Ancient TL

www.ancienttl.org · ISSN: 2693-0935

McFee, C., 1996. *TL measurements of single grains from selected feldspar samples.* Ancient TL 14(1): 13-20. https://doi.org/10.26034/la.atl.1996.249

TL measurements of single grains from selected feldspar samples

Chris McFee.

Research Laboratory for Archaeology, 6 Keble Road, Oxford. OX1 3QJ1.

(Received 12 January 1996; in final form 19 March 1996)

Abstract
The thermoluminescence (TL) properties of single feldspar grains from both sediments and mineral specimens have been measured using an imaging photon detector (IPD). The TL sensitivity of the grains to a known laboratory dose was found and a range of grain-to-grain sensitivities were observed. Analytical SEM and optical analysis were then used to attempt to explain the grain-to-grain behaviour in terms of the physical or optical properties of each grain. However, the range of TL sensitivities found could not be correlated with any of the physical or optical properties measured.

Introduction

Thermoluminescence (TL) and infrared stimulated luminescence (IRSL) from feldspars are increasingly common as methods of dating in quaternary science and consequently many studies have concentrated on the luminescence behaviour of bulk samples of feldspar grains (i.e., the total luminescence from many thousands of individual grains), for example, Duller (1992).

In contrast, little work has been published in which the TL behaviour of single feldspar grains has been investigated, Southgate (1985) made TL measurements on feldspar grains to find individual EDs, but no final results were presented. Grün et al. (1989) measured the TL from fifty feldspar grains and although the TL varied greatly from grain to grain, the mineralogical properties of these grains, as determined with an SEM, were broadly similar. A recent paper by Lamothe et al. (1994) reported measurements on 120 large (500-1000 m) feldspar grains with the natural IRSL from individual grains being measured. A large variability in IRSL intensities was found but the majority of the grains were not examined further.

In this paper, the second glow TL (the TL after a laboratory beta dose) was measured from single feldspar grains prepared from both sedimentary and specific mineral types. The TL was measured using an imaging photon detector (IPD) which is a position-sensitive photon counting device previously used to measure the single grain TL of both zircons (Smith et al.1991) and quartz grains (McFee and Tite, 1994).

Sample selection and measurement

The following sediments and geological sediments were selected for measurement:

Belcroute (a) : a modern beach sand from Belcroute, Jersey. The sediment is assumed to be completed bleached. (Lab ref : 760a1)

Belcroute (b) : an aeolian dune sand from Belcroute, Jersey. The ED of the sediment is around 220 Gy. (Lab ref: 760c1).

Le Gurp : a beach sand from Le Gurp, Atlantic coast of France. The ED (IRSL) is 238 (+57,-66) Gy (measured by the author). (Lab ref: 787a1).

Blassac : an aeolian sediment from Blassac, France. The ED (TL) is 65 (+6,-14) Gy (measured by the author). (Lab ref : 785a1).

Labradorite : a mineral specimen from Madagascar.

Orthoclase : a mineral specimen from Norway.

Microcline : a mineral specimen (Spooner 1993). (Lab ref : Z5).

The labradorite and orthoclase mineral specimens were purchased from Gregory, Botley and Lloyd Ltd. (London). The chemical composition of each mineral specimen was checked using an X-ray Energy Dispersive Spectrometer (EDS) attached to an SEM and was found to be consistent with the stated mineral type.

The sediments were prepared under subdued red light and were first sieved to obtain the 180-220 μm grain fraction. Grains were then left in dilute HCl for several hours to facilitate the removal of any iron oxide coating on the grains. Belcroute (a) and (b), and Le Gurp were density separated using sodium polytungstate, the grains with densities

1 Present address: Building 042, DRA Farnborough, Hampshire GU14 6TD. UK.

below 2.58gcm^{-3} being selected. The sediment from Blassac was density separated to remove only the heavy minerals (>2.70gcm^{-3}), i.e., this sediment contained both quartz and feldspars. The sediments were then etched for 40 minutes in 10% HF to remove the outer alpha affected layer (Mejdahl, 1985) and were again washed with HCl to remove any fluorides formed.

Two of the mineral samples (labradorite and orthoclase) were supplied as large crystals. These were gently crushed and the 180-220 m grain size selected by sieving. These grains were also left in dilute HCl for several hours to mitigate any possible spurious TL generated as a result of the crushing process (Aitken 1985, p192). The microcline specimen was supplied as 75-125 m size grains and received no further treatment.

Individual grains were picked up on a small needle in dim red light using an image intensifier attached to a binocular microscope, and were placed in a known pattern onto a rhodium-plated copper planchette.

Grains from all the samples were heated to 450 C at 5 /s to measure the natural TL, these results are summarised elsewhere (McFee and Tite, in preparation).

All the samples were then irradiated on-plate with a ^{90}Sr - ^{90}Y beta source. The following doses were given, which were chosen to allow a reasonably large amount of TL to be obtained from each grain without causing a pile-up of counts within the IPD:

Belcroute (a) and (b), Le Gurp, Blassac and orthoclase each received 30 Gy, labradorite received 60 Gy and microcline 120 Gy.

All measurements were made with quartz optics together with a 1mm BG39 filter, the effective wavelength range being 330-600 nm.

	N	min value	Q1	median	average	Q3	max value
Belcroute (a)	253	217	729	1035	2398	1708	41892
Belcroute (b)	264	26	356	659	1717	1291	33261
Le Gurp	241	852	3521	6623	9312	10936	63506
Blassac	107	126	496	1134	3424	3395	38960
Labradorite	317	182	633	850	1061	1276	4677
Orthoclase	324	232	923	1482	2113	2606	15681
Microcline	206	51	94	149	263	287	4663

Table 1

Descriptive statistics for the sediment and mineral samples. N is the number of grains, min value shows the minimum TL measured, Q1 is the value of the first quartile, median shows the median TL value, Q3 shows the value of the third quartile and max value shows the maximum recorded TL value for each sample.

Results and discussions

The chemical composition of grains from the Blassac sample, which consisted of both quartz and feldspar grains, was investigated using the EDS. The results from grains found to be quartz were excluded from this study.

Around 75% of grains from each sample were sufficiently bright to be observable on the IPD. Table 1 shows descriptive statistics for all the samples and figure 1 shows box and whisker plots for the second glow TL of all the samples. Figure 2 shows second glow TL frequency histograms for the grains from the Blassac sediment and the orthoclase sample. Each count frequency bar shows the number of grains in the range of TL intensity from the value shown to the next value above.

The second glow variations within each sample (figure 1) are very similar, irrespective of the type of sample (sediment or mineral) measured. In addition, outlying grains can be seen in each sample (figure 2). These outliers are usually around 10 to 100 times brighter than the median second glow TL.

When the intensity of such grains is considered in terms of the total light sum from all the grains, such outlying grains contribute about 30-40% of the total light sum. For example, the total light sum from the Blassac sediment was 360,000 counts and the contribution from the brightest 5% (6 grains) was 145,000 counts. Thus, the brightest 5% of the Blassac grains contribute 40% of the total light sum.

The effect of the outlying grains can also be clearly seen in table 1, which includes the median and average TL values found for each sample. In all cases, the average value is much larger than the median value, for three of the samples (Belcroute (a), Belcroute (b) and Blassac), the average values are larger than the values found at the third quartile point (Q3).

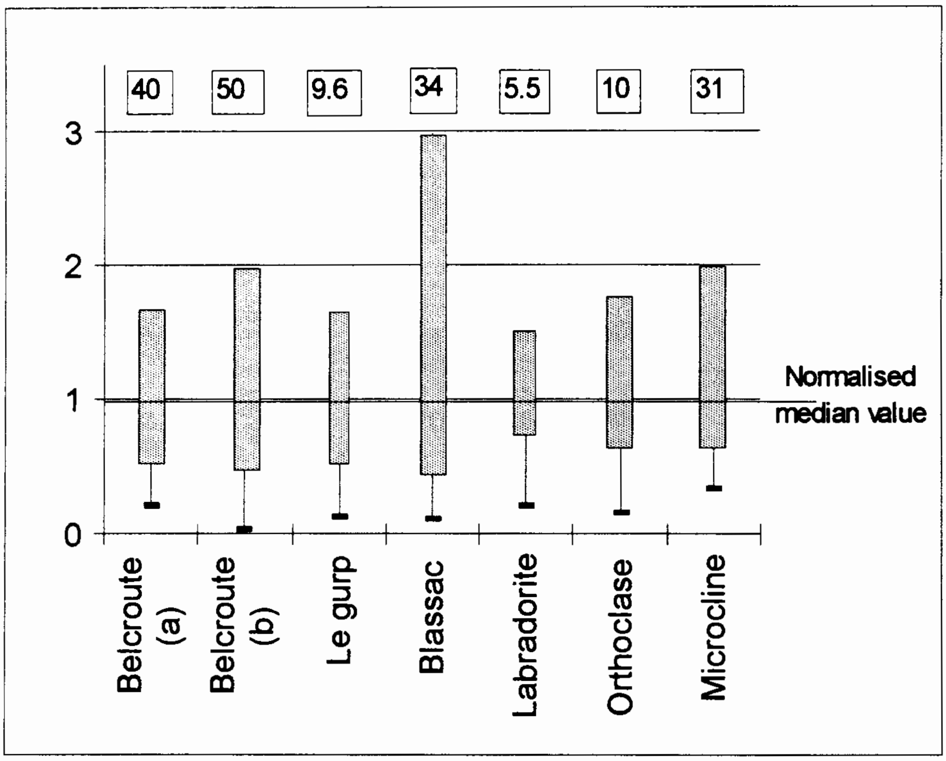


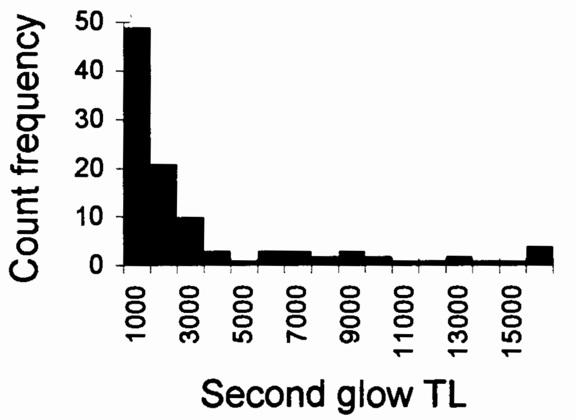
Figure 1

Box-and-whisker plots for the sediment and mineral specimens. The maxiumum TL values are shown only at the top of the graph to allow a reasonable scale. The TL from each sample has been normalised so the median TL from each sample has the value 1.

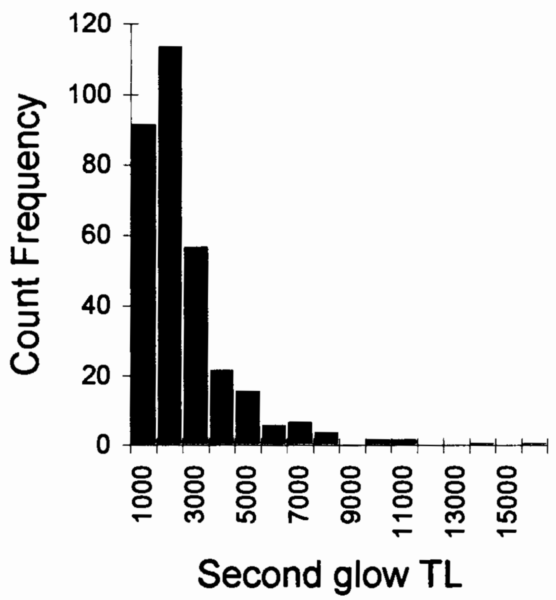
Therefore, the outlying grains do not "dominate" the light sum in the sense that only one or two grains do not contribute almost all of the observed light. However, the contribution of the brightest grains in each sample will be large enough to lead to substantial variations in the bulk TL corresponding to variations in the numbers of these grains present (McFee, 1995).

The TL results found for single feldspar grains are similar to those found previously for single quartz grains by McFee and Tite (1994), with a small number of grains from each sample having a high TL sensitivity. These grains are outliers from the main second glow TL populations with high intrinsic sensitivity to radiation. Such sensitivity will, at a fundamental level be determined by the concentration of impurities and defects (i.e. trapping/luminescence centre concentration within the grains). However, at a macroscopic level, this high intrinsic TL sensitivity may also be associated with physical characteristics of grains such as:

(1) grain size
(2) physical orientation of the grain or grain shape
(3) degree of weathering
(4) grain mineralogy
(5) grain transparency



(a) The second glow TL for the Blassac sediment sample



(b) The second glow TL for the orthoclase sample.

Figure 2
The second glow TL for two samples described in the text. Each count frequency bar shows the number of grains in the range of TL intensity value shown to the next value above

To attempt to identify which of the above explanations were most probable all the grains from which TL had previously been measured were viewed with a low power (x20) binocular microscope in white light and the approximate size of each grain was noted. All the grains measured from the Blassac sediment were also examined at high power (x100 - x500) in both transmitted and reflected white light. Each grain in the Blassac sediment was assigned four indices which provided a measure of angularity, sphericity, transparency (frosty/clear) and the presence or absence of possible inclusions or significant iron oxide staining (it was difficult to differentiate between these two properties using the binocular microscope, even at high powers). The distribution of grain sizes observed was not sufficient to explain either the distribution around the median TL for the majority of the grains nor the presence of outlying grains with very high TL values. For example, the maximum second glow TL for the Belcroute (a) sediment is some forty times larger than the median values which, to be explained solely in terms of grain size, would require a grain of 700 m diameter. Such a grain was not observed. However, it was apparent that, due to the crushing, the labradorite and orthoclase grains exhibited a great range of shapes, many of them not being even approximately spherical and it was difficult to estimate their true volume. For the labradorite and orthoclase grains the maximum observed second glow TL could be explained in terms of a grain volume equivalent to a spherical grain only some 300 m in diameter. Thus, the effect of grain size on the outlying second glow TL values for the labradorite or orthoclase samples could not be ruled out.

A statistical comparison between the sensitivities of grains from the Blassac sediment and their morphological or optical indices found that the range of TL behaviour amongst these grains could not be explained in terms of physical orientation or grain transparency. As the Blassac grains had a very similar appearance to the grains from all other samples, both sediments and mineral types, it is expected that neither physical orientation nor grain transparency could explain the range of second glow TL measurements observed for the other samples.

A random sub-sample of grains from the Belcroute sediments, as well as all the grains from the Blassac sediment were examined using an analytical SEM. The grains were encapsulated into resin blocks, which were then polished. The grains were examined with a Cameca SU30 SEMPROBE in both back-scattered and secondary emission mode, with an accelerating voltage of 15 kV and an operating current of 10nA. The grains were viewed in backscattered mode, the shape of the grain and the effect of weathering being noted. Regions of the grain which consisted of different feldspathic minerals such as perthitic intergrowths could be easily identified as areas of differing contrast on the back-scattered image display of the grains. It should be noted that, because of the small penetration depth of the incident electrons, only a thin surface layer (3-5 m) could be examined.

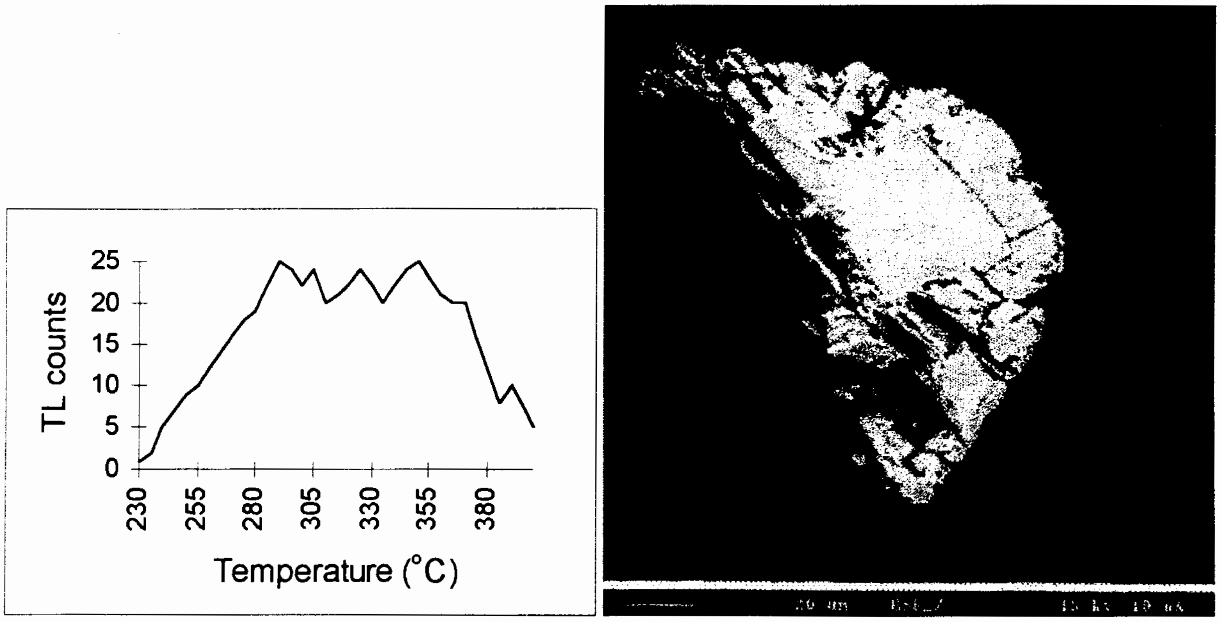


Figure 3

The backscatterd electron image of a grain of feldspar from the Belcroute (b) sediment, along with the second glow TL obtained from the grain.

It was found that the 10% HF etch had removed much more of the outer surface of the feldspar grains that had been previously expected from consideration of the TL literature. This loss of much of the outer surface meant that it was not possible to see any unambiguous signs of weathering. In addition, it was seen that one or two grains had been heavily attacked by the HF etch and were severely degraded with loss of much of the mineral structure. For example, figures 3 and 4 show the backscattered electron images from two grains from the Belcroute (b) sediment, along with the second glow curves associated with each grain. Figure 3 shows a feldspar grain which typifies most of the grains examined with the SEM in that it is intact but signs of attack along the cleavage planes are clearly visible. Figure 4 shows a second feldspar grain which shows signs of an intense attack by HF such that most of this grain has been dissolved away. The darker areas of this second grain were investigated using the EDS and were found to be reprecipitated silica. It is likely that, as a result of the HF etch, most of the K, Ca and Na ions in the feldspar matrix migrated into the HF solution leaving the silica to be reprecipitated. Small areas of surviving K-feldspar are visible in the photograph as regions of brighter contrast. However, this grain had virtually no second glow TL.

In summary, it was found that the effect of the "routine" HF etch was to completely remove all traces of weathering from the grains, and in a few cases the grain was almost destroyed by the HF etch. At the start of these studies such a severe HF attack was not expected but this effect has recently been confirmed by other authors (eg Duller 1994) and consequently any etching of feldspars has been abandoned. Thus, because of the HF etching, it is not possible to identify the effect of weathering on the TL of the majority of the grains measured in this paper. Nevertheless, grains which were severely attacked by the HF etch were found to have an extremely low TL, both natural and second glow (for example, figure 4). Therefore, it is likely that grains which had undergone severe chemical weathering would tend to show very low TL. These grains would not contribute any significant TL to the overall light sum when conventional TL measurements are made using bulk samples of grains.

The possible effect of grain mineralogy on the corresponding TL was also investigated on the grains from the Blassac sample. Previous measurements on bulk feldspathic samples, containing several thousand grains, have suggested that there is a large variation in TL sensitivity between different feldspar types. For example, Spooner (1993) found a difference of up to six orders of magnitude in the second glow TL sensitivities. A factor of 10 times was observed in this study between the median TL intensities of grains from the microcline and orthoclase geological samples. Thus, a range of TL sensitivity may be expected within a sediment sample containing different feldspar types such as will be present in the Blassac sample.

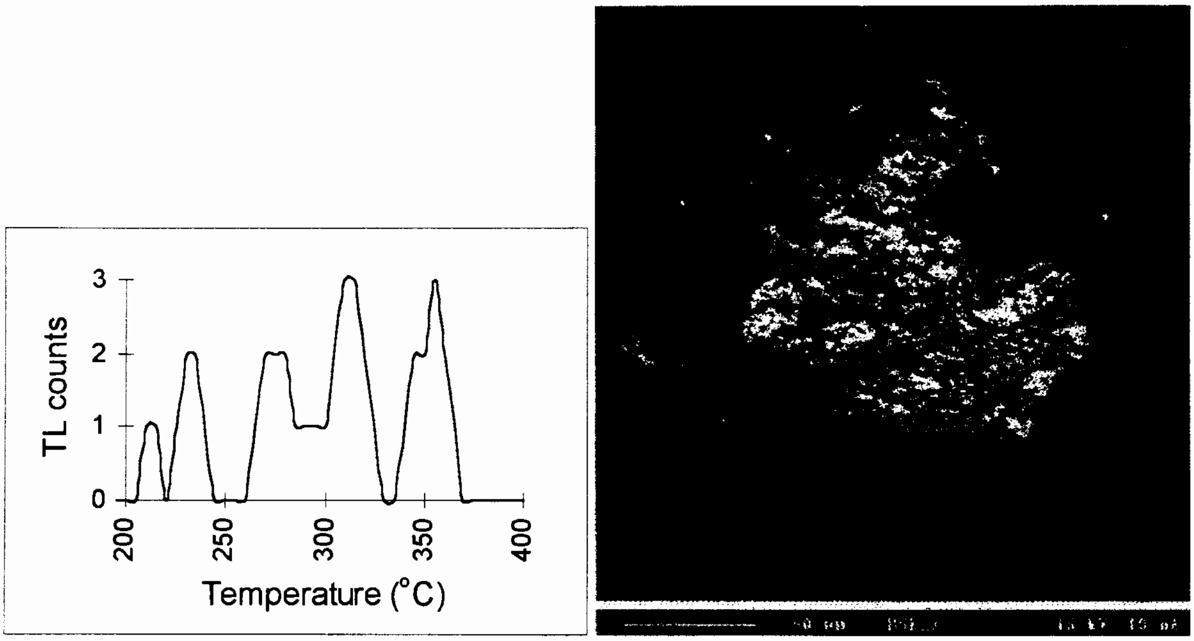


Figure 4

The backscattered electron image of a heavily degraded feldspar grain from the Belcroute (b) sample, along with the second glow TL obtained from the grain.

All the grains from the Blassac sediment were examined with the EDS and the percentage oxides present of silicon, aluminium, potassium, calcium , sodium, manganese, magnesium, iron, phosphorous and titanium were determined. The elemental concentrations obtained by the EDS measurements were converted into the percentage of the three end members of the feldspar ternary diagrams using the procedures outlined in Deer, Howie and Zussman (1983, p515), that is, orthoclase (high potassium), anorthite (high calcium) and albite (high sodium). It should be noted that to completely characterise each grain it would also be necessary to obtain information on the crystalline phases present, using X-ray diffraction. Although this data could not be obtained, nevertheless, in a general study of single grain luminescence the information obtained from the chemical composition alone is sufficient to allow the grains to be assigned approximate positions on the ternary diagram.

Figure 5 shows the feldspar composition for grains from the Blassac sediment plotted against the corresponding second glow TL for each grain. The sample contains a range of feldspar types within the single population of grains measured. However, it is difficult to see any corresponding general trend in the TL, but instead there is a wide variation in TL intensity for feldspar grains independent of chemical composition. This suggests that, within any one sample, mineral type by itself may not be the dominant cause of variations in TL sensitivity.

Conclusions

In summary, the bulk TL sensitivity varies on a sample-to-sample basis, as expected. However, the second glow TL variations within each sample are very similar, irrespective of the type of sample (sediment or mineral type) measured. Also, any outliers observed are not several orders of magnitude brighter than the majority of grains in each sample but are usually only some twenty to thirty times brighter than the median second glow TL in each case. The outliers within the labradorite and orthoclase samples can be explained simply in terms of grain size effects. However, the outlying grains from the other samples cannot be so explained, nor can they be explained in terms of grain-to-grain differences such as physical orientation or grain transparency.

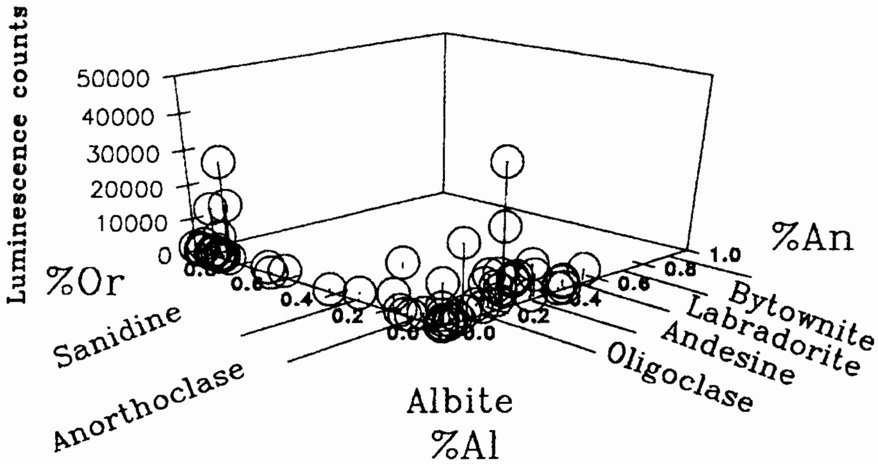


Figure 5

The second glow TL for single mineral grains from the Blassac sample plotted against the corresponding mineral composition for each grain.

Investigations of grain mineralogy found that, whilst there was a variation in both the TL sensitivities and the grain mineralogy within a sediment sample, variations in the TL could not be correlated with differences in mineralogy of the corresponding grains. The effect of weathering on the TL sensitivities of the sediment samples could not be investigated because of the unexpected strength of the HF etch. Nevertheless, it was possible to see that grains which had been significantly altered chemically by the etch, and which could correspond to heavily weathered grains in nature, had negligible TL.

Therefore, the results presented in this paper suggest two important findings about the second glow TL behaviour of single feldspar grains. Firstly, grains having a high second glow TL do exist within most samples and these grains are bright enough to contribute almost half of the total light sum. Secondly, the grain-to-grain variation within a sample which at a fundamental level will be determined by the concentrations of traps and luminescence centres present, does not appear to be related in a simple manner to observable physical parameters such as grain shape or the mineralogy of each grain.

Acknowledgments

The help and advice of Professor Mike Tite, Chris Doherty and Lindsey Shepherd is appreciated. I would like to thank Nigel Spooner and J. P. Reynal for providing some of the samples measured in this paper. Funds for this project were provided by RLAHA.

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J. Faïn