

NOTES AND NEWS

NOTES ON THE STAINING OF POTASH FELDSPAR WITH SODIUM COBALTINITRITE IN THIN SECTION

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ABSTRACT

Several simplifying modifications of the staining procedure are described. The behavior of soda-rich or minutely perthitic potash feldspars is anomalous but the stain is highly selective for microcline in the presence of plagioclase and will not obscure perthitic intergrowths coarse enough to be readily resolved in unstained thin sections. The stain greatly facilitates both modal analysis and ordinary petrographic examination.

The staining of potash feldspar by sodium cobaltinitrite was suggested by Gabriel and Cox (1929) and extensively used by Keith (1939*A* and *B*), who combined it with an aniline dye stain for feldspathoids in his study of the "leucolitchfieldite" of Blue Mountain, Ontario. The stain takes excellently on the potash feldspar of most granites. It eliminates any possibility of confusing potash feldspar with untwinned acid plagioclase and materially reduces the time spent in modal analysis. Even where questions of identification and analysis are not involved, a satisfactory stain greatly facilitates study of the habit and distribution of potash feldspar in the slide. In the course of the last three years I have stained well over 300 thin sections of calcalkaline granites and have made a number of minor revisions in the technique of Keith and of Gabriel and Cox. These revisions are hardly of fundamental importance but they considerably simplify and shorten the staining operation. They are described here in the hope that a more general appreciation of the simplicity of the technique and the elegance of the result will encourage petrographers to make staining a standard procedure in the study of granitic rocks.

UNCOVERING OLD SLIDES' FOR STAINING

It is easier to work with new slides which have never been covered, but it sometimes may be desirable to uncover old ones. If the balsam is firm and hard the cover slip is easily sprung off with a razor blade. If the balsam is tacky the slide should be baked at about 80° C. until the tackiness disappears. In warm weather it is sometimes helpful to immerse the slide in cold water during removal of the cover slip.

After the slip has been removed, excess balsam is dissolved off the rock surface with xylol and alcohol. Gentle brushing with xylol for a minute, followed by brushing and rinsing in alcohol for two or three minutes, is usually sufficient. The surface will be sticky when the slide is removed from the xylol but should be firm and clean when it is taken from the alcohol. Following the alcohol rinse the slide should be washed thoroughly in running water. Any remnants of the covering medium still persisting may be removed by gentle rubbing with a little rouge or fine

alundum, and a poorly stained slide may be prepared for retreatment in the same fashion.

At nearly every step of the uncovering procedure the slide is in grave danger; the first few slides are almost certain to suffer serious damage and may be lost. After a little practice, however, losses during uncovering are negligible.

THE HF ETCH

A lead bath is usually prescribed for the HF etch, but a plastic container is cheaper, lighter, and easier to keep clean. The old-fashioned square-shouldered butter dish sold in most 5-and-10-cent stores for very little more than 10 cents works admirably. The top serves as the bath. The base, on which the butter ordinarily is set, is used as a lid. A rack or bridge which will support the specimen tray is easily fashioned out of other plastic novelties. At the time my apparatus was assembled the local dime store was featuring something called a "plastibasket," the center of which, severed from the rest with a wire clippers, made an excellent bridge. The specimen tray consists of a piece of 1/4 inch plywood cut to fit loosely into the bath. As near each end as possible a wood screw is mounted; the tray is lifted into and out of the bath by tweezers which hold firmly under the head of each screw. A better specimen tray could surely be designed but this one has served satisfactorily for more than two years and is still as good as new. The level of acid in the bath should always be well below the surface of the bridge.

The backs of the slides are covered, either by taping with a strip of cellophane or by painting with a thin solution of rubber cement in xylol, and the slides are brought up to the temperature of the bath, between 30° and 40° C. A one-minute etch at 30° is the equivalent of several minutes of exposure to fumes at room temperature, but if the temperature is much above 45° the balsam is quickly attacked and the slide is ruined. High temperatures will also melt the plastic bath. The slides are brought to the temperature of the bath or a little higher before treatment in order to hold condensation to a minimum; they should be dry when they enter the bath and not more than barely moist when they are withdrawn. Any droplets which have condensed on the surface should be allowed to dry before the slide is stained.

Three or four slides are placed *face up* on the specimen tray, the cover of the bath is removed, the tray is placed in the bath by tweezers applied to the wood screws, and the bath is immediately covered again. For most calcalkaline granites a 45-second etch is sufficient and in general the shorter the etch the more even the stain. Working with a hot etching bath and a long staining period, a 15-second etch is often adequate. An etch of more than 2 minutes is likely to bubble and discolor the balsam and usually leads to patchy, ragged staining. The micas, particularly biotite, may also be attacked, but they rarely stain properly. When the mounting medium is not attacked, a stain which takes the form of minute

equant crystals rather than a smooth, even coating is a good indication of overetching. Proper etching usually produces a faint frosting of the exposed upper surface of the slide; this frosting is often iridescent and can be removed by gentle rubbing. There is no need to remove it, however, for it will be washed off with the excess staining solution.

If the slides have been backed with cellophane tape the backing is removed as soon as the etch is completed. If rubber cement has been used it is more easily rubbed off during the rinse which follows the staining.

THE COBALTINITRITE STAIN

Instructions for making up the sodium-cobaltinitrite solution are given in both papers mentioned above, but the reagent can be purchased in powder form and is readily soluble in water. Both Keith and Gabriel and Cox recommend saturated acetic acid solutions, but the acetic acid is unnecessary and concentrations far below the saturation point sometimes do a better job than saturated ones. Three or four grams of reagent should be taken up in 6 cc. of water. Heating is not necessary but hastens solution. The liquid is at first yellowish red but becomes intensely brown or almost black when all the salt is dissolved. It should always be used at or below room temperature. Hot staining solution reacts as soon as it hits the slide, before it can be properly spread over the surface. The result is an irregular, blotchy stain.

The staining solution is quite stable but can be made up readily as needed, does not seem to improve with aging, and deteriorates rapidly with use. Ten cc. will usually be enough for 25 or 30 slides and each portion should be discarded after it has once touched an etched surface. It is best applied with a medicine dropper. Five or six drops are discharged onto the etched surface and then spread evenly over it with a glass rod or the side of the dropper. The surface of the slide must not be scratched during this operation, for each scratch will later appear as an unstained streak. When the liquid has been spread evenly over the entire surface a fresh drop or two may be added from the dropper. The slides are now allowed to stand for at least five minutes but much longer exposures do no harm and may be essential if a short etch or a weak staining solution has been used. It is to be remembered that the surface contains only a minute amount of available potassium and that every precaution has been taken to *avoid* prompt reaction. Overlong staining may permit the solution to evaporate to dryness or to work under the rock surface; in either case washing is likely to be difficult and unsatisfactory. If large batches are treated, the first slide is usually ready for rinsing a few minutes after the last one has been treated.

THE RINSE AND THE FINISHED PRODUCT

The potash stain is remarkably sturdy and the slides are best rinsed in a gentle stream of cold water from the tap. Excess reagent is quickly

removed and the slide is ready to cover as soon as it is dry. If the slides have been backed with rubber cement instead of cellophane tape, this should be rubbed off during the rinse.

In a properly stained slide the bright lemon yellow color of the potassium compound is prominent in plane light, almost imperceptible under crossed nicols, and sharply confined to potash feldspar. It has no noticeable effect on the index, extinction or birefringence of the potash spar, nor does it obscure or conceal perthitic intergrowths large enough to be resolved readily in unstained sections. In most rocks it is an immense aid in identification, modal analysis, and ordinary petrographic examination.

Cryptoperthites and non-perthitic or sub-microscopically perthitic alkali feldspars do cause trouble. Sometimes, as in the Deloro, Ontario, granite, they refuse to stain at all or stain very unevenly despite rather severe treatment. Sometimes, as with the pulaskite of Granite Mountain, Arkansas, they show little or no response to the ordinary procedure, but with more severe treatment take a smooth even stain which completely obscures the cryptoperthitic texture. My experience with rocks of this sort is still very limited but I am confident that much useful information would be generated by a careful study of the staining reactions of alkali feldspars, whether in plutonic or volcanic rocks.¹

Such a study would be impossible without an abundant supply of first-grade sections, and in general it is one of the handicaps of work of this type that much wastage of thin sections is involved. Slight modifications of technique which lead to striking differences in sections may have no perceptible effect on sawed or polished tablets examined under reflected light.

¹ Two modifications of the procedure, developed after this note was submitted to the journal, have eliminated most of the difficulty with cryptoperthite and alkali feldspar. The technique described above is adequate whenever the albite content, dissolved or exsolved, is low. When much albite is present or suspected the slide should be completely immersed and *gently agitated* in the staining solution. Suitable agitation of the slide largely overcomes the fatiguing effect noted above, and the staining solution need not be discarded after each application. The immersion technique is so satisfactory that I have begun to use it in routine work on calcalkaline rocks. Agitation materially shortens the necessary staining period. A 30-second etch followed by a 2- or 3-minute stain will usually be ample, but the alkali feldspar of some volcanic rocks may require as much as 10 or 15 minutes in the staining bath.

The smoothness and resolution of the stain are also considerably improved if the surface of the slide is smoothed before etching; the slide should be handlapped on a glass plate with #600 alundum for 2 or 3 minutes.

REFERENCES

- GABRIEL, A., AND COX, E. P. (1929), A staining method for quantitative determination of certain rock minerals: *Am. Mineral.*, **14**, 290-292.
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