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The use of thermistors in the NPL electron-beam calorimeter

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ABSTRACT

Thermistors have been employed in the primary standard electron-beam graphite calorimeter for several years. This report describes the system developed for calibrating thermistors and the results of investigations into self-heating and dose response. The overall uncertainty in measuring temperature rises is estimated to be $\pm 0.1\%$ at the 95% confidence level. The self-heating of the thermistor type used in the calorimeter was determined to be constant over the temperature range 14-29 °C. The same thermistor type showed no change in response within the uncertainties to an accumulated dose up to 3000 kGy. A second type of thermistor (to be used in a new calorimeter) showed no change in sensitivity although there was a change in absolute resistance of approximately 0.1% per MGy.

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Approved on behalf of Chief Executive, NPL,
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1 INTRODUCTION

Thermistors, or thermally sensitive resistors, were first developed during the Second World War. Over the forty years since then their use in temperature sensing has become widespread. Two types of thermistor have been developed: NTC (negative temperature coefficient) and PTC (positive temperature coefficient) exhibiting very different properties. These two types lend themselves to different applications with NTC thermistors most commonly used for temperature measurement and control. This report deals exclusively with the NTC type.

Although thermistors have been available for many years their application to radiation dosimetry is more recent. Their use in this field is a consequence of the development of calorimeters to measure absorbed dose in ionizing radiation beams. At the NPL the primary standards of absorbed dose for both high energy x-rays and electron beams are graphite calorimeters employing thermistors.

This report deals with the electron-beam graphite calorimeter and covers the development of thermistor usage in that device. Measuring absorbed dose accurately depends on accurate temperature measurement and therefore much work has been carried out to obtain improved accuracy in the thermistor system. The main purpose of the report is to provide technical detail on the developments made at the NPL but it also attempts to compare this approach with that of other researchers.

2 THERMISTORS IN RADIATION CALORIMETRY

The mean absorbed dose, D_a , in an absorber a is defined¹ as

$$D_a = \frac{dE}{dm} \quad (1)$$

where dE is the energy imparted to the mass dm of the absorber. If no energy is lost in chemical reactions or changes of state then this reduces to

$$D_a = c_a \Delta T \quad (2)$$

where c_a is the specific heat of the absorber and ΔT is the temperature rise. Therefore knowledge of the specific heat and measurement of the temperature rise leads directly to the absorbed dose. This is the basis of radiation calorimetry.

Equation 2 is complicated by the fact that absorbed dose is material dependent and therefore any foreign material present (such as the temperature sensor) will affect the measurement. Unless the temperature sensor is of the same composition as the absorber it must be made as small as possible. The former is ruled out since the calorimeter material is usually graphite or water; therefore the choice of sensor is determined by size (along with other considerations such as sensitivity, robustness, etc). Platinum resistance

thermometers (PRTs) are too big and insensitive. Thermocouples are small enough but their sensitivity only allows their use at high doses (above 1 kGy) and the standard PVC insulation degrades in radiation beams. Thermistors can be made very small and are radiation resistant (see later) and are therefore the best choice. All calorimeters designed for radiotherapy use employ a thermistor as the sensing element.

Much has been written on the theory, construction and use of thermistors so only a brief overview will be given here. A detailed discussion is given by Macklen². NTC thermistor materials are produced by heat treating a mixture of metal oxides to around 1100°C so that they combine chemically to form a mixture of compounds which exhibit semiconducting properties. The thermistor resistance R at temperature T is approximated by

$$R = R_0 \exp \left[B \left(\frac{1}{T} - \frac{1}{T_0} \right) \right] \quad (3)$$

where R_0 is the known resistance at temperature T_0 (usually taken as 298 K), T and T_0 being measured in Kelvin. The constant B is a function of the thermistor material. Modern manufacturing techniques can result in a very small tolerance on the B -value. The temperature coefficient of resistance, α , is given by

$$\alpha = \frac{1}{R} \cdot \frac{dR}{dT} = -\frac{B}{T^2} \quad (4)$$

and is typically 4% per K. This value is larger than for the other commonly used temperature sensors - thermocouples and PRTs.

There are various ways to use thermistors in calorimetry. The simplest method is to calibrate and measure the resistance directly. This eliminates the need for any complex circuitry but does not have the accuracy of other systems. The large measuring current involved in such resistance measurements (typically 0.1-1 mA) leads to a large thermistor self-heating and thus an additional uncertainty in the temperature measurement. The main application of this method is to high-dose calorimetry where the large temperature rises experienced allow self-heating to be neglected. The majority of calorimeters use an electrical bridge arrangement - either AC or DC. The choice usually depends on factors such as accuracy, resolution, signal-to-noise and design simplicity. At the NPL examples of both are in use: the x-ray microcalorimeter³ uses an arrangement of 3 AC bridges while the electron-beam calorimeter employs a much simpler single DC Wheatstone bridge⁴.

A schematic of the electron-beam calorimeter is shown in Figure 1. The Wheatstone bridge is constructed from bulk-metal-foil resistors (temperature coefficient ± 0.6 ppm per K). The precision DC supply is designed specifically for the calorimeter⁵ and gives a very stable output voltage of 1.41 V (24 hour stability is of the order of ± 1 μ V). The thermistor used is a glass bead type with a nominal resistance of 47 k Ω (@298 K), diameter 1.5 mm and length 5 mm. The balance resistor (R_{bal} in the diagram) is chosen to give zero bridge output close to 20°C which is the normal temperature environment in which the calorimeter is operated. Due to variations in R_0 between thermistors of the same type the

balance point can be in the range 17-23 °C. The measurement of the specific heat of the graphite calorimeter is described by Williams *et al*⁶.

3 EXPERIMENTAL SET-UP FOR THERMISTOR CALIBRATIONS

It has been noted earlier that thermistors can be used in AC or DC bridge systems. There is a further distinction that can be made and that is in the method of calibration. Three main methods are summarised in the table below.

Calibration method	Thermistor	Example
substitution of radiation beam by electrical power input	calibrated <i>in-situ</i>	NPL microcalorimeter ³
comparison with a calibrated temperature sensor	calibrated <i>in-situ</i>	Domen-type water calorimeter ⁷
comparison with a calibrated temperature sensor	calibrated separately then placed in calorimeter	NPL electron-beam calorimeter ⁴

Each has its own advantages - the *in-situ* method is closest to the actual irradiation conditions although it tends to make the calorimeter design more complex. A separate thermistor calibration makes for a simple calorimeter and enables one to optimize the calibration conditions. A comparison of the two NPL calorimeters⁴ gives close agreement within the uncertainties indicating that the different techniques do not introduce any large systematic errors.

The procedure for calibrating the thermistor/bridge arrangement of the electron-beam calorimeter has been described in detail previously by Burns and Morris⁴; it is summarised briefly here, not least because the procedure has been further developed since then. A schematic of the apparatus used is shown in Figure 2. The starting point for the procedure is a PRT, which is calibrated in terms of absolute temperature by comparison with triple-point cells. The thermistor/bridge out-of-balance voltage is then compared with the resistance of the PRT over the required temperature range. Steps of 1 K are used and at each temperature the system is allowed to settle to within ± 10 mK before a measurement is taken.

The main developments of the system described in reference 4 are:

- i) the graphite block housing the PRT and thermistor. Originally the thermistor was calibrated in the small core of the calorimeter with the PRT in an identical graphite disc, the two being mounted back-to-back. However it was found that the PRT temperature varied slightly with insertion depth. A larger block ensures that the entire PRT probe is in graphite, thus removing a possible source of error in the calibration.
- ii) the dip cooler. This allows a greater temperature range over which thermistors can be calibrated. There are two advantages of this - a more accurate determination of the thermistor sensitivity at normal operating temperatures and the ability to use the calorimeter in more hostile, less well controlled environments. The exposure rooms at the NPL linear accelerator facility are kept around 19-20 °C (daily stability ± 0.1 K) and the normal calibration range is 16-35 °C. The calibration is extended to higher temperatures as the calorimeter is also used at high dose rates (typically 10 kGy min⁻¹) where temperature rises of several degrees are experienced. In addition this larger temperature range makes fitting higher order polynomials less sensitive to noise (see later).
- iii) computer control. This major improvement has made calibrations much less labour intensive and removed a possible source of human error in taking manual readings. The computer constantly monitors the PRT and bridge outputs via the IEEE bus and displays them on the VDU screen. This makes it easier to determine when the temperature has stabilised enough to take a measurement (this can take up to an hour). In addition the computer is also used to control a separate heater unit via a 16-bit DAC, resulting in fully automated operation. Computer operation has reduced the random uncertainty of a calibration by roughly a factor of five (see later).

4 DATA FITTING

Having obtained a set of out-of-balance voltage versus temperature data the next step is to fit some model to the data. Referring to Figure 1 this voltage, V_{br} , is given by

$$V_{br} = V_s \left(\frac{R_{bal}}{R_{th} + R_{bal}} - \frac{R_4}{R_3 + R_4} \right) \quad (5)$$

where V_s is the supply voltage. The theoretical curve is S-shaped going from $V_{br} = +\frac{1}{2}V_s$ at low temperatures ($R_{th} \rightarrow \infty$) to $V_{br} = -\frac{1}{2}V_s$ at high temperatures as $R_{th} \rightarrow 0$. Around balance the data is best fitted by a polynomial

$$T = \sum_{i=0}^n a_i V_{br}^i \quad (6)$$

the order being determined by the temperature span chosen. Experience has shown that a quadratic ($n=2$) is sufficient for a 4 K range, a cubic for a 15 K range and a fourth order for a 20 K range. As higher order fits ($n>3$) are sensitive to noisy data a good experimental set-up and procedure, as described earlier, is needed. The best fits obtained to date (40 points over 15-35 °C) give an rms deviation of 0.27 mK with a mean uncertainty on the gradient estimated to be $\pm 0.03\%$ at the 95% confidence level. An example is shown in Figure 3. By way of a comparison the original procedure of manual calibration gave a typical rms deviation of 6 mK with an uncertainty on the gradient of $\pm 0.15\%$.

The uncertainty quoted above is the random (statistical) uncertainty. It does not include any uncertainty in the PRT calibration (estimated to be $\pm 0.1\%$ at the 95% confidence level⁴) nor does it include any systematic effects of the calibration procedure. A number of these were investigated and are described later.

Accurate calorimetry requires that the thermistor response does not change once it has been calibrated. Any gross changes should show up in the routine comparison of the calorimeter with other dosimeters but this may not always be the case. The only sure way of avoiding such a possibility is regular recalibration of the thermistor. By comparing two calibrations it should be possible to detect any change in thermistor response. Referring to equation 3 changes could occur in both R_0 and B . The way to obtain information on this is to compare the fitted temperature T and gradient dT/dV_{br} over the calibration range. For unirradiated thermistors this gives a measure of the repeatability of the calibration procedure. Results to date have shown very good repeatability with differences in dT/dV_{br} of 0.03%, close to the experimental uncertainties of each calibration.

5 FURTHER INVESTIGATION OF POSSIBLE SYSTEMATIC ERRORS

5.1 THERMISTOR SELF-HEATING

Electrical power dissipation in the thermistor raises its temperature above that of its environment. This effect is non-linear as the thermistor resistance and thus the power dissipation changes with temperature. It is therefore desirable to make this self-heating as small as possible to minimise any effect it may have on the calibration. The power dissipated, P , in the thermistor is given by

$$P = V_s^2 \cdot \frac{R_{th}}{(R_{th} + R_{bal})^2} \quad (7)$$

There are therefore three ways to reduce this power dissipation: reduce the supply voltage, increase the balance resistor or increase the nominal thermistor resistance. Each

method alone reduces the sensitivity of the bridge system which is undesirable. However, increasing R_{th} and R_{bal} in the correct ratio decreases the power dissipation without loss of sensitivity. This is therefore the best solution although very high resistances may make the output signal noisier. The system adopted for the electron-beam calorimeter results in a power dissipation of $8 \mu\text{W}$ and noise typically of $\pm 10 \mu\text{K}$. Even though this power dissipation is small the size of the thermistor and the nature of its thermal environment may result in a significant self-heating which cannot be ignored if an accurate calibration is required.

The experimental set-up used to determine the extent of this self-heating is that described in section 3 but with a modification. The 1.41 V supply was replaced by a variable supply consisting of seven voltage reference band-gap devices connected in series. This provided a selectable output voltage to the bridge of between 1.28 V and 8.83 V in steps of approximately 1.27 V.

The procedure used was as follows. With the water bath maintained at a constant temperature different supply voltages were applied to the bridge and the out-of-balance voltage measured. The data were corrected for any drift in the ambient temperature during the period of measurement. This procedure was then repeated at six temperatures between 14°C and 29°C . A typical plot of the ratio V_{br}/V_s varying with V_s is shown in Figure 4. Equation 5 shows that if there is no self-heating then one would expect a constant value for V_{br}/V_s . This is not the case here. The data is clearly not linear in V_s and therefore the model used to fit to the data was

$$\frac{V_{br}}{V_s} = p \cdot V_s^2 + q \quad (8)$$

where p and q are constants. This is the simplest model with the necessary zero gradient at $V_s=0$ (there can be no self-heating with zero power input). Rewriting equation 8 gives

$$V_{br} = p \cdot V_s^3 + q \cdot V_s \quad (9)$$

The term qV_s can be thought of as the bridge output extrapolated to zero power and pV_s^3 as the self-heating term (voltage excess), V_{ex} . If the self-heating is small the resulting temperature rise or excess can be calculated thus

$$T_{ex} = \frac{dT}{dV_{br}} \cdot V_{ex} \quad (10)$$

where dT/dV_{br} is the sensitivity of the thermistor obtained from a normal calibration. The appropriate temperature excess is that evaluated at the supply voltage used in the calibration.

The results are shown in Figure 5. There is no obvious dependence on water bath temperature and the mean value over the range $14\text{-}29^\circ\text{C}$ is

$$T_{ex} = (1.8 \pm 0.1) \text{ mK}$$

To confirm the analysis the data were re-analyzed according to Galloway⁸. Reassuringly this gave a mean temperature excess of 1.8 mK. Finally a computer simulation of the situation was also used. This employed an iterative technique to calculate the equilibrium self-heating for a one-dimensional system after power had been switched on. Using standard values for the various parameters (thermal conductivity, *etc*) this gave a value of 1.7 mK, in very close agreement considering the simplicity of the model.

The fact that the temperature excess is small and constant over a wide temperature range means that the self-heating will not introduce a systematic effect into the thermistor calibration, only a constant offset that can be corrected for if desired. As calorimetry involves measuring temperature differences this offset will cancel out. Another possible source of error is the self-heating of the PRT. Measurements in water over the range 0-36 °C gave a constant value of (2.6 ± 0.2) mK.

5.2 THERMISTOR RESPONSE TO ACCUMULATED RADIATION DOSE

Radiation can cause degradation of many materials, e.g. semiconductors and plastics. If a detector is made of such a material then exposure to radiation can alter its response. As stated earlier commercial thermocouples have a limited life as the PVC insulation breaks up. The calorimeter relies on the thermistor calibration remaining unchanged and therefore an investigation was undertaken into the response of the thermistor to accumulated dose. Previous work⁹ on this had reported only very small changes, even at doses up to 4000 kGy, indicating that thermistors were very radiation 'hard'. However it cannot be assumed that all thermistors respond in the same way and therefore it was necessary to test the thermistor type used in the primary standard.

In addition to the glass bead thermistor a second type was tested. This was of a polyimide and epoxy construction, originally designed for the medical industry. There was quite a difference in physical size - the glass beads were approximately 5 mm long and 1.5 mm in diameter while the medical thermistors were much smaller, 3 mm long and 0.5 mm in diameter. They also had different nominal resistances, 47 k Ω and 22 k Ω respectively which meant that a second bridge with $R_{bal}=25$ k Ω had to be constructed.

The basic experimental procedure was simple. Twelve thermistors of each type were calibrated. These were then irradiated to different dose levels in a 10 MeV electron-beam. The thermistors were then recalibrated and the pre- and post-irradiation calibrations compared.

Calibration conditions

The basic apparatus as described in section 3 was used. A thermistor of each type was calibrated at the same time, the bridge outputs being read by two 5½-digit DMMs. An interval of 1 K between points was used over the range 17-32 °C; the results showed that

the reduced resolution of the DMMs had little effect on precision with the fits giving a typical rms deviation of 0.7 mK (using a cubic model).

Irradiation conditions

The NPL linear accelerator (linac) was used to provide a 10 MeV electron beam at a dose rate of approximately 6.5 kGy min^{-1} . The electron-beam calorimeter was used to calibrate a charge monitoring system¹⁰ which then monitored the linac output during the irradiations which were carried out over two days. Four of the twenty-four thermistors (two of each type) were kept as controls leaving twenty for the irradiations. Increments of approximately 300 kGy were used up to a maximum of 3000 kGy (dose-to-graphite). The thermistors were positioned in a graphite block at a depth of 1.5 g cm^{-2} and the block was mounted on a water cooled target. The temperature of the block was monitored during the irradiations and the water flow adjusted to keep it below 70°C (well below the maximum operating temperature of either thermistor type).

Recalibration

All twenty-four thermistors were recalibrated as soon as was practicable after irradiation. About half were also recalibrated eight months later to detect if any induced effect decayed with time. The recalibrations were carried out in an identical fashion to the original calibrations.

Analysis

As mentioned earlier the two parameters of interest were the absolute temperature T and the gradient dT/dV_b ; the analysis looked at differences in these for each pair of calibrations. Two methods of analyzing the data were used. Firstly, the pre- and post-irradiation calibrations for each thermistor were compared over their whole temperature range ($17\text{--}32^\circ\text{C}$). Secondly, to counter any systematic effect introduced by the curve fitting program the two calibrations were compared at a fixed temperature, namely 21°C (chosen as being close to balance for the majority of the thermistors). For each parameter T and dT/dV_b , the results for the two methods agreed within their uncertainties and are shown in Figures 6 and 7.

Discussion

The results for the glass bead thermistors were in accordance with previous results⁹ - within the uncertainties no significant effect was seen up to 3000 kGy. This is reassuring as it means that calorimetric procedures do not need to be changed. It is doubtful that any calorimeter in use at the NPL would ever receive such a large dose in its lifetime. The eight month calibrations showed no change with time which further confirms the radiation hardness of these thermistors.

The medical thermistors did not fare quite so well showing an indicated temperature that increased linearly with dose. This equates to an increase in R_0 of $(0.16 \pm 0.04)\%$ per MGy - a measurable change but small. There was no equivalent effect on the thermistor sensitivity. As mentioned earlier calorimetry is not greatly affected by changes in absolute temperature as long as the sensitivity remains the same. This means that these thermistors could still be employed in the calorimeter, especially in low dose applications such as radiotherapy. Figure 8 shows how the medical thermistors had changed after eight months with a reduction to $(0.08 \pm 0.04)\%$ per MGy and again no change in sensitivity. This decay does appear to be linear with dose but there is large scatter on the points. The main point to note is that the effect is small and poses little of an obstacle to calorimetry.

A new graphite calorimeter is currently being developed at the NPL which will be able to operate at lower beam energies than the present calorimeter (limited to >8 MeV). This will require a smaller thermistor than the glass bead type. From the study carried out here it should be possible to use the medical thermistors without any problem.

6 CONCLUSIONS

The calibration system that has been developed provides an accurate way of calibrating thermistors for use in the primary standard electron-beam calorimeter. Combining all the uncertainties gives an overall estimated uncertainty on the measurement of temperature rises of $\pm 0.1\%$ (at the 95% confidence level) which is almost entirely due to the uncertainty in the PRT calibration. Investigations have shown that systematic effects are small - the glass bead thermistors presently used in the calorimeter have a constant self-heating of (1.8 ± 0.1) mK in graphite and the effect of accumulated dose on the thermistor response is negligible up to a dose of 3 MGy. A second, smaller type of thermistor, to be used in a new calorimeter, showed no change in sensitivity up to the same dose although a small change in the absolute resistance was seen (approximately 0.1% per MGy).

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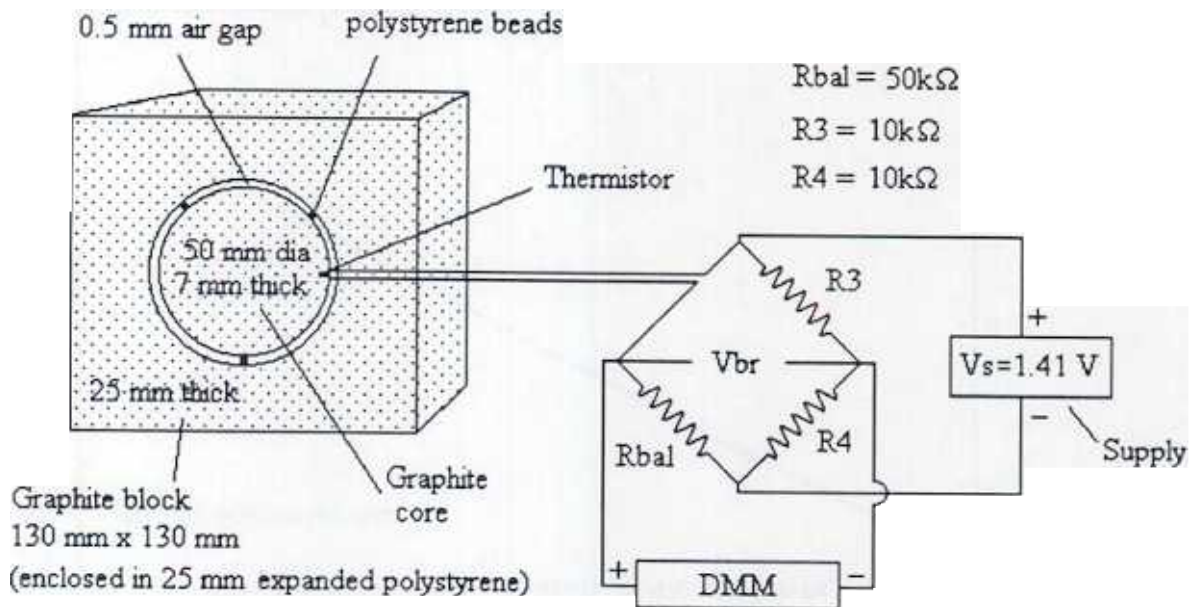


Figure 1 Schematic of the electron-beam graphite calorimeter

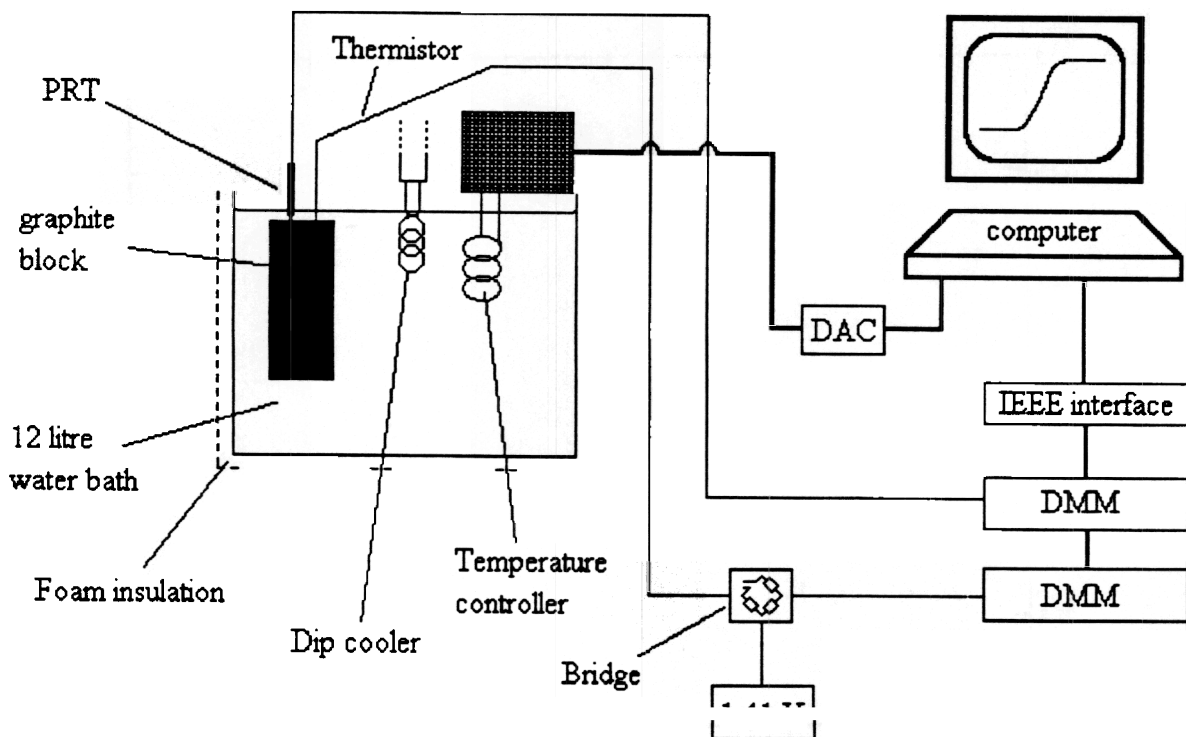


Figure 2 Schematic of the thermistor calibration apparatus

Temperature
(°C)

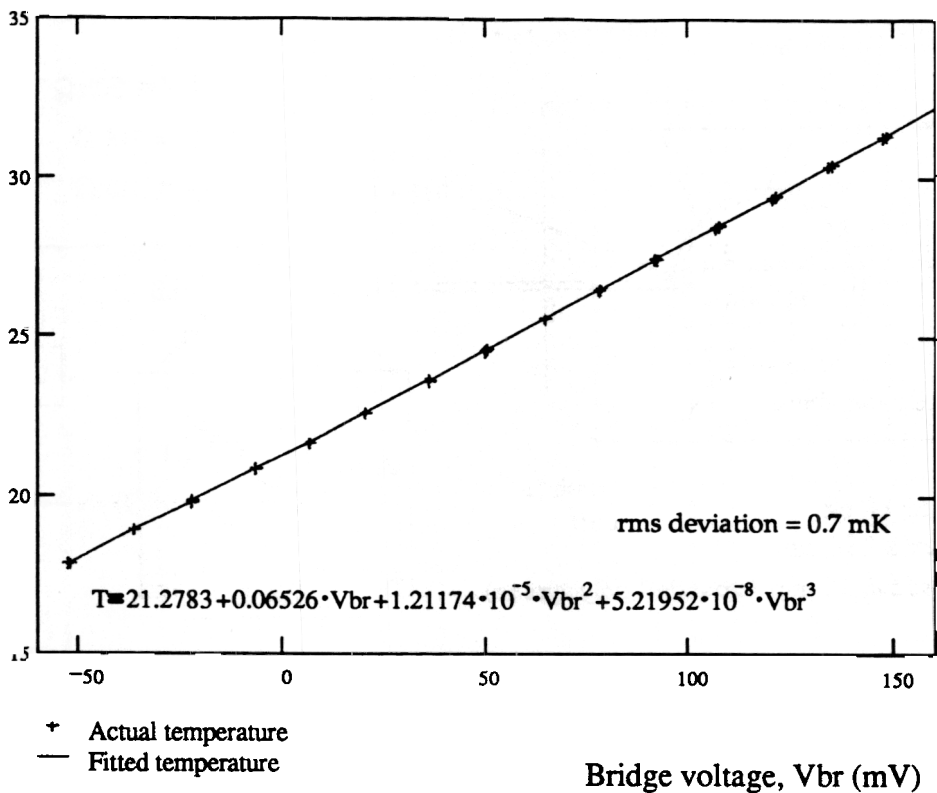


Figure 3 An example of a thermistor calibration

Bridge
response

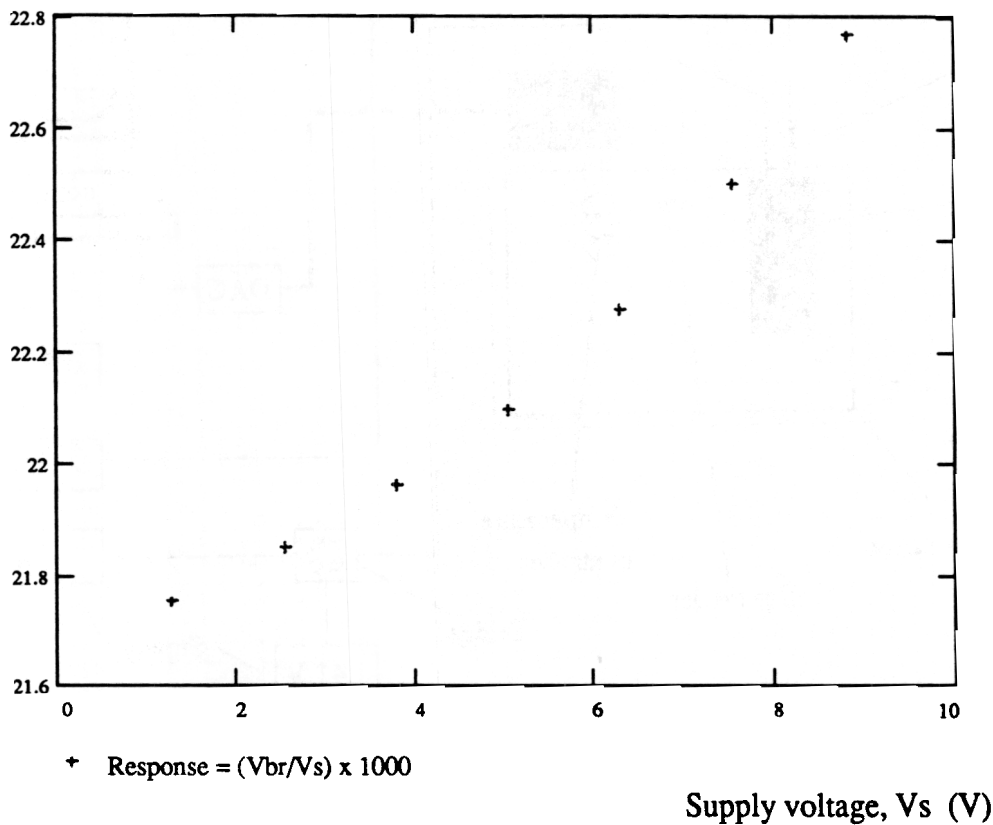


Figure 4 Temperature excess measurement @ 23.0 °C

Temperature
excess
(mK)

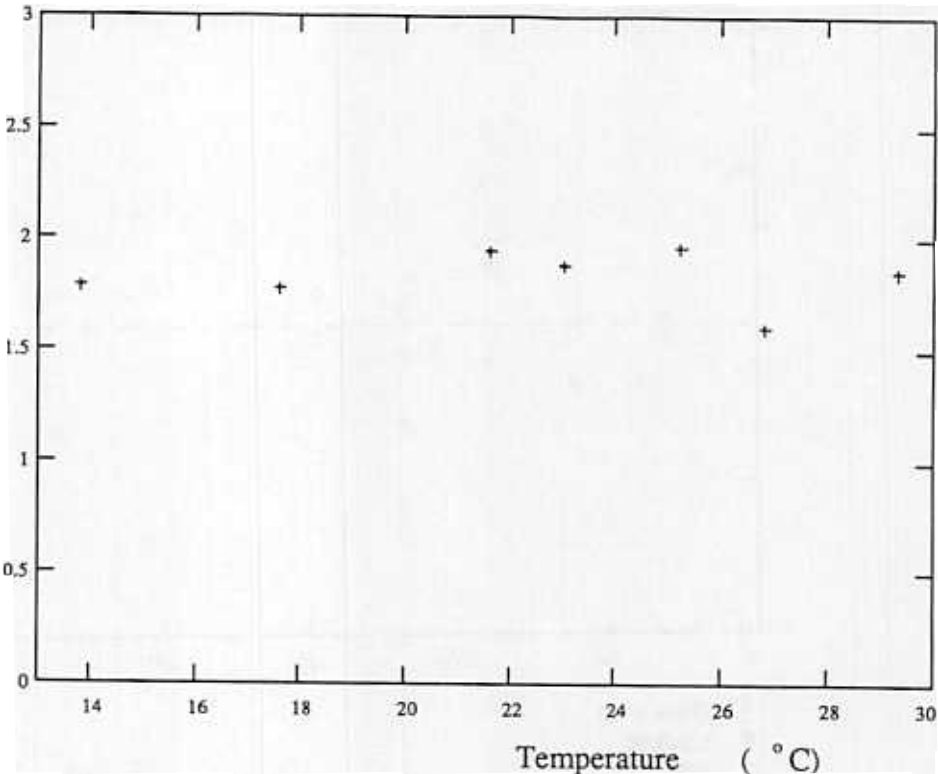


Figure 5 Temperature excess as a function of temperature

Temperature
difference
(before
and after
irradiation)
(mK)

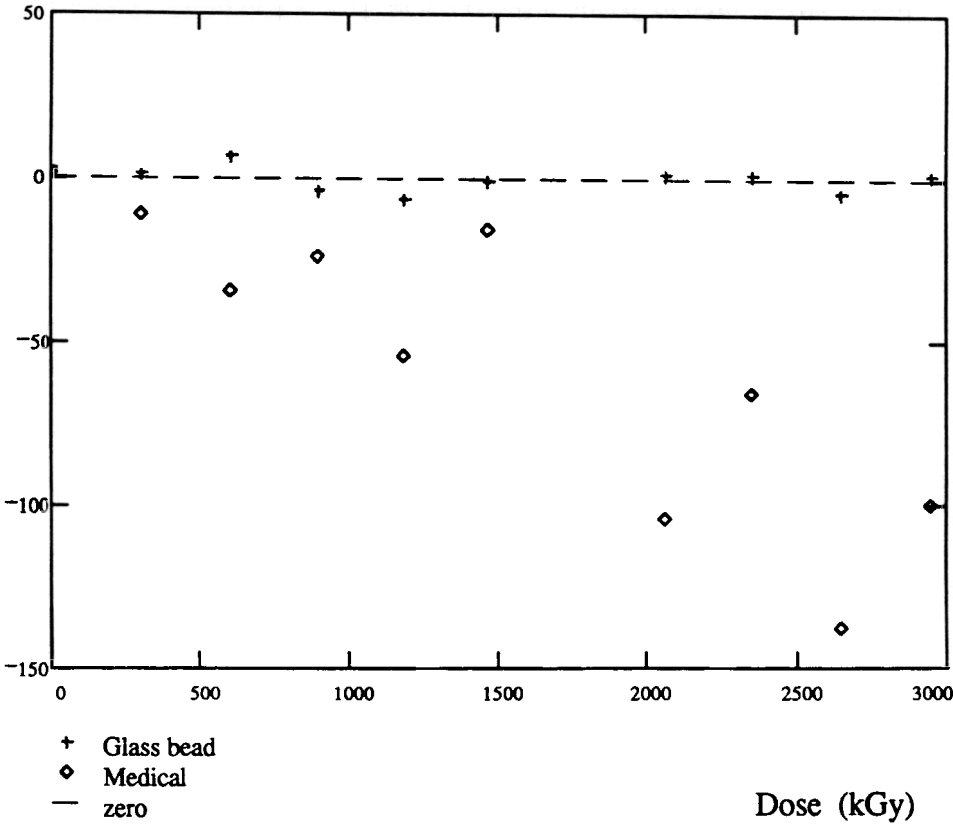


Figure 6 Dose response of the thermistor calibrations

Percentage change in gradient (before and after irradiation)

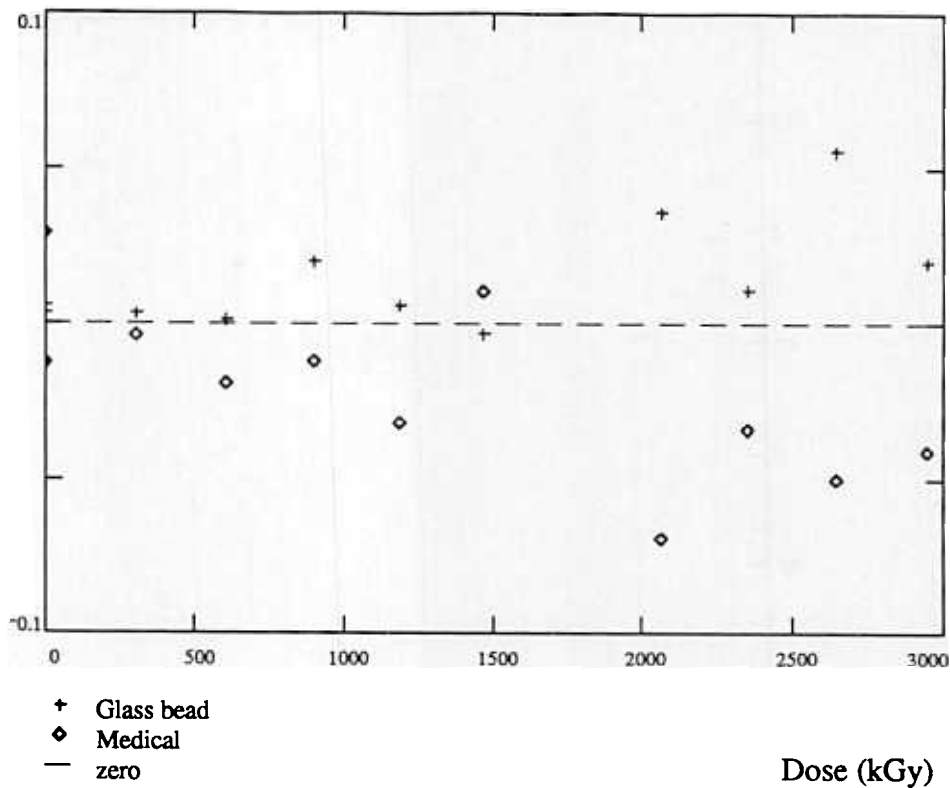


Figure 7 Dose response of the thermistor calibrations

Temperature difference in mK (before and after irradiation)

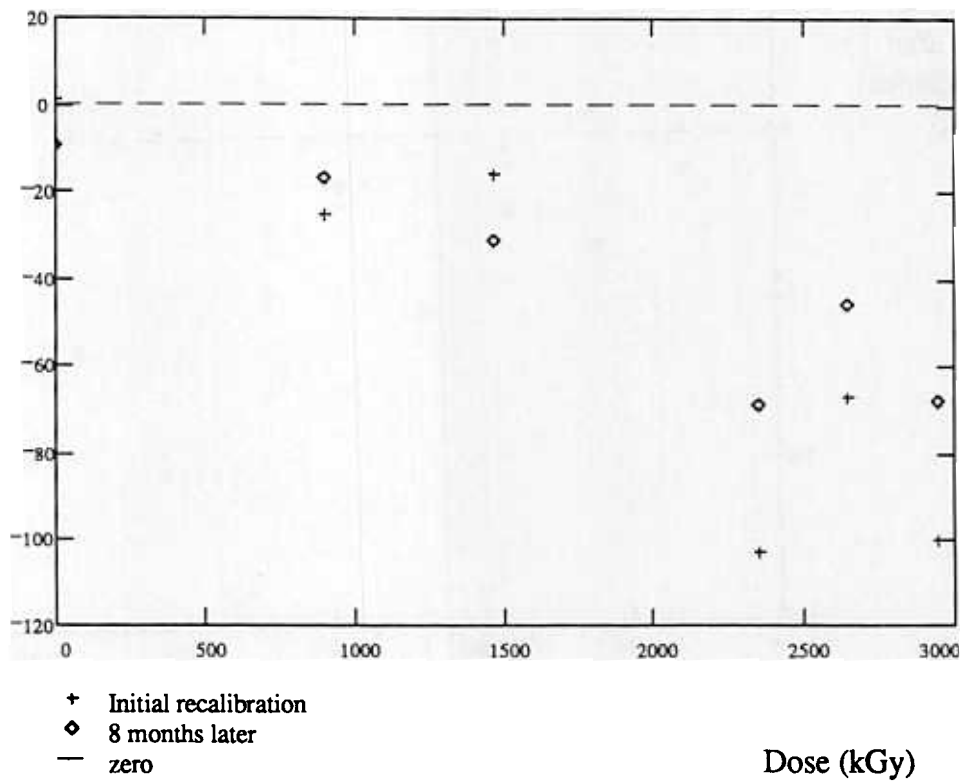


Figure 8 Change with time of medical thermistor calibrations