the lowest, which minimizes heating of the holder material itself under irradiation. The shape of the holder was designed to facilitate the needed thermal equilibrium. The dosi-

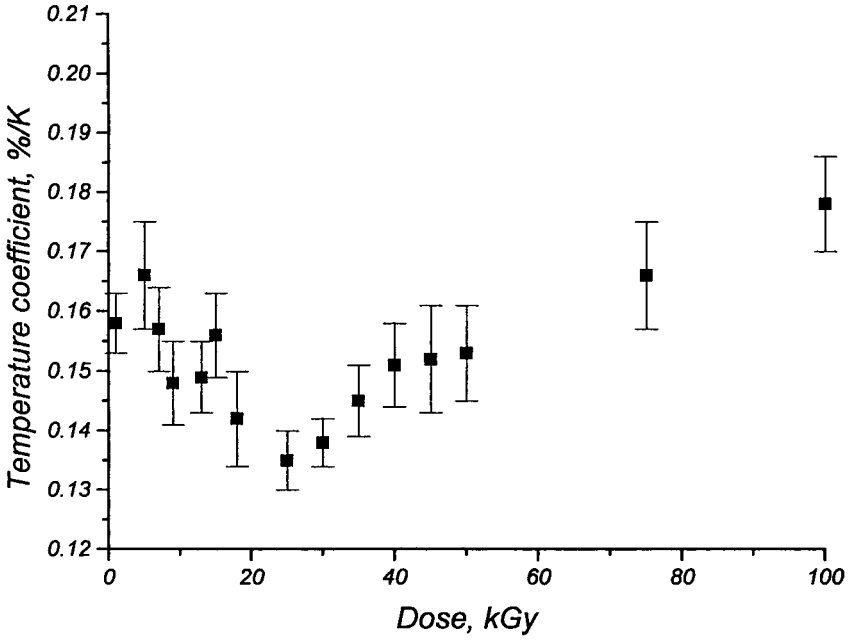

Fig. 5. Dose dependence of the temperature coefficient for Bruker dosimeters (Batch No. 3). The error bars show uncertainties calculated as described in Section 2.4.

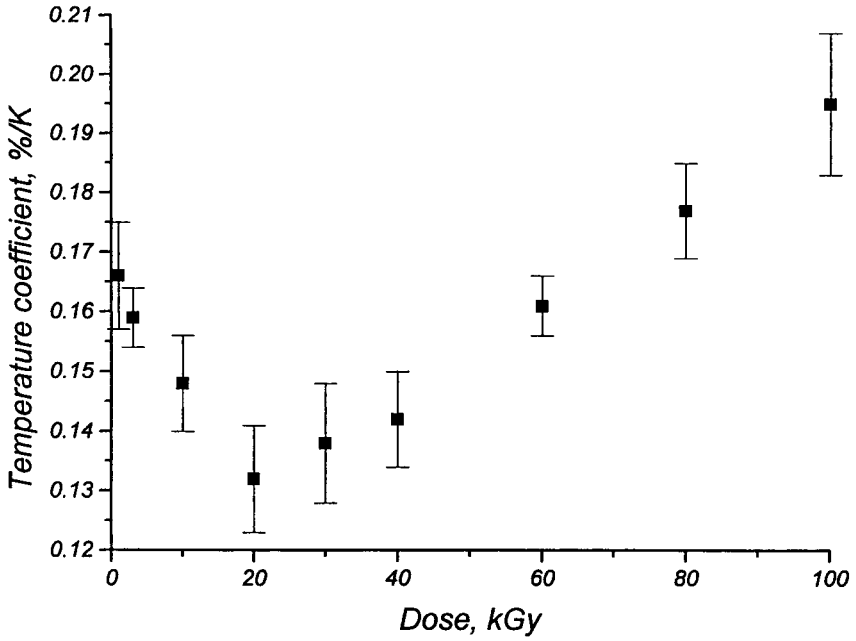

Fig. 6. Dose dependence of the temperature coefficient for NIST dosimeters. The error bars show uncertainties calculated as described in Section 2.4.

meters held in the center disk were separated from the temperature-controlled air on each side by just 2.5 mm-thick highly thermoconductive aluminum layers, and the large aluminum base attached to the upper, dosimeter-holding, part by means of a heat-conducting aluminum stem served as an additional radiator. An air flow of more than 2 L/s in combination with a large aluminum–air interface ensured an effective heat exchange. The long-term temperature stability of the system can be illustrated by the typical temperature data shown in Fig. 4. The fact that no temperature changes were observed between the periods of preliminary thermostatting and long-term irradiation demonstrates an effective removal of the irradiation-produced heat. The temperature of the holder was measured at 1 min intervals. The mean of these values was used in the regression analysis. It is also noteworthy that the aluminum assembly used in this study, in contrast to that employed by Wieser et al. (1989), does not keep the dosimeters exposed to the air flow. Multi-hour ventilation with heated air was likely to significantly change the moisture content of the dosimeters and, thus, bring in additional complications related to variations in kinetics of the radical reaction and Q-factors of the spectrometer cavity (Sleptchonok et al., 1999).

The temperature coefficients obtained are presented in Fig. 5 (Bruker) and Fig. 6 (NIST). For this batch (No. 3) of Bruker dosimeters, a somewhat greater interspecimen scatter and anisotropy of their EPR signals necessitated additional measurements to more reliably define the temperature dependence. Nevertheless, both plots clearly show a strong non-monotonic dependence of the temperature coefficient on the absorbed dose. The coefficient exhibits a pronounced minimum at doses of 20–30 kGy and increases for both higher and lower doses.

As can be seen from Table 1, some studies have reported dose dependence of the temperature coefficient over a wide dose range (e.g. Kojima et al., 1992, 1996). Regulla and Deffner (1982) mentioned an increase in the temperature coefficient at doses above 10 kGy, but did not specify this dependence explicitly. Wieser et al. (1989) quoted an empirical equation showing no dose dependence of the temperature coefficient below 10 kGy and its monotonous increase at higher doses. To our knowledge, there is only one paper reporting a minimum in the temperature coefficient dose dependency (Chen et al., 1992). Interestingly, although these quoted temperature coefficients are significantly higher than those found in the present study, the ratios of the measured coefficients at different doses within each study are comparable.

For dosimetry, a key finding of the present study is that the temperature coefficient is significantly lower than reported earlier, and, consequently, the potential errors from differences in irradiation temperatures are not as large as previously thought. Moreover, the lowest values of the coefficient are in the dose range 15–40

kGy, where most industrial dose measurements are performed. In fact, only our highest temperature coefficients, observed at the both extremes of the 1–100 kGy range, approach the lowest values reported in the literature for pellets.

The differences between our data and the values reported in Table 1 are as large as a factor of two. Several factors may be responsible for such discrepancies. First, the binder type/concentration and dosimeter shape may play a role, because both these factors definitely affect the heat transport and exchange between the dosimeter (which is not a very good thermal conductor) and the environment. In fact, the only noticeable discrepancies between our coefficients for the NIST and Bruker pellets, which differ in these characteristics, were observable at high doses, where heating effects of radiation are most pronounced. Also, it may not be a coincidence that the lowest temperature coefficient reported in the literature (0.13%/°C, Janovsky, 1991) corresponds to a film where heat transport should be most efficient.

Second, a reason for differing values may be due to irradiation of the dosimeters in plastic vials, which are, in fact, thermal insulators. It is difficult to predict a relationship between the real temperature of the dosimeters in the vial, the temperature of the plastic vial walls (which do not provide rapid heat transfer and get heated by irradiation themselves), and a controlled temperature of the air flowing outside. Therefore, the temperature measured by other researchers most probably did not represent the real temperature of the dosimeters, and might have resulted in a biased coefficient. Our limited trial experiments with closed polystyrene vials resulted in higher coefficients for high doses.

Third, the time between the irradiation and EPR signal measurements may be a substantial factor. For example, Kojima et al. took their measurements within one day of irradiation. According to our data (Nagy and Desrosiers, 1996), the signal changes are most pronounced during this time frame. Conversely, measurements reported here were obtained on the third day following irradiation, when the changing signal substantially levels out.

Finally, comparing two values is difficult when the uncertainty of one is unknown. In our experiments, each temperature coefficient value was obtained from about 30 experimental points, each representing two dosimeter signal measurements. For the Bruker and NIST dosimeters, the coefficients have been measured at 15 and 9 doses, respectively, and the values obtained agreed with each other both within each dosimeter type and between them. There is no published evidence of such abundant data in the previous experiments. In fact, the plots published by different authors suggest that the temperature coefficients were often calculated from a limited number of data.

As mentioned in the Introduction, an accurate knowledge of the temperature coefficient is only a half of the solution of the temperature-dependence correction problem. Exact temperatures of the test and reference dosimeters during their irradiation are equally important. In a sense, the latter is more difficult. First, most radiation sources do not have any kind of temperature control. Second, an accurate measure of the dosimeter temperature is needed, not of the air separated from them by an effective thermal insulator. Of course, there exists some relationship between the temperature of the pellet inside the vial and the external air temperature. However, this will obviously be determined by the effectiveness of the heat exchange, which is dependent on the throughput of the air pump, air dynamics in the chamber, dose rate, closeness of the other radiation-heated objects to the vial, and so forth. It is impossible to make all these parameters identical both in the test and reference source. The complex processes of slow heat exchange in such systems using thermoinsulating plastics make the standardization difficult. One approach to solving the temperature correction problem may be to change the vial material from a plastic to a much more thermoconductive substance. Although one might thus improve knowledge of the actual temperature of the pellet, the use of good thermal conductors such as metals for the vial/buildup material would require significant corrections (with their uncertainties) in the dosimetric analysis to account for the perturbing effects of the non-waterlike material.

4. Conclusion

This systematic study has shown that the temperature dependence of the L-α-alanine EPR signal is not as steep as previously reported. Nevertheless, it is significant and must be taken into account for any precise dose measurement. The temperature coefficient significantly depends on dose, and this dependence is not monotonic. Due to the unusually large body of data obtained in this study and the additional precautions taken for the first time in such kind of experiments, the temperature coefficients should be more reliable than those published before (American Society of Testing Materials, 1998) and it is recommended that they be used in practice.

In performing temperature corrections, a dosimetrist should take into consideration the following issues:

- reliability of the temperature coefficient used (number of measurements used for its determination; details of the experiment that produced the value);
- applicability of that value to a particular type of dosimeters (specific dosimeter shape, binder type and concentration);

- dose rate; and,
- reliability of the dosimeter temperatures in the calibration experiment.

Additional experiments may be necessary to verify the applicability of available values in specific cases.

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