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PURITY TESTING OF TL MINERAL SEPARATES BY CATHODOLUMINESCENCE

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Many TL measurements are performed on mono-mineralic powders which have been separated from the sample of interest. A familiar example is the measurement of quartz separates in the quartz inclusion method of pottery dating. In such instances, it is usually difficult to ascertain the level of purity of the mineral separate. Sample purity is extremely important, particularly when measuring TL minerals of low sensitivity such as quartz, because only a very small amount of contamination by bright TL mineral grains, such as feldspar, can result in a significant level of aberrant TL. This type of contamination can be catastrophic for several reasons. First, the contaminating minerals might exhibit anomalous fading. Second, their dosimetry may be drastically different from that of the desired mineral. And, third, the variable number of stray grains in each TL aliquot will lead to poor reproducibility.

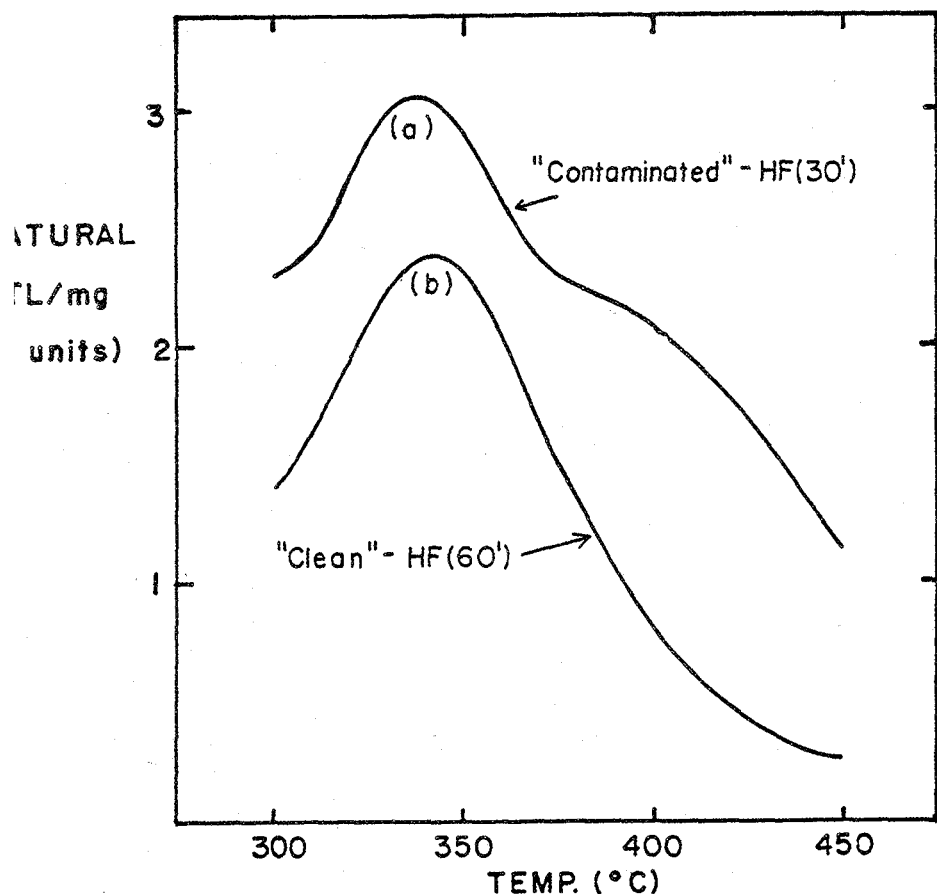
A particularly valuable and convenient tool in verifying the purity of TL mineral separates is cathodoluminescence (CL). The instrument used in our laboratory is the "Luminoscope" manufactured by Nuclide Corporation (Acton, Mass. U.S.A.). Basically, the device is a cold-cathode electron gun attached to a small vacuum chamber (roughly, 13cm x 20cm x 3cm). Samples in the form of powders, slabs or thin sections can be placed in the chamber and continuously bombarded with electrons up to 18keV (typical operating current is 0.3mA). The chamber is equipped with leaded glass windows on the top and bottom for viewing the sample and is well-suited for attachment to the stage of an optical microscope. Our particular instrument is attached to a Bausch and Lomb Stereozoom binocular microscope (with camera attachment) providing a maximum magnification of 140x.

In practice, a dozen samples can be easily loaded simultaneously. Evacuation of the chamber to about 30 microns pressure requires on the order of ten minutes and a usable beam can be obtained in as little as a few seconds. For optimum stability, the chamber should be evacuated to 10 microns and the beam maintained by dry inert gas (e.g., helium) introduced through the supplied needle valve attachment. The electron beam is focussed and centered in the microscope field of view using focusing magnets and samples are introduced to the beam by varying the position of the internal sample tray.

The excitation of mineral powders of small grain-size ($<1\text{mm}$) is somewhat complicated by the tendency of the grains to charge and assume ballistic trajectories. However, we find that this is usually avoided by operating at high voltage but low current. In the case that high current is necessary to excite adequate luminescence from the sample, our standard procedure is to epoxy the grains to a microscope slide being careful not to completely cover the grains with epoxy. (This can easily be accomplished by spreading a very thin layer of epoxy on the slide with the edge of another slide, sprinkling on the grains and curing at room temperature).

An example of the usefulness of this technique is provided by some measurements performed on quartz separated from a heated granite rock from a Colorado archaeological hearth. The basic separation procedure was to soak 3mm diameter chips of the rock in concentrated HF acid to eliminate the majority of non-quartz material (principally, feldspar). After 30 minutes soaking followed by 15 minutes in aluminum chloride solution (Carriveau, Ancient TL no.1, 1977) a very clean-looking powder of white grains was obtained. Subsequent TL analysis resulted in very unreproducible weight-normalized natural TL (standard deviation of 30%). The natural TL glow curve of this material (curve a) consisted of a peak at about 340°C with a noticeable bulge near 400°C. The equivalent dose curve (plateau test) also showed a bump at 400°C. CL viewing of the separate revealed the presence of a very few, bright blue feldspar grains. The level of contamination was on the order of a few grains in a 5 milligram aliquot or about 1:1000. An additional 30 minutes in the acid eliminated these grains as confirmed by CL and the reproducibility improved to 5%. In addition, the 400°C TL component disappeared (curve b) and the equivalent dose curve became more level. Obviously, the poor reproducibility resulted from the variable number of bright grains in the aliquots.

"QUARTZ" SEPARATE GLOW CURVES



Natural TL glow curves for aliquots of 74-420 micron "quartz" powder separated from the Colorado granite TL measured with Corning 5-60 plus 5-58 filters at a heating rate of 30°C/sec. Background negligible to 450°C.

- (a) 30 minute HF soak-
CL shows contaminating bright grains.
- (b) 60 minute HF soak-
CL shows clean quartz separate

Similar experiences have been encountered with quartz separates from pottery sherds. In one such case, quartz separates from Texas pottery were found to yield poor plateaus (measurements by D. Zimmerman). CL examination showed the presence of a few very bright zircon grains. Heavy liquid extraction of the zircon was required in this instance and good plateaus for the "pure" separate resulted.

Of course, this procedure prohibits subsequent use of the electron-excited grains for TL determination of archaeological dose. In the case that sample size is limited, the CL examination can be performed on "glew" material to verify separate purity.

5 SPURIOUS TIDBITS

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(1) Ordinary aluminium sample discs show some spurious TL themselves and this is a serious problem with samples too thin to obscure the disc. We have eliminated this problem by cleaning the discs in hydrofluoric acid before each use as follows. After removal of grease with an organic solvent such as trichloroethylene the discs are placed in a glass beaker (~100 ml) with about 30 ml of water. About 3 ml of concentrated HF is added and stirred in; almost immediately there is considerable fizzing and the mixture becomes cloudy. After a few seconds the mixture is poured down the drain along with a large flow of water, and repeatedly rinsed with more water until the acid is all removed. We perform all these operations in a fume hood with a built-in water tap and drain.

(2) When some spurious TL is evident, but not dominant, it is tempting to subtract the reheat from the first glow to obtain the true TL. This is not the correct thing to do. The error of this method is immediately apparent if the reheat curve is higher than the first glow, as occurs on occasion. Another method of detecting this error is to stop the first glow at a lower temperature and then reheat. It is obvious that the first heating enhances the spurious signal although the mechanism for this is not known.