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PRIMARY AND INTERLABORATORY CALIBRATION OF BETA SOURCES USING QUARTZ AS THERMOLUMINESCENT PHOSPHOR

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1. INTRODUCTION

In thermoluminescence (TL) dating it is necessary to determine the TL sensitivity of the material used for the measurement of the so called natural TL. Since it is generally assumed that the response to gamma rays is the same as to beta rays, most laboratories use beta sources for sample irradiations, because they are easier to handle in routine work. However, beta sources introduce a disadvantage due to the difficulty of their exact primary calibration as compared to gamma sources. In previous work (Zimmerman 1971; Wintle and Murray 1977) the TL of natural CaF_2 was used to compare known gamma doses with the dose delivered by a beta source. This paper describes a similar approach but with chemically pure quartz as TL phosphor to circumvent the uncertainty introduced by the calculation of the beta doserate delivered to ceramic material from that delivered to natural fluorite.

2. EXPERIMENTAL

Commercial coarse-grained quartz (Merck, Quarz feinkörnig, gewaschen und geglüht, p.A.,0.1 - 0.5 mm) was crushed and sieved. The grain size fraction of 125 - 200 µm was etched with 40% hydrofluoric acid as in the sample preparation for the quartz inclusion technique (Fleming 197o), so that the resulting quartz sample had a grain size range of 7o - 15o µm. After annealing at 700°C for three hours, samples were prepared for the gamma irradiations by packing about 1oo mg quartz grains in small polyethylene bags so that the sample thickness was less than 1 mm. Alternatively 2 mg samples were placed in small polyethylene containers.

^{6o}Co gamma irradiations ($\bar{E}$ = 1.25 MeV) were accomplished at the Institut für Strahlenschutz of the GSF at Neuherberg/München (Germany) and the Studienbesellschaft für Atomenergie (SGAE) at Seibersdorf (Austria). In both cases the exposure was known to an error of less than ± 1% at one standard deviation from calibration with a graphite cavity absolute chamber.
To achieve secondary electron equilibrium conditions the gamma irradiations were carried out behind o.5 cm perspex absorbers at a source to sample distance of 1oo cm. The small build up effect due to the perspex-quartz interface is estimated to be less than o.5%. In addition this is partly compensated by the attenuation in the quartz grains.

The dose delivered to the sample was calculated using the ratio of the mass energy absorption coefficients $\frac{\mu_{en}}{\rho}$ quartz and air, knowing that 1 röntgen is equivalent to 0.869 rad in air, at that energy so that

$$D = f \cdot X \qquad (1)$$

$$f = 0.869 \times \frac{\left(\frac{\mu_{en}}{\rho}\right)_{quartz}}{\left(\frac{\mu_{en}}{\rho}\right)_{air}} \qquad (2)$$

Where D is the absorbed dose delivered to quartz and X the exposure. The value of mass energy absorption coefficient for quartz using the values for silicon and oxygen (Attix and Roesch, 1968) was calculated to be $0.0265_5\ cm^2/g$ at 1.25 MeV. The value of $\frac{\mu_{en}}{\rho}$ for air is $0.0266\ cm^2/g$ which yields f=0.867 at 1.25 MeV.

The beta sources used were identical 40 mCi disc sources of ^{90}Sr - ^{90}Y, type SIP 13, from the Radiochemical Centre, Amersham. The active area is 12 mm in diameter and 0.02 mm thick. The sources are screened with a 0.1 mm silver window, which absorbs the low energy beta radiation of ^{90}Sr. The maximum energy for ^{90}Y is 2.26 MeV.

The beta irradiations were performed with the quartz grains on the nichrome heating plate at a sample to source distance of 16.0 mm. Quartz is known to exhibit a predose effect not only at the 100° peak but also in the high temperature region (Aitken and Fleming, 1972) so that direct matching of the TL signals after gamma and beta irradiations could lead to a wrong estimation of the dose-rate delivered by the beta source. For this reason all measurements were normalized to a second standard beta irradiation. Due to the predose effect the dose-rate of the beta source is overestimated, if the beta dose is delivered after the gamma dose. This is, however, the most convenient way, because the sample should not be removed from the heating plate after the first TL measurement. This preliminary value is used in the second step of the calibration, where the normalized TL intensities after gamma irradiations are compared with the normalized signals after the beta irradiations on another quartz aliquot. The beta doses are chosen so that the ratio of gamma to beta dose is constant and nearly unity. Assuming equal predose sensitivity for beta and gamma rays, a graph of normalized TL intensities after beta and gamma doses should give a straight line ideally passing through zero. The slope of the graph is used to calculate the exact dose rate of the beta source in the following way:

$$TL_\beta = DL_\beta \cdot t_\beta \cdot E_\beta \qquad (3)$$

$$TL_\gamma = X_\gamma \cdot f_\gamma \cdot E_\gamma \qquad (4)$$

TL_β, TL_γ are the TL signals after beta and gamma irradiations and E_β, E_γ are the TL efficiencies for beta and gamma radiations. DL_β is the dose rate of the beta source and t_β the beta irradiation time in minutes. t_β is the beta irradiation time when $TL_\beta = TL_\gamma$ and assuming equal TL efficiency for beta and gamma radiation

$$DL_\beta \cdot t_{\beta\ equ} = X \cdot f \qquad (5)$$

so that using (1)

$$t_{\beta\ equ} = \frac{1}{DL_\beta} \cdot D \qquad (6)$$

the inverse slope of the calibration graph (Fig. 1) yields the dose rate of the beta source in rad/min.
TL intensities were measured using the 375 °C peak maximum of the quartz glow curve. This peak shifted slightly to lower temperatures with increasing dose, but this had apparently no effect on the results.

3. RESULTS OF THE PRIMARY CALIBRATION

Fig. 1 shows the calibration graphs of the two beta sources now used for TL dating programmes in Heidelberg (HD) and Vienna (W). The error of the individual measurement is ± 3% at one standard deviation and that of the slope ± 2% also at one standard deviation and calculated after the method of Kerrich (Sachs, 1974). For source HD a dose rate of 101 ± 2 rad/min was determined and for source W,125 ± 3 rad/min. These values are for quartz grains with a grain size of 70 - 150 μm deposited on 0.5 mm nichrome and irradiated from a distance of 16.0 mm in the same irradiation geometry.

4. INTERCOMPARISON OF THE BETA SOURCES

In the same manner as in the primary calibration the two beta sources were compared with each other. The primary calibrations imply a ratio of 0.81 ± 0.02 for source HD relative to source W. The experimentally determined ratio was 0.79 ± 0.03 in good agreement with the primary calibrations.

5. DISCUSSION

It was mentioned by Dr.M.J.Aitken (Aitken 1978) that the glow curve of quartz is highly dependent on the annealing procedure. In addition other phosphors such as natural CaF_2 have much higher sensitivies as compared to quartz. Therefore quartz was not considered a good phosphor for beta source calibrations.

In a preliminary check the quartz used in this study was annealed in different ways. So far only an increasing sensitivity was observed with increasing annealing temperature and time. However, the shape of the glow curve did not change as to be seen in fig.2. Since only normalized TL intensities were used in our calibration procedure, a different sensitivity should not effect the results. In addition, the quartz grains for each calibration were annealed under the same conditions.

6. CONCLUSION

We used quartz grains as phosphor for the primary calibration of two 40 mCi ^{90}Sr - ^{90}Y beta sources against two different ^{60}Co gamma irradiation facilities. These primary calibrations were checked by a direct comparison of the beta sources. The values agreed within the experimental error limits. The dose rate values obtained by calibration with quartz can directly be used for age determinations of ceramics. This excludes any error introduced by the dose rate conversion from other phosphors to ceramic material. In addition quartz as we used it is easily obtainable in large quantities at a very low price. These advantages may outweigh the lower sensitivity, especially in cases where more material is needed than for just one calibration, such as interlaboratory comparisons.

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FIGURE CAPTIONS

Fig. 1: Calibration graphs of two ^{9o}Sr - ^{9o}Y beta sources used in Heidelberg (HD) and Vienna (W) from comparison with ^{60}Co gamma irradiations. The ratio of gamma and beta doses was chosen to be approximately unity to minimize predose effects. All TL signals were normalized to a second standard beta irradiation of the same sample.

Fig. 2: Glow curves of 3 mg samples of quartz grains (Merck, Quarz feinkörnig, p.A.) of the 7o - 15o um grain size fraction, after different annealing procedures:

1 - three hours at 7oo°C

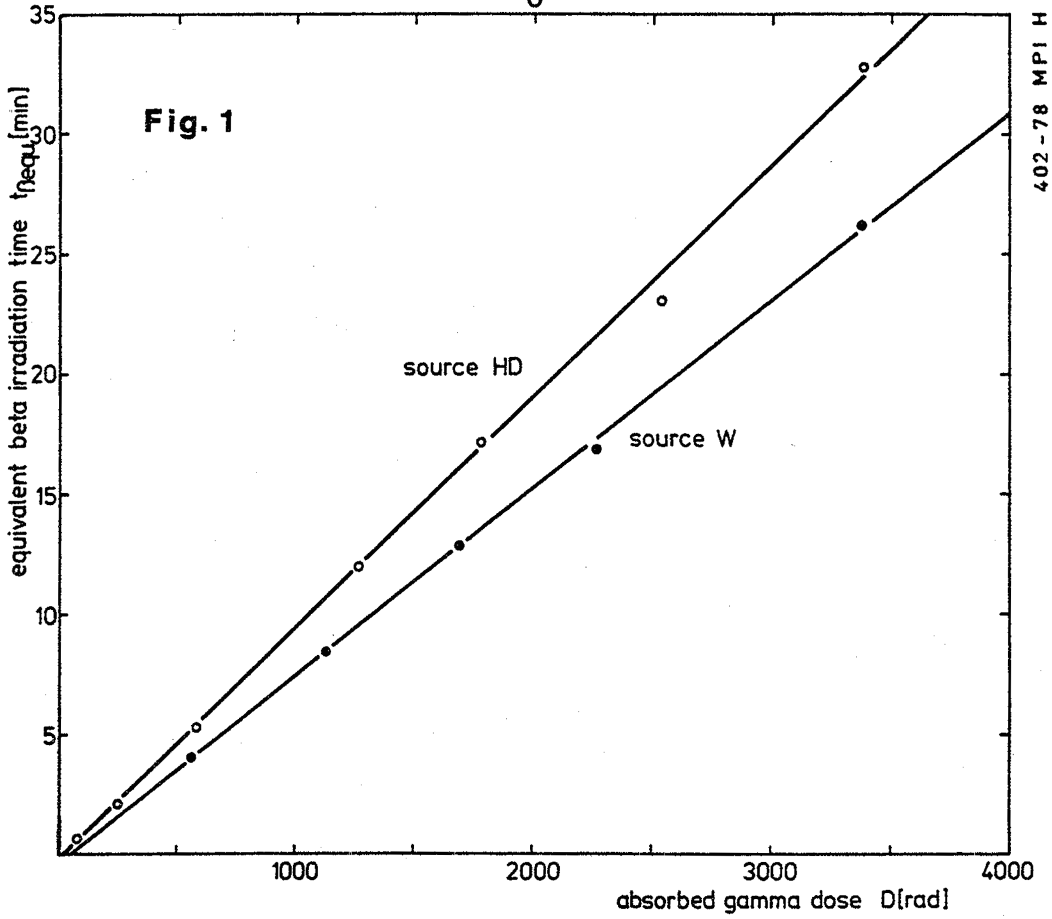
2 - six hours at 7oo°C

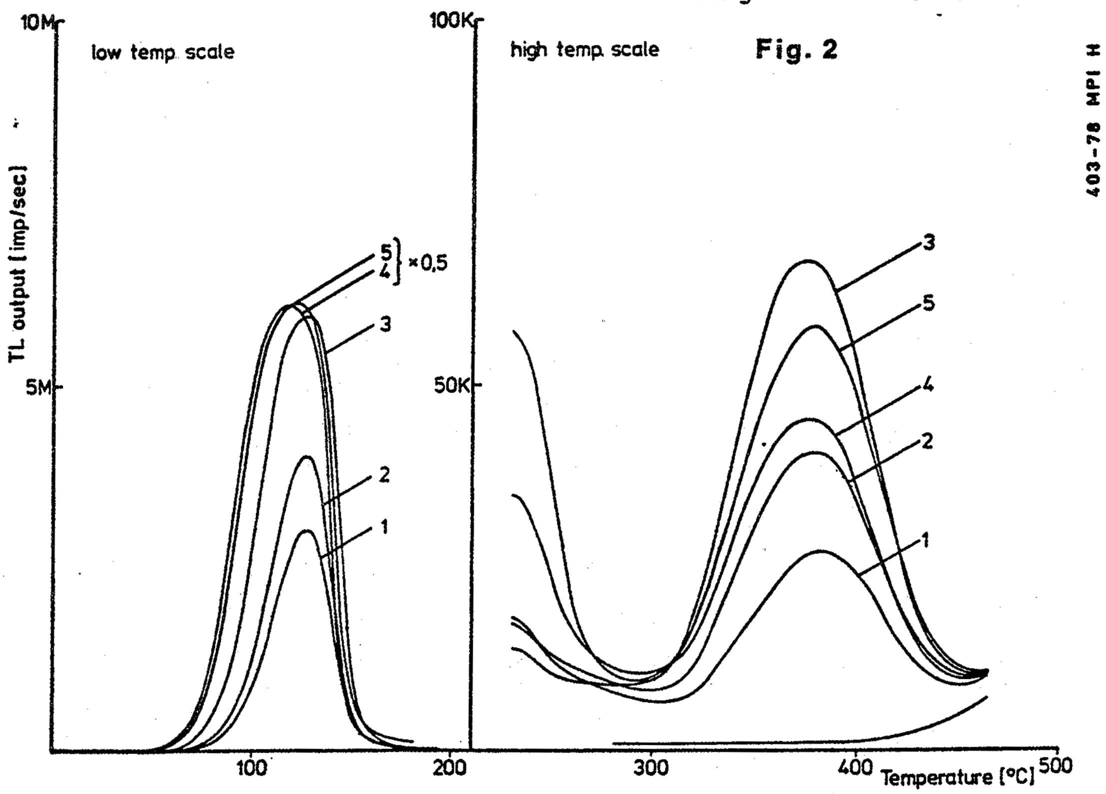
3 - twentyone hours at 7oo°C

4 - three hours at 8oo°C

5 - three hours at 9oo°C

All annealings were carried out in air.





Following a suggestion by Dr. M. J. Aitken a limited number of irradiated quartz samples will be available free by May 1979. One set consists of five 200 mg samples, which have been irradiated in a ^{60}Co beam to an absorbed dose of 600 to 4000 rads. A 300 mg sample of unirradiated quartz from the same batch of annealing and etching procedures will be provided.

For samples and additional information apply to Dr. E. Pernicka at the address on page 2.