Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Kortekaas, M. and Murray, A., 2005. *A method for the removal of mica from quartz separates.* Ancient TL 23(2): 43-46. https://doi.org/10.26034/la.atl.2005.386

This article is published under a *Creative Commons Attribution 4.0 International* (CC BY): https://creativecommons.org/licenses/by/4.0

© The Author(s), 2005

A method for the removal of mica from quartz separates

M. Kortekaas[a,b] and A.S. Murray[a]

[a] Nordic Laboratory for Luminescence Dating, Department of Earth Sciences, Aarhus University, Risø National Laboratory, DK-4000 Roskilde, Denmark

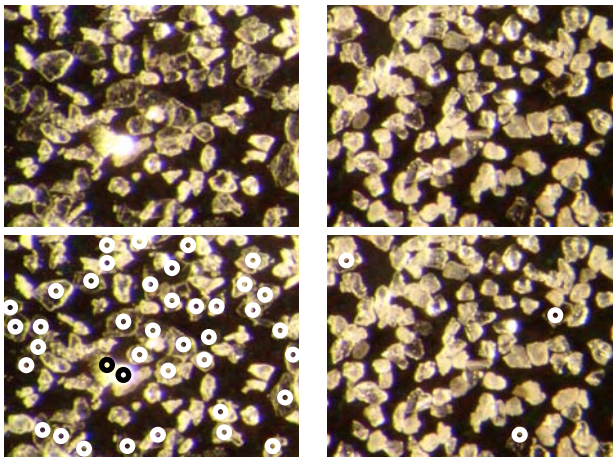
[b] GeoBiosphere Science Centre, Department of Geology, Quaternary Sciences, Lund University, Sölvegatan 12, SE-22362 Lund, Sweden

(Received 21 November 2005; in final form 15 February 2006)

Sediment samples often contain appreciable amounts of mica, a complex group of alumino-silicate minerals with varying chemical composition which have a strong cleavage that results in their platy structure (Klein and Hurlbut, 1993). Muscovite and biotite are usually the most common forms found in detrital sediments. Standard laboratory treatments (Aitken, 1985) to obtain pure quartz or feldspar for luminescence dating do not seem to reject mica minerals. A few studies have described the luminescence properties of mica (e.g. Kristianpoller et al., 1988; Clark and Sanderson, 1994). Not much is known about whether mica is actually a contaminant in luminescence dating studies, in the sense of whether it contributes significantly to the luminescence signals measured from quartz or feldspar. Excess mica can be removed by spreading the sample over statically charged surfaces (e.g. plastic envelopes or beaker surfaces) on which mica particles tend to stick. This, however, has the disadvantage that a considerable amount of quartz is also lost, especially when small grain sizes are used. We describe an effective and simple procedure to remove mica from etched quartz samples using a detergent solution in an ultrasonic bath.

Removal of mica using a detergent

The sediment used for this study was from a sediment core from the southern Baltic Sea (Kortekaas et al., submitted). The samples were wet-sieved to obtain the 63-106 µm diameter fraction. Purified quartz was obtained by treatment with H_2O_2 (10%) to remove any organic material, HCl (10%) to dissolve carbonates, concentrated HF (38%) to etch the surface of the quartz grains and to remove feldspar, and HCl (10%) again to remove any remaining soluble fluorides. However, after these standard laboratory treatments, some of the quartz samples contained up to 60 % mica by volume (by eye; Figure 1a). The low sand content of these samples precluded the use of statically charged surfaces to remove mica, because too large a fraction of the quartz was lost in this process. Instead, the samples were put in a detergent solution (sodium pyrophosphate solution ($Na_4P_2O_7$; 22.3 g/l) or dishwashing detergent solution) in an ultrasonic bath for ~30 minutes. Subsequently, the detergent solution was decanted and the sample rinsed with distilled water. The visible mica contamination, relative to quartz, decreased by ~90 % (see Figure 1) and the measured loss of total mass was 24 %. Presumably, the mica simply floats off when the soap is decanted. To estimate the quantitative quartz loss when applying this procedure, a clean (mica-free) quartz sample (63-106 µm) was processed in the same way, and the loss was <1% of the total starting mass.



(a) (b)

Figure 1: *Photographs of (a) a contaminated sample and (b) the same sample after treatment with detergent in an ultrasonic bath. Mica content decreased by ~90% (by eye). The open circles on the copies of the photographs at the bottom of the figure indicate the mica grains. The sample was taken from the same depth as sample F580, but from the half of the core which had been exposed to light.*

Luminescence measurements

All measurements used a Risø OSL/TL reader with a beta source giving a known dose rate to quartz. Optical stimulation was with blue (470 nm) light emitting diodes (LED), or with infrared (880 nm) LED. Detection was through 7 mm of U-340 glass filter and all stimulation was at 125ºC, the temperature at which routine quartz OSL measurements are performed.

To investigate the possible influence of mica on quartz luminescence measurements, three aliquots of pure mica grains (manually selected under the microscope) were measured using a single aliquot regenerative dose (SAR) protocol with a preheat of 220ºC and a cut heat of 160ºC (as was used with the quartz samples). The mica consisted mainly of muscovite and some biotite. The blue light stimulated natural signals from these 3 mm diameter aliquots of mica (63-106 µm) were detectable from two of the three aliquots, and was low (respectively 18, 84 and 698 photons in the first 0.8 s) (Figure 2a). Clark and Sanderson (1994) have shown that mica gives a luminescence signal under infrared (IR) stimulation, but we did not detect any IR stimulated luminescence following a beta dose of ~30 Gy (Figure 2b). However, a measurable sensitivity to blue light was observed. The blue light OSL decay curves after a beta dose of ~60 Gy (approximately double the quartz D_e of the sample) are shown in Figure 2c for all three aliquots. The natural signal from purified quartz of similar burial dose to the mica sample F580 is shown in Figure 2d. Note that both aliquot 3 and sample F560 were measured on a different reader from aliquot 1 and 2, but nevertheless it appears that the mica signal is small compared to the quartz signal. Although it is not possible to directly compare the relative brightness of the signals, it should be noted that the rate of optical eviction from the mica samples (Figure 2c) and the quartz sample (Figure 2d) is similar.

It is possible that the blue light stimulated OSL signal from sedimentary mica is from fine quartz grains that adhered to the mica grains. The samples were treated with concentrated HF, so fine grain quartz contamination seems unlikely, but it is possible during manual mounting of mica grains that quartz grains were inadvertently included. In order to check this, we measured OSL and TL on museum specimens of mica (muscovite) and compared them with the signal from sedimentary mica and purified quartz. The museum specimens of muscovite were crushed to approximately 2 mm diameter, and rinsed with distilled water in a 150 µm sieve to remove possible dust particles. The low temperature TL glow curve of our museum sample (after heating to 350°C

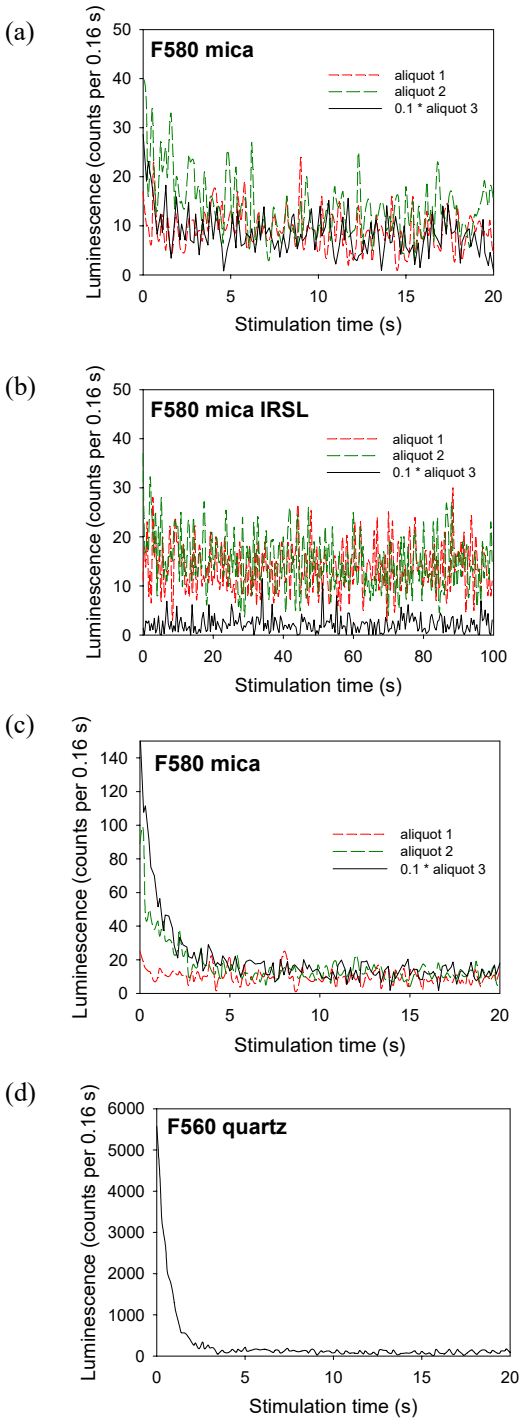


Figure 2: *(a) OSL decay curves for three 3 mm aliquots mica (63-106 µm) show a very weak natural sensitivity to blue light (aliquot 2 and 3) and (b) no visible sensitivity to IR. (c) The OSL decay curves after the same aliquots have been given a 60 Gy dose. (d) A typical OSL decay curve for a purified quartz sample of similar depth and burial dose (D_e ~30 Gy). Note that in (a), (b) and (c), the results for aliquot 3 have been multiplied by 0.1 for display purposes.*

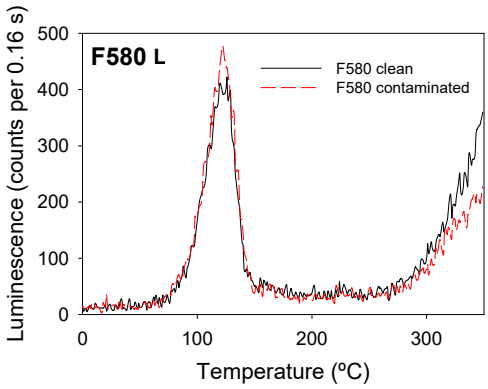


Figure 3: *TL glow curves for both a mica contaminated sample and the same sample, after removal of mica using the method described in this paper, after a ~50 Gy dose (light exposed F580). Heating rate was 5ºC/s.*

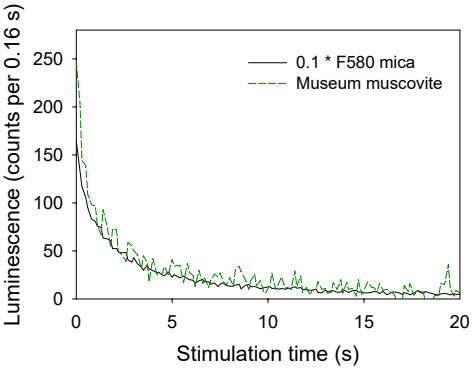


Figure 4: *OSL decay curves for an aliquot of sedimentary mica F580 (solid line) and an aliquot with museum specimens of muscovite (dashed line) after a 700 Gy dose. The dark count of the reader system was subtracted from these data sets to enable comparison of the curve shapes.*

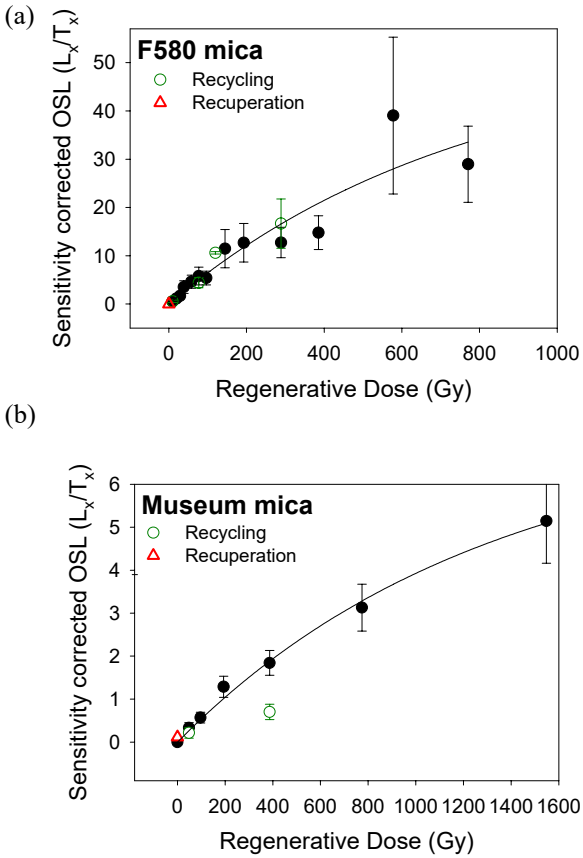


Figure 5: *Single aliquot regenerative dose growth curves using a 220ºC preheat for 10 s and a 160ºC cut heat for (a) sedimentary mica (F580) and (b) the museum specimen of muscovite.*

and giving a dose of 200 Gy) showed a peak just above 100ºC which was visually indistinguishable from the 110ºC TL peak from a purified quartz sample, although several orders of magnitude less sensitive. Thus the presence/absence of a similar weak TL peak in the sedimentary mica aliquots cannot be taken as evidence for quartz contamination. Figure 3 shows the TL glow curves from both a mica contaminated quartz sample and the same sample after removal of the mica (light exposed F580) after a ~50 Gy dose. No significant differences can be seen in the shape of the TL glow curve after removal of the mica. The OSL decay curves were also indistinguishable although the contaminated sample had a lower absolute light level. Figure 4 shows the blue stimulated OSL decay curves from aliquots of both museum muscovite and sedimentary mica (F580) after a 700 Gy dose; the high dose was used to make the shape of the decay curve clearer. The OSL decay curve of the museum muscovite specimen shows an initial fast decaying component, similar to that of quartz, but the signal also contains a significant slow component. The OSL decay from the sedimentary mica is similar. As a further check we have measured the growth curve of both the sedimentary mica (F580) and museum muscovite (Figure 5a and b). Although the sedimentary mica (Figure 5a) shows significant scatter, saturation seems to be at much higher doses than for quartz. The museum muscovite is still not in saturation at 1600 Gy.

We conclude that we cannot be completely sure that these mica luminescence signals are not, in fact, derived from quartz contamination, but given the similarity to the response of the museum sample, this seems unlikely. In any case, it seems prudent to minimise mica content when this forms a large part of a quartz sample after etching, especially in older samples. The method that we have proposed for

removing mica, using a detergent solution, is effective and is less likely to involve inadvertent loss of quartz. In this study, most of the sedimentary mica consisted of muscovite and some biotite. These were not separated, and we do not know whether different mica minerals possess different luminescence characteristics. Further investigations are necessary to determine whether these observations are generally applicable and whether mica itself has any potential as a luminescence dosimeter.

Acknowledgements
A NorFA mobility scholarship enabled MK's stay at Risø. We thank Prof. A.K. Singhvi and Prof. G. Duller for constructive comments.

References

Aitken, M.J. (1985). *Thermoluminescence Dating*. Academic Press, London, 359 pp.

Clark, R.J. and Sanderson, D.C.W. (1994). Photostimulated luminescence excitation spectroscopy of feldspars and micas. *Radiation Measurements* **23**, 641-646.

Klein, C. and Hurlbut, C.S. (1993). *Manual of Mineralogy*. John Wiley and Sons Inc, New York. 681 pp.

Kortekaas, M., Murray, A.S., Sandgren, P. and Björck, S. (submitted). OSL chronology for a sediment core from the southern Baltic Sea; a complete sedimentation record since deglaciation. *Quaternary Geochronology*

Kristianpoller, N., Kirsh, Y., Shoval, S., Weiss, D. and Chen, R. (1988). Thermoluminescent properties of mica. *Nuclear Tracks and Radiation Measurements* **14**, 101-104.

Reviewer
Ashok Singhvi