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Uncertainties involved in the measurement of TL intensities

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Computer simulations (Grün & Rhodes 1991, 1992) have shown that the uncertainty in the estimation of dose, D_E, is critically dependent on the intrinsic uncertainty of the TL intensity measurement, which was also predicted by Berger et al. (1987). The fitting procedure of the dose response curve is also fundamentally dependent on the weight that is assigned to the data points. Usually, two weighting models are applied to the data sets: equal weights and weights proportional to variance (see e.g. Berger and Huntley 1986). If equal weighting is the correct procedure, the measured uncertainty for a sufficient number of repeated measurements should be constant, i.e. independent of the measured TL intensity. In case of weights inversely proportional to variance the relative uncertainty is constant for all values of TL.

We present the glow curves of three data sets of quartz, K-feldspar and Na-feldspar mineral separates from a Danish Cover Sand sample from Eggebaek and integral values of 23 other dose response data sets for each of the mineral separates. The samples have been the subject of an intercomparative dating study (Grün et al., 1989, Kolstrup et al., 1990). Quartz was obtained by heavy liquid separation and etching in conc. HF. The K-feldspar and Na-feldspar samples were separated by heavy liquids using 2.58 s.g. and 2.58-2.62 s.g., respectively. Samples were irradiated with 0, 8.8, 17.6, 44, 88 and 176 Gy. Each irradiation dose was applied to 12 aliquots of the respective mineral separate (and between 4 and 12 aliquots per dose for the integral values). Residual glow curves (6 for each mineral separate) were measured after exposure to light in a Hönle sun light simulator for 160 min which corresponds to about 15 h of sun light exposure. All samples, N+dose and residual levels, were preheated in an oven for 140 h at 140 °C. TL glow curves were recorded with an automated Risø TL reader and a heating rate of 10 °C/s. The glow curves were digitised with a resolution of 50 channels in the temperature range 200 - 500 °C (which is the resolution for the glow curves in all figures). The filters used for recording the glow curves were: quartz: 5-60 + HA3; K-feldspar: UG11; Na-feldspar: 7-59.

Quartz

The left and right hand sides of figure 1 show the glow curves of the natural and irradiated samples and the residual level, respectively. The top figures (1A and 1G) show all recorded glow curves. The respective averaged glow curves are shown below (1B and 1H) with the standard deviation, henceforth s.d., (1C and 1I) and relative error (1D and 1J). The two lower most diagrams show the plots of the standard deviation (1E and 1K) and the relative error (1F and 1L) versus the measured TL intensity, respectively.

Figure 1C and 1E show increasing s.d. values with increasing TL intensity. The irradiated samples show a relatively narrow band of the relative error over most of the temperature range (1D), however, the natural sample shows significantly higher relative uncertainties at lower temperatures. Above about 1/5 of the maximum intensity the relative error remains more or less constant (1F). This implies that at least for this region weights inversely proportional to variance should be used in the fitting procedure. Figure 1E implies that even for the lower range, where a large scattering of the relative error can be observed, the standard deviation is not constant but increases with intensity. It follows that equal weighting is not the correct procedure for the lower intensity range.

Figures 1H to 1L show the same analyses as figures 1B to 1F for the measurements of the residual level. Again, above about 1/5 of the maximum intensity, the relative error remains more or less constant, but increases with

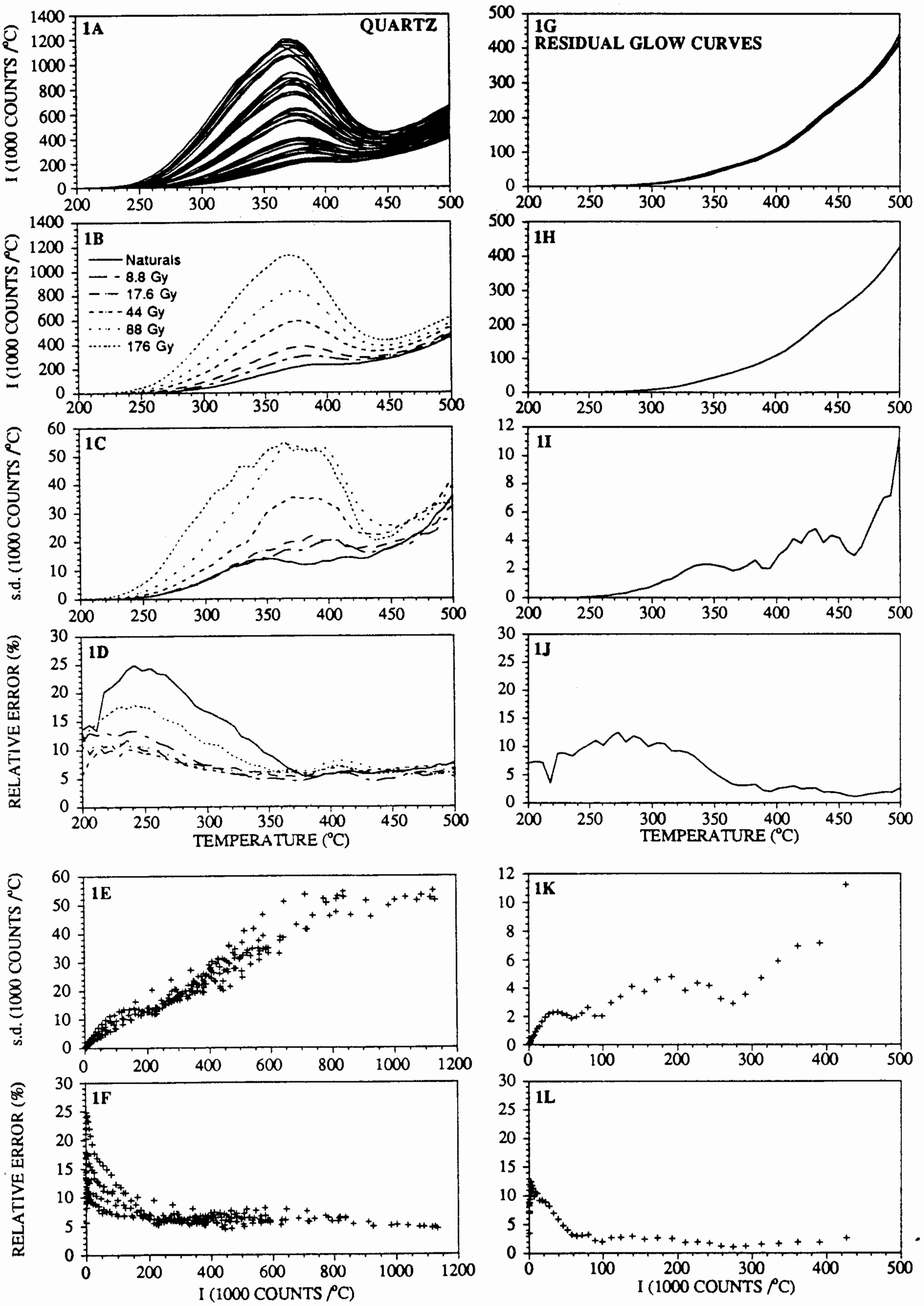
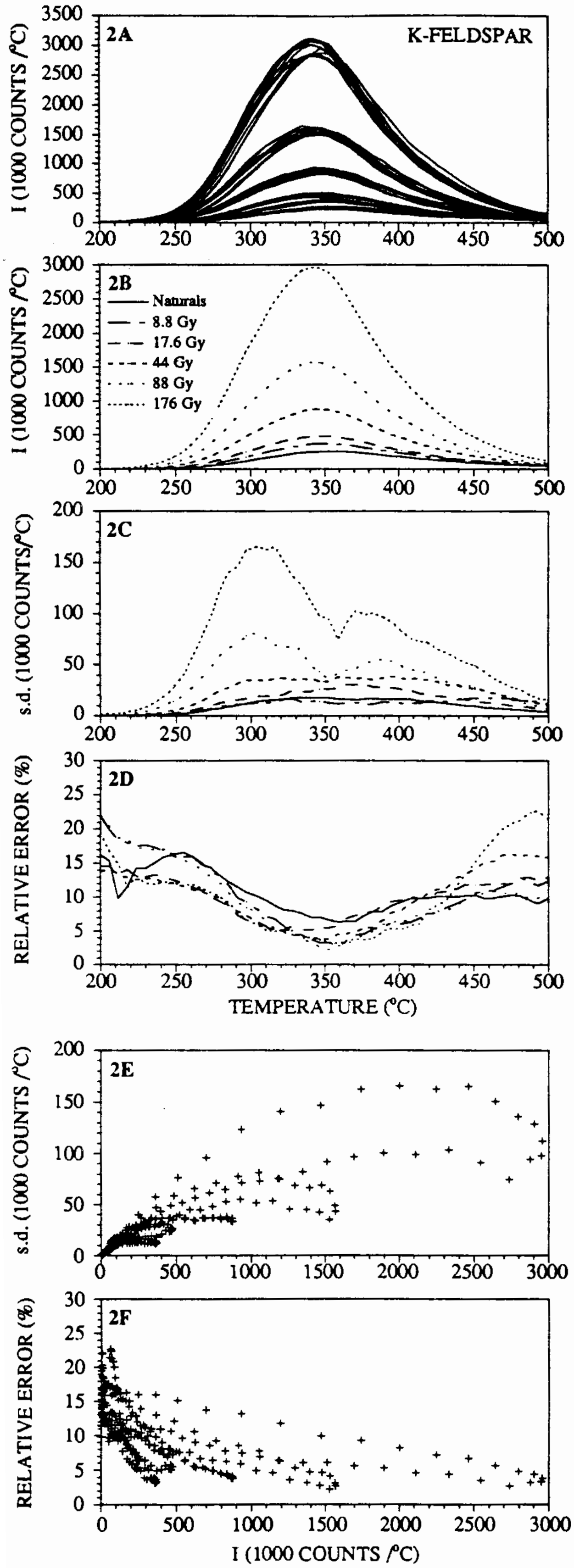
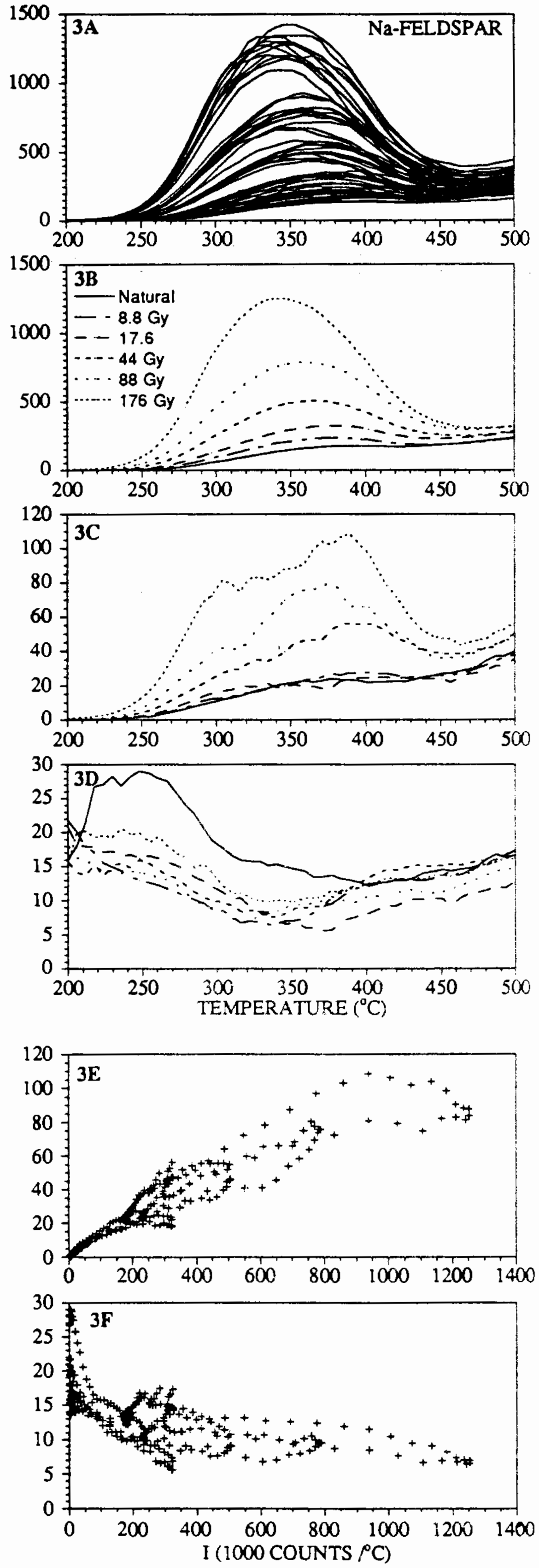


Figure 1.

Irradiated samples (left) and residual levels (right) for a quartz sample from Eggebaek, Denmark.

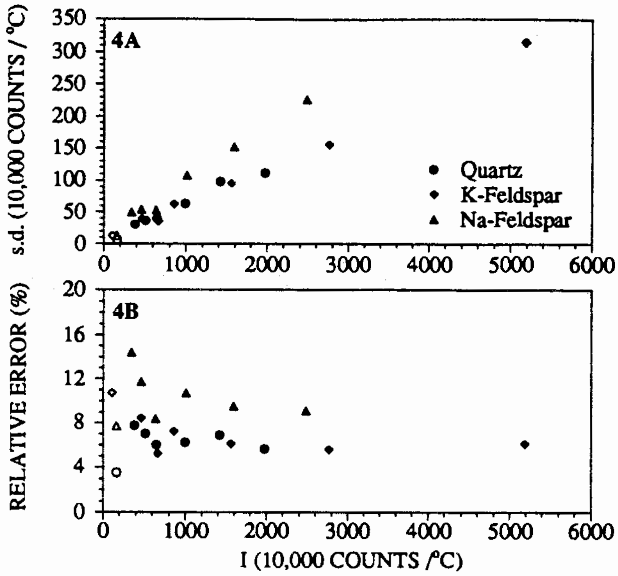
1A, 1G: all glow curves; 1B, 1H: averaged glow curves; 1C, 1I: plot of standard deviation, s.d., versus temperature; 1D, 1J: plot of relative error versus temperature; 1E, 1K: plot of s.d. value versus measured TL intensity; 1F, 1L: plot of relative error versus measured TL intensity.





Figures 2 (left) and 3 (right).
Irradiated samples for a K-feldspar (left) and a Na-feldspar (right) separate from Eggebaek, Denmark.
A: all glow curves; B: averaged glow curves; C: plot of standard deviation, s.d., vs temperature; D: plot of relative error vs s.d. ; E: plot of s.d. value vs TL intensity; F: plot of relative error vs measured TL intensity.

Figure 4.
Plot of standard deviation (4A) and relative error (4B) of the integral 239 to 461 °C versus intensity of the glow curves shown in figures 1 to 3. Full symbols: natural and irradiated samples; open symbols: residual levels.



lower intensities. It is interesting to note that the measurement of the residual levels, which are much smaller than the levels of the irradiated samples, are associated with much smaller errors (2% compared to about 6%). This implies that for this sample the distribution of trapped electrons in non-light sensitive traps is more even than the distribution of trapped electrons that are light sensitive.

The integral values of the averaged glow curves for the range 261 to 439 °C are shown in figure 4. The relative errors of the irradiated glow curves show no particular trend and are in the range of about 5.5 to 7%, the natural glow-curve has a somewhat higher relative uncertainty of 8%.The residual level has an uncertainty of 3.5%.

Figures 5A to 5D show the analysis of the integral range of 261 to 439 °C for all 23 dose response data sets. It can be seen that there is a clear trend of increasing average intensity with increasing dose (5A) and increasing s.d. values with intensity (5C). There is no relationship, however, between the relative error and dose or measured TL intensity (5B and 5D).

K-feldspar

The display of figure 2 shows the analyses for the K-feldspar sample.The results differ to some extent from the ones for the quartz sample. There is not a trend towards a constant relative error (figure 2F) or constant absolute error (2E). The largest standard deviations are actually observed at the steepest slopes of the TL peak and coincide with the maxima of the first derivative of the glow curve (compare figure 2C with 2B; see also Grün & Packman 1992). This effect may be attributed to small temperature shifts (temperature jitter). However, below 440 °C, the relative error at a particular temperature is more or less constant (2D), which means that for the dose versus temperature plot fitting with weights inversely proportional to variance seems appropriate. The natural glow curve is associated with the largest relative errors. The residual levels show a similar behaviour as the irradiated samples and do neither display a constant s.d. value nor a constant relative error versus measured TL intensity.

The integral values for the range 261 to 439 °C are shown in figure 4. The relative errors show no trend and are generally in the range of 5 to 8.5 % which is very much the same range as for the quartz sample. The residual levels have an s.d. value of 10.7%, which seems rather large.

Figures 5E to 5H show the analysis of the integral range 261 to 439 °C of 23 dose response data sets. The average relative uncertainty of the natural sample is marginally larger than for the irradiated samples (5F), however, there is basically no correlation between relative error and dose or measured TL intensity, respectively (5F and 5H).

Na-feldspar:

The display of figure 3 shows the error analyses of the Na-feldspar sample. The irradiated samples show basically the same results as for the K-feldspar sample. The standard deviations of the averaged glow curves for one radiation dose step are largest at the steepest slopes

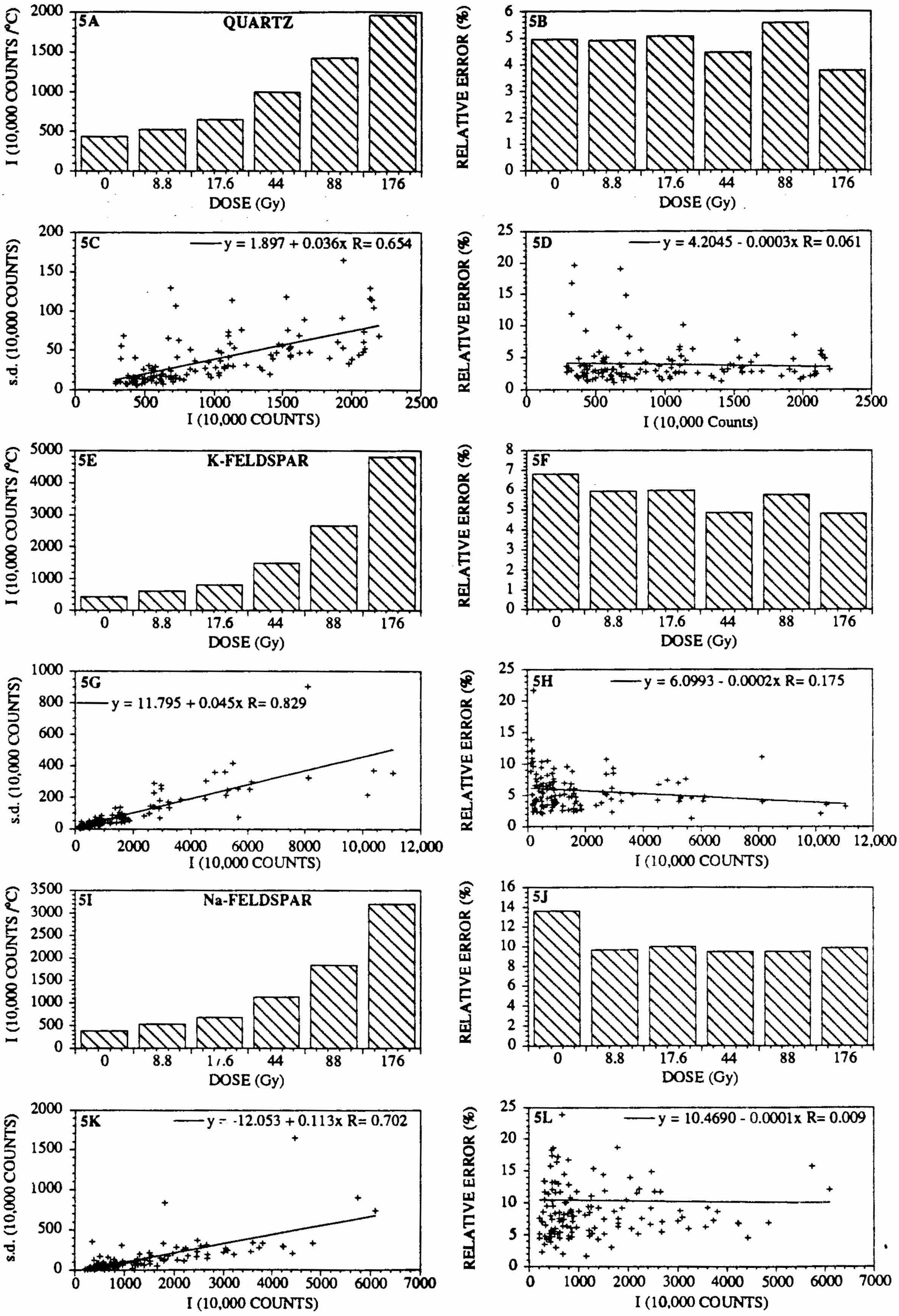


Figure 5.
5A, 5E, 5I: Average integral (239 to 461 °C) intensities for 23 data sets. 5B, 5F, 5J: Average relative errors at each dose point. 5C, 5G, 5K: Plot of s.d. value versus measured TL intensity. 5D, 5H, 5L: Plot of relative error versus intensity.

of the TL peak (compare figures 3C and 3B). The relative error of the irradiated samples is not constant versus the TL intensity, however, the values are within a relative narrow band over the temperature. The natural sample shows a much larger relative error in the temperature range below 400 °C than the irradiated sample. The residual level shows a rather constant relative error over most of the temperature and intensity ranges.

Integral values for the range 261 to 439 °C are shown in figure 4. The relative errors of the irradiated samples show no trend and are generally in the range of 7.5 to 12%, whereas the natural sample has an uncertainty of about 14.5%. The residual level has a s.d. of 7.7%.

Figures 5I to 5L show the analysis of the integral range 261 to 439°C for 23 dose response data sets. The irradiated samples show the same trends as the quartz and K-feldspar samples. The relative error of the natural samples seems considerably higher than for the irradiated ones (5J). However, for the irradiated samples there is no relationship between relative error and dose or measured TL intensity, respectively (5K and 5L).

Discussion

The size of the intrinsic uncertainty of a TL measurement has influence on the uncertainty in the D_E estimation (see Berger et al.,1987; Berger and Huntley, 1989; Grün and Rhodes, 1991). As shown above, this value may be intensity dependent and 12 aliquots are apparently not enough to get a better estimate of the intrinsic uncertainty. However, this value need not be known for the correct estimation of the D_E value, provided the dose steps have been selected appropriately (Grün and Rhodes, 1991 & 1992) and it is known whether TL measurements have constant standard deviations or constant relative errors. The form of the uncertainty has influence on the selection of the weighting model used and, hence, on the estimation of the D_E value, because the fitting procedure is very model dependent (Grün & Rhodes, 1992). Although one has only very little influence on the intrinsic error of a TL measurement, the error in the D_E estimation can be critically reduced by measuring repeatedly at each dose step (see Grün & Rhodes, 1992).

The quartz sample shows most clearly constant relative errors versus measured TL intensity (figure 1F). Although the other two mineral separates do not show this general trend, the relative error is in a more or less narrow (but not constant) band over large ranges of the temperatures (figures 2D and 3D). All samples show a clear trend of increasing s.d. values with increasing TL intensities. The biggest absolute s.d. values, however, do not occur at the maximum TL intensity, but in regions where the slope of the TL peaks are the steepest (maxima in the first derivative of the TL glow curves (Grün & Packman 1992); see figures 2C and 3C). The steep slope regions are most affected by small temperature shifts (temperature jitter). All natural samples of the three data sets show larger relative uncertainties than the irradiated samples over most of the temperature range. All three sample sets show increasing s.d. values with increasing measured TL intensities and basically no relationship between relative uncertainty and measured TL intensity. This clearly implies that equal weighting is not the appropriate procedure for TL dose estimations.

The sets show that the relative error is within a more or less narrow band over most of the temperature range which indicates that weight inversely proportional to variance is the correct procedure for the fitting of the dose versus temperature plot. Whether this has any relevance for the correct estimation of the gamma equivalent dose that the sample has received in its geological past is another matter (see Grün & Packman, 1993). The relative error in the TL intensity for the integral irradiated samples of the three sets show also no particular trend (which basically follows from the above observation) and, hence, weighting inversely proportional to variance is appropriate.

The integral values and uncertainties of the 23 other sets reproduce the results of the three selected samples. There is basically no relationship between relative uncertainty and measured TL intensity and a stronger relationship between s.d. value and measured TL intensity. For quartz and K-feldspar, the relative uncertainty of the natural samples is within the range of the irradiated samples, however, the relative uncertainty of the naturals of the Na-feldspar separates seems somewhat larger than the relative uncertainty of the irradiated samples. Again these results imply that weighting inversely proportional to variance is the correct method. It is interesting to note that the average uncertainty of the quartz samples (in the 3-4% range) is smaller than of the K- and Na-feldspar samples with relative uncertainties in the 5 to 6% and 9% range, respectively, although the quartz integrals have less counts.

For the estimation of the intrinsic precision of TL measurements the sample sets ought to contain more than 12 replicate TL measurements at each dose point. However, the results may only be valid for a particular sampling site and have actually little relevance for the correct estimation of the D_E value.

Conclusion

The analyses of the three TL glow curve sets and integral values of 23 different data sets imply that fitting using weights inversely proportional to variance is the appropriate weighting method for determining dose values for these quartz, K-feldspar and Na-feldspar separates.

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