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Short Communication

A rapid method for hand picking potassium-rich feldspar from silicic tephra

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ABSTRACT

We detail a method for hand picking potassium-rich feldspars from silicic tephra. The method, based on the well known sodium cobaltinitrite staining technique, is applicable to any sample where K content of feldspar exceeds ~2 wt%. The method is well suited to mineral grains >0.25 mm and useful for grains as small as ~0.125 mm. Alkali feldspar, plagioclase, and quartz are easily distinguishable after staining. Rinsing the stained feldspars along with ultrasonic treatment in distilled water and nitric acid removes the stain. $^{40}Ar/^{39}Ar$ analysis of sanidine separated by staining yields precise age estimates which are in excellent agreement with published ages for the youngest (2.06 Ma) Cerro Galán ignimbrite, NW Argentina.

1. Introduction

For many analytical purposes (e.g., $^{40}Ar/^{39}Ar$ single crystal dating) it is desirable to separate potassium-rich feldspars (sanidine, anorthoclase, microcline, orthoclase) completely from other components of a rock. We report a simple, inexpensive, procedure for staining grain mounts to identify K-rich feldspars. This is based upon the well known method for staining feldspars with sodium cobaltinitrite (Gabriel and Cox, 1929; Keith, 1939; Chayes, 1952; Jackson and Ross, 1956; Hayes and Klugman, 1959; Bailey and Stevens, 1960). This method is most useful once K-feldspar has been concentrated using standard mineral preparation techniques (i.e. magnetic and density). Principally, staining permits accurate screening of samples such that contaminants can be easily removed and K-feldspar can be concentrated to purity. This method has been previously employed for $^{40}Ar/^{39}Ar$ age dating of feldspars from silicic tephra (cf. Chesner et al., 1991), but its details have not been reported and knowledge of its utility is not widespread.

The method described herein is neither limited to silicic tephra nor to $^{40}Ar/^{39}Ar$ geochronology; however, these applications are most commonly in need of pure K-feldspar. Modern, high-precision dating of Quaternary tephras that relies on a population of single-crystal analysis, or multi-crystal aliquots, has increased the demand that feldspar samples are pure (e.g., Chen et al., 1996; Ton-That et al., 2001).

Heavy liquid and magnetic techniques effectively concentrate K-feldspar, but complete purity can only be achieved by hand picking. A variety of techniques are available to ensure sample purity. Hand picking can be accomplished in immersion oils using the Becke line method to distinguish plagioclase and potassium-rich feldspars (e.g., Smith et al., 2003). A similar technique, focal masking, is described by Wilcox (1983), and was utilized extensively by Obradovich (1993). Of these immersion methods, focal masking has the advantage of simultaneously providing information for all grains in the field of view. Scanning Electron Microscopy and Energy Dispersive Spectrometry have also been successfully employed to distinguish K-rich feldspars from plagioclase (cf. Roger et al., 2000), but these techniques can be time-consuming and somewhat expensive.

Staining of quartz/feldspar concentrates with sodium cobaltinitrite provides a rapid method for positive identification of potassium-rich feldspars. It also confers the distinct advantage of requiring minimal lab equipment and facilitating rapid picking under a binocular microscope. Here we outline the method as applied to silicic tephra and assess the range of feldspar potassium content over which it is applicable. It is demonstrated that removal of the stain is easily accomplished and we also directly address concerns regarding potential influence of the staining procedure on $^{40}Ar/^{39}Ar$ age estimates of feldspar crystals.

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2. Pre-staining mineral preparation

Tephra samples with an abundant and coarse quartz/feldspar fraction may be quickly prepared by HNO_3, HF and H_2O washing in an ultrasonic bath. Aggressive decanting is effective at removing a large amount of the glass and biotite and is commonly sufficient to concentrate quartz and feldspar. If needed, quartz and feldspar may be further concentrated in the non-magnetic fraction using a Frantz Isodynamic Separator™. Some of the sanidine grains used for this experiment were also concentrated by density in lithium metatungstate. Staining and picking are greatly aided by HF treatment sufficient to remove adhering glass. If this is not done, the distinction between plagioclase and alkali feldspar is less obvious because most silicic volcanic glass has a K content ranging from 2 to 5 wt% and also stains yellow.

Standard 27 × 46 mm glass slides affixed with double sided adhesive (cellophane tape) are a convenient substrate for staining prepared mineral grains. Grains can be generously poured onto the slide and then thinned by tilting the slide. Slides with too many grains will take the stain poorly. Once the desired density of grains is achieved they can be more firmly affixed to the adhesive by covering the slide with weighing paper and applying pressure. For grains larger than ~0.5 mm, a single 27 × 46 mm petrographic slide generally holds in excess of 1 g of sample.

Once affixed, minerals are etched for ~5 min by HF vapor. Slides are placed upside down over a shallow container with concentrated HF. The grains should be closely exposed to the HF, but not be wetted with HF. The goal is to activate the surface of the mineral grains to take the stain. This surface is fragile; physical abrasion or wetting with acid quickly destroys the surface intended for staining. After etching, but prior to staining, the grain mounts should be allowed to dry in the fume hood.

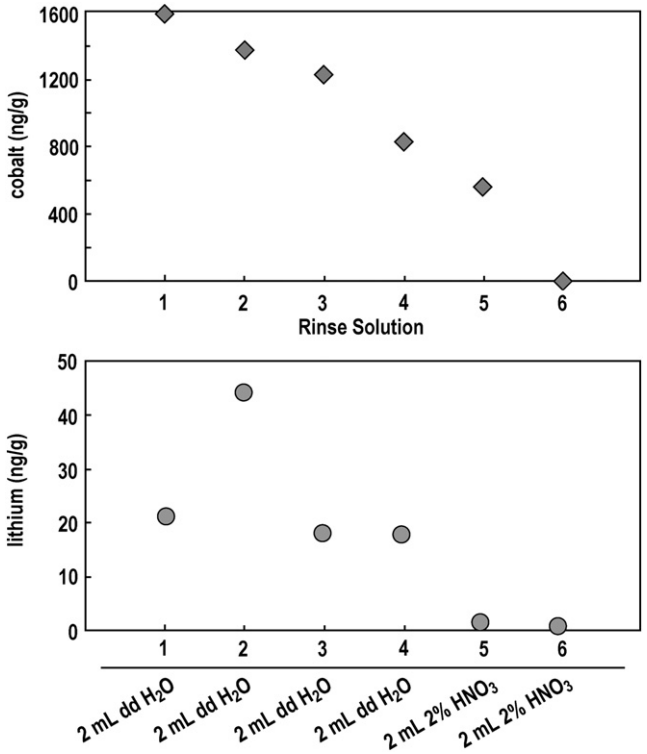

Fig. 2. Inductively coupled plasma mass spectrometry measurements of Co and Li concentrations (nanograms/gram) in rinse solutions from ten handpicked sanidine grains (0.25–0.5 mm) with K content > 11 wt%. The ten grains were agitated by hand for 30 s during each rinse step; the first four rinse steps were carried out in deionized distilled water (dd H_2O) and the final two steps were carried out in 2% HNO_3. Between rinse steps 5 and 6 the grains were subjected to ultrasonic treatment in 50 mL of dd H_2O, this solution was not measured.

3. Staining

The staining solution should be super-saturated by slow heating and addition of sodium cobaltinitrite powder until no more will dissolve. A 125 or 250 mL beaker on a small hotplate provides sufficient stain for 50 or more slides. If the beaker is covered with parafilm, it can be stored for days, and then refreshed with heat, water and sodium cobaltinitrite prior to use. A shallow container large enough to submerge a full slide is ideal for staining grain mounts. Slides should be submerged in the staining solution for no more than 2–3 min. If the slide is submerged too long all minerals will have a rusty orange coat. Forceps are necessary to remove the slide from the staining solution and should be used to immediately rinse the grain mount in water. It is helpful to use several beakers of water; a few short dips in the first beaker remove the majority of the stain and a second rinse in cleaner water greatly improves results.

Plagioclase, quartz, and alkali feldspar are easily distinguished after drying (Fig. 1). Quartz does not etch in HF and has a vitreous luster. Plagioclase etches in HF vapor, but does not take the sodium cobaltinitrite stain, resulting in a dull white coat. Alkali feldspars with high K content stain a bright yellow. The brightness of the yellow stain on alkali feldspars correlates with K content (Fig. 1). With care, anorthoclase containing as little as 2.5 wt% K can be successfully separated using this method. Oligoclase with 0.7 wt% K does not stain, so we infer that several wt% K is needed to positively identify K-rich feldspars using sodium cobaltinitrite.

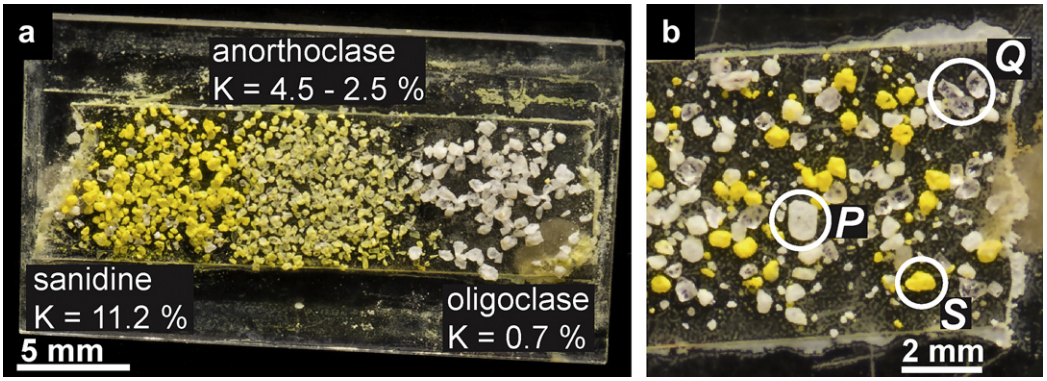

Fig. 1. Photographs of typical stained grain mounts: a) three different feldspar separates illustrating the range of K content for which the method is useful, b) a quartz/feldspar concentrate illustrating the differential effects of staining on quartz (Q), plagioclase (P), and sanidine (S).