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Experimental TL techniques for the Inclusion method

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Our TL data-gathering is based on weight-normalized procedures using a modified Littlemore 711 TL system. Laboratory gamma doses are administered separately in a Co-60 facility for the additive dose method. The TL data are collected in a clock-driven multichannel scaler (MCS), which necessitates a strictly linear temperature-time relation. The Littlemore TL system compares a chromel-alumel thermocouple output voltage responding to the temperature of the heating strip, with an electronically developed voltage ramp rising linearly with time. The difference signal drives the heater current source in the feedback loop. However, the chromel-alumel junction voltage output is not linear, and this not only misidentifies the temperature to be associated with a given channel in the clock-driven MCS recording of the glow curve but also produces a systematic error in the temperature interval corresponding to the channel width. This latter error is the more serious of the two and has been measured to be as large as 14% in the range 100°C to 500°C.

The necessary linearity is achieved by using a Fluke thermocouple reader with automatic ice point correction and a microprocessor circuit to remove the chromel-alumel thermocouple nonlinearity. The temperature read out is accurate to ± 0.1°C. Attempts to use the analog readout of the Fluke directly as the input to the Littlemore temperature feedback loop failed because of the long time constant involved in the sampling time of 3 msec in the Fluke. this problem was overcome by using two identical thermocouples welded to the same spot on the heating strip. One fed the Fluke and the other was used as a fast responding signal capacity coupled to an adding circuit is the input to the Littlemore temperature feedback loop. A capacitor wad adjusted to give critical damping. The resulting slope, dT/dt (temperature/time), was constant to within the limits of measurement i.e. approximately 1 or 2%.

The MCS has a relatively long deadtime (τ ~6 μs). To avoid excessive correction for high counting rates, the PM pulses were fed, after appropriate discrimination, to very fast stacked decade prescalers (τ ~30 ns). The effect of the prescalers is not only to reduce the effective counting rate seen by the MCS but also to de-randomize the pulse sequence so that pulse overlap is minimized. Thus the only deadtime correction we have applied is that for the input to the fast prescaler.

At the end of a run the contents of the MCS, which have had the oven glow subtracted, are dumped to an Apple IIe Computer. the deadtime corrections are made, weight normalization performed and the data stored on disk for subsequent processing. The temperature-channel relation is also computed and stored with the data.

Aliquots of the sample of about 1 mg in weight are contained in a small bowl set in a hemispherical depression in the Mo heating strip. To minimize thermal gradients the bowl is made of copper, but the surface must be stabilized (Templer, 1986). Oxidizing the surface by boiling 2 hours in H_20_2 produced an adherent black surface of low reflectivity, minimizing the effect of changes in the surface properties. This worked well with quartz but proved to be unstable with calcite. Electroplating first nickel and then gold on the copper produced a very stable surface that has worked with calcite as well as with quartz, and produces no spurious TL.

The measured temperature is that indicated by chromel-alumel thermocouples spot-welded to the underside of the hemispherical well in the heating strip. With the aliquot in the copper bowl nestled in the well, there is thermal resistance between the material of the strip and the bowl and between the material of the strip and the bowl and the grains of TL material. The desired temperature within these grains can lag seriously behind the recorded temperature of the thermocouple. Elementary calculations indicate that the effect of this lag is to shift the glow response by a constant temperature.

The presence of this lag can be reduced by increasing the thermal conductance of the contact. Wintle and Dijkmans (1985) suggest the use of thermal contact substances. However, we have been concerned with the possible condensation of volatile substances on the photomultiplier tube window and have avoided this expedient. The use of helium as an atmosphere and the use of the lowest possible heating rate help minimize the thermal lag. We use about 1°C/s, somewhat lower than customary in the TL dating field.

There are actually two thermal barriers of interest in the system, between the heating strip and the copper bowl and between the bowl and the grains of the specimen. We cannot eliminate the latter, but because only a simple linear shift in temperature is involved we can reduce or eliminate the former by calibration. Some characteristic point on the glow curve is chosen, such as the point on either the low or the high temperature side at which the intensity is some fixed fraction of the peak intensity (we usually use half). Each experimental run is corrected in the computer by sliding the data along the temperature axis so that this characteristic point for the experimental run coincides with that for the temperature calibration run. Shifts ranging up to 25°C are required. Figure 1 illustrates the technique, showing two runs with TLD 400, one in the copper bowl and one with the dosimeter powder directly in the well in the heating strip. The data are normalized to the same area. Fig. la. shows the data as taken, fig. 1b the same data after a downward shift of 22°C for that taken with the copper bowl. The shifts we observe are similar to shifts reported by Wintle and Dijkmans (1985).

With the normalized glow data and the corresponding shifted temperatures stored in the computer repeat runs can be averaged channel by channel to produce mean glow curves with standard deviations at each channel. Manipulations such as calculating plateau's are then readily performed.

In the additive dose method of obtaining the Estimated Dose it is necessary to extrapolate the TL data to the dose axis. Provided the glow growth curve is linear the extrapolation is relatively simple using some form of linear regression. However, non-linearity is often present. Mejdahl (1985) has shown that in a number of cases a "saturating exponential" involving the added dose x fits the data quite well:

$$y = y_\infty \{ 1 - \exp [- \beta(x + x_o)] \}$$

A generalized non-linear least squares program can be used to produce a best-fit of eqn. 1 to the data. We have found a simpler technique that produces fits almost identical to the generalized result. By rearranging eqn. 1 and taking natural logarithms of both sides we produce

$$\ln (y_\infty - y) = A - \beta . x$$

with $A = \ln y_\infty - \beta . x_o$

A simple one-parameter least squares program then searches for the value of y_∞ producing the minimum variance of the experimental data about the calculated line, obtaining values for β and x_o for each value of y_∞ examined by the linear regression implied by eqn. 2. Invariably the generalized method produces a lower variance but the difference in parameters is usually small. At the very least, the simplified technique gives a good starting point for the generalized method.

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References

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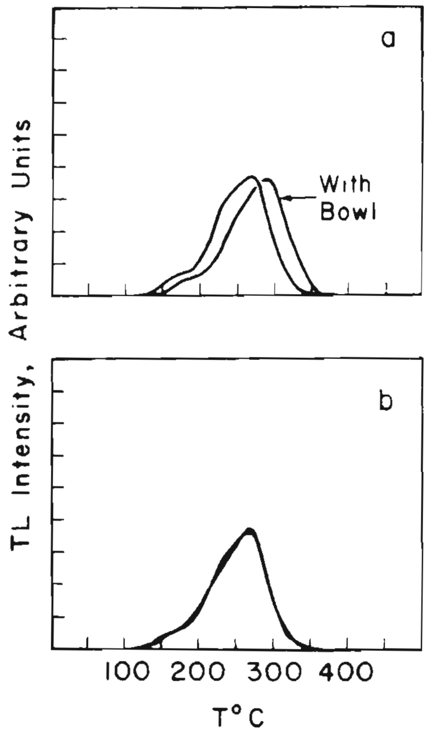

Figure 1

a. Glow curves of TLD 400, with sample in the copper bowl, and without the bowl (sample directly in the well of the heating strip).

b. Glow curves as in 1a, after temperature - shifting.