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# A simple staining method for K-feldspars before sample preparation for IR-OSL dating

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## Introduction

Potassium-feldspar (K-Fs) grains are favored for dating the last insolation of sand deposits when IR-OSL (optically stimulated luminescence using near-infrared light) is used (Aitken, 1994; Berger, 1996; Wintle, 1994). For this purpose, the K-Fs grains are floated in a heavy liquid (e.g., sodium polytungstate, available from Geoliquids Inc., 15 E. Palatine Rd., Suite 109, Prospect Heights, IL 60070, USA) at a relative density of 2.58 g/cm<sup>3</sup>. In eolian deposits such as sand dunes, the concentration of K-Fs is often very low (<1-5%), because feldspars are less resistant to weathering, comminution and dissolution than is quartz. If the K-Fs concentration is low, one can choose to date only the quartz fraction (e.g., Rendell et al., 1994), using either TL or G-OSL (OSL using green stimulation). However, if one prefers K-Fs grains for their several advantageous luminescence properties (compared to quartz), then K-Fs can be separated from quartz with a little patience.

We have found that a quick visual means of estimating quantitatively the concentration and physical character of K-Fs grains in dune sand is useful for planning efficient subsequent steps in sample preparation. X-ray diffraction is insensitive to detection of K-Fs concentrations below ~5%. The optical-microscopy Becke-line test (e.g., Stoiber and Morse, 1981) can be straightforward, but "not all grains give rapid, unequivocal answers" (Stoiber and Morse, op. cit.). Thus we have found it's use to be ambiguous and tedious for our samples, especially when searching for a minor component such as K-Fs. Other analytical procedures (e.g., scanning electron microscopy) require elaborate and expensive equipment for such low concentrations. Staining of the K-Fs grains followed by moderate-power optical microscopy offers an alternative, simple, visual, quantitative means for estimating concentration of K-Fs grains in sand dunes.

Staining techniques for geological purposes have been used for over a hundred years (e.g., Allman and Lawrence, 1972; Hutchison, 1974). Generally, certain dyes or reagents have the ability to color only a specific mineral, even in the presence of other similar minerals. Specifically, sodium cobaltinitrite (Gabriel and Cox, 1929) colors K-Fs lemon yellow but leaves quartz unaffected, and other feldspars generally uncolored. The disadvantage for IR-OSL sample preparation, in our view, of this and other published techniques for staining K-Fs (and other feldspars) is the relative effort they require. Either polished thin sections or (unpolished) grain mounts (using a heated cement) are required, to be exposed to controlled fumes of hydrofluoric (HF) acid and certain rinsing solutions in specially fabricated etching containers (e.g., Teflon), followed by drying and application of a staining reagent and other steps. We have employed a much easier and quicker procedure, which is a variant and simplification of the procedures compiled by Hutchison (1974).

Our procedure employs loose grains, and thus avoids the need to fabricate grain mounts and polished thin sections. It also obviates use of special containers, HF fumes, and sample heating. Grain-size separation is not necessary, but prior removal of small grains (e.g., <50-100 µm) is beneficial. Only a small fraction of the total sample is needed for staining. Following the usual removal of carbonates (with HCl acid) and organics (with H<sub>2</sub>O<sub>2</sub>), the general steps to be performed for staining are: removal of rust (iron) stains from the sample to facilitate laboratory staining and easy visual discrimination of stained from unstained grains; staining of the K-Fs grains; visual grain counting, using either plane-polarized transmitted light through a petrographic microscope, or unpolarized reflected light through a binocular low-power microscope. Use of plane-polarized light

can enhance (Allman and Lawrence, 1972) the target lemon-yellow color.

#### Removal of rust stains

Common yellowish rust stains hamper the further staining of samples, and later visual discrimination of artificially stained K-Fs grains. A saturated solution of oxalic acid dihydrate (a strong reducing agent) is added to a small amount of each sample to remove rust stains. This solution is best for removal of limonite stains. For haematitic deposits, a heated bath of concentrated hydrochloric (HCl) acid may be necessary as a substitute. Other, more elaborate, methods could be used (e.g., citrate-bicarbonate-dithionite or CBD). Our simple oxalic-acid procedure was successful in removing all but the most stubborn stains and individual (opaque) iron-mineral grains.

The oxalic acid solution is prepared, in quantity, on a hot plate in a fume hood. Solution temperature may be 50-80°C. To a chosen volume of distilled water in a beaker containing a magnetic stirring bar, add oxalic-acid powder slowly, permitting it to dissolve. Remove the solution from heat and cool to room temperature. There should be a small amount of undissolved powder on the beaker's bottom when cool.

Add the solution to the sample in a volume ratio 2:1. Cover the beaker and place in an oven at 40-50°C overnight or a full day. The solution will change to a yellowish color. A bright yellow color indicates that several applications of the oxalic-acid solution are needed. Care is required to prevent reprecipitation of used (yellow) solution onto the sample. This can be prevented by not allowing the sample to cool or evaporate significantly before decanting. Decant the solution and thoroughly wash the sample in distilled water.

#### Staining of a cleaned sample

Efficient staining is accomplished on bulk grains using a saturated solution of sodium cobaltinitrite (NCN hereinafter, available from standard chemical suppliers), following wash-etching of the sample with concentrated (~49%) HF acid on a fast, hardened filter paper. HF leaches away silica and leaves an etch-residue containing K and other elements. NCN reacts with K to form a yellow precipitate of potassium cobaltinitrite. Unetched grains will not noticeably stain (Bailey and Stevens, 1960). Once stained, the samples should not be shaken or stirred too briskly prior to visual examination, so that none of the coating is removed.

In a fume hood, place a Teflon funnel into a support ring, with a Teflon beaker beneath. Use a fast, hardened (may be ashless) filter paper, such as Whatman 541. The saturated NCN solution is

viscous, thus requiring a fast filter paper. Wet the paper with a few drops of distilled water. Add a small amount of cleaned sample. Add just enough HF to wet the sample and to produce just one drop on the lower end of the funnel. Let stand for 15 seconds. Rinse the sample with distilled water. Add a small amount of saturated NCN solution, allowing to stand for ~1 minute. Rinse the sample thoroughly with distilled water, remove the filter paper from the funnel and permit to air dry.

#### Visual grain counting

To a good first approximation, in the stained sample, K-Fs grains (excepting sanidine) will have a bright (lemon) yellow powdery coating, Na-Ca feldspars (plagioclases) will have a white powdery coating, and quartz will be unaffected. Increasing Na seems to reduce the effectiveness of NCN, and with sanidine grains, only pits and corners will take much of this stain. However, sanidine is unlikely to be a noticeable component in most sand dunes, occurring mainly in deposits derived directly from volcanic materials.

We have tested the above procedures with samples from three temperate-zone sand dunes. We had no specific mineralogical information about these dunes before. Grain counts were performed under plane-polarized light in a petrographic microscope. We classified the grains as "white" (plagioclase), "yellow" (K-Fs) and "total" (unstained, stained and opaque), and counted all grains in each of 4 quadrants of the field of view (moderate power) for 5 subsamples from each stained sample. The results are shown in Table 1.

| Sample | Total grains | %Yellow<br>(mean±S.E.)<br>(n=20) | %White<br>(mean±S.E.)<br>(n=20) |
|--------|--------------|----------------------------------|---------------------------------|
| A      | 1577         | 0.62±0.20                        | 5.48±0.70                       |
| B      | 1416         | 2.37±0.46                        | 5.81±0.64                       |
| C      | 1309         | 3.38±0.43                        | 4.72±0.51                       |

**Table 1** Grain-counting

These data demonstrate both the sensitivity of the method (sample A), and the ability to estimate the relative proportions of K-Fs and plagioclases. Even if half the total number of grains were counted, such data would likely still permit the deduction that sample A is problematical and will require special effort to produce sufficient K-Fs for all expected dating analyses. We note in passing that the statistical distributions of the populations of yellow and white grains were not all symmetrical nor normal.

### Conclusions

We conclude that the simplified staining procedures described here can permit relatively low-effort, low-technology quantitative estimates of both K-Fs and plagioclase concentrations in sand dunes having relatively low (<5%) concentrations of K-Fs. Samples can be processed in batches. The resultant grain-count data can be useful in planning efforts toward separation of K-Fs for IR-OSL dating. For the same reason, the concomitant knowledge of plagioclase concentration may be useful if plagioclase grains will be used for IR-OSL dating.

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Reviewer

**H.P. Schwarcz**

### Comments

This is a useful and simple method to determine the presence of IRSL-datable K-feldspar in dune sands. Where K-feldspar abundance is very low, a quantitative estimate of its abundance helps to predict the yield from heavy-liquid flotation. This method will probably not detect K-feldspar inclusions in quartz which are sometimes the last surviving, IRSL-datable component in mature sand (see Huntley, Hutton and Prescott, *Geology*, 21:1087-1090, 1993).