SELECTIVE STAINING TO FACILITATE ROSIWAL ANALYSIS

M. L. KEITH, *Massachusetts Institute of Technology, Cambridge, Massachusetts.*

INTRODUCTION

There is a recent tendency in the geological sciences to introduce the quantitative factor in laboratory investigation. In petrography the development of the micrometer stage and more recently the development of electric counters for thin-section analysis[1] has greatly facilitated the modal determination of rocks. Larsen and Miller[2] have studied the accuracy and limitations of Rosiwal analyses and outlined the conditions as to grain size, area of thin section and number of traverses, under which the mode of a rock can be determined by the Rosiwal method with an accuracy of 1 per cent.

If the constituent minerals can be rapidly and accurately recognized without repeated optical tests a Rosiwal analysis can be made with an electric counter in about 35 minutes. If, on the other hand, it is necessary to stop and identify individual grains, both the speed and the accuracy of the method are reduced.

Among the common rock-forming minerals, untwinned plagioclase and potassium feldspar are in some cases difficult to distinguish from one another and from quartz by inspection. In the undersaturated rocks nepheline may present similar difficulties of rapid identification and distinction from untwinned feldspar. A combination of staining methods is here presented which solves this difficulty and thus allows increased speed and accuracy of Rosiwal determinations.

COMBINED STAINING METHODS

The proposed staining technique is based on two previously described methods for staining nepheline and potassium feldspar, respectively. So far as is known the two stains have not previously been used in conjunction.

The nepheline stain involves gelatinization with hydrochloric acid, followed by coloring with an organic dye which is adsorbed by the silica gel. This method is described in several textbooks of petrographic methods, but the original reference is unknown.

The potassium feldspar stain depends upon decomposition of the sur-

[1] Hurlbut, C. S., An electric counter for thin-section analysis: *Am. Jour. Sc.*, **237**, 253 (1939).

[2] Larsen, E. S., and Miller, F. S., The Rosiwal method and the modal determination of rocks: *Am. Mineral.*, **20**, 260 (1935).

face with hydrofluoric acid, followed by staining with a solution of sodium cobaltinitrite, a potassium test solution. This method was described by Gabriel and Cox,[3] for use principally in distinguishing between potassium and plagioclase feldspar in granular material. Slight modification of their procedure was found necessary to adapt the method for thin sections. Tests were made on nepheline-bearing alkaline rocks, metamorphic schists, and on syenitic and granitic rocks from the Bancroft area in Ontario. It was found that these could all be successfully stained without change in the reagents or timing, but, as noted below, the procedure for quartz-bearing rocks omits the nepheline stain.

Procedure

(1) A preliminary microscopic examination of the thin section is made and the minerals identified qualitatively. It is advisable to make any necessary optical determinations before staining, since the treatment partially obscures the optical properties of some minerals.

(2) Stains are applied to thin sections which have been prepared without cover glasses, or to sections from which the covers have been removed. In the latter case it is necessary to thoroughly clean the surface of the rock section, first with xylol and then with alcohol.

(3) The exposed glass of the slide, (not the rock section itself), is covered with a thin coating of celluloid, (solution 1)[4] to protect the glass from hydrofluoric acid during subsequent fuming. The celluloid solution is applied with a small camel's-hair brush.

(4) For Nepheline-Bearing Rocks: The section is laid flat on a table and covered with concentrated hydrochloric acid, applied with a pipette. The acid is allowed to stand for *4 minutes* and is then washed off gently by repeatedly dipping the section in water. Care must be exercised to avoid washing away the gelatinous silica, which is not held tenaciously to the nepheline.

(5) While the section is still wet it is covered with an aqueous solution of malachite green, (solution 2), applied with a pipette. This is allowed to stand for *50 to 60 seconds* and is then washed off (again gently), by dipping in water. At this stage nepheline is stained a strong green.

(6) The section is now dried thoroughly for 24 hours. It should not be heated, as this tends to cause the stained silica gel to chip and come off. If the section is damp at the hydrofluoric acid-fuming stage it will be completely decomposed.

(7) The dried section is set in a lead tray and placed in the lead fuming

[3] Gabriel, A., and Cox, E. P., *Am. Mineral.*, **14**, 290 (1929).

[4] Reagents and apparatus are described at the end of the article.

box (apparatus 1), and exposed to hydrofluoric acid fumes at 65°C. for *50 seconds*. The green color of the nepheline stain is destroyed at this stage, but is brought back during subsequent treatment with the feldspar stain.

(8) After fuming, the section is covered with the feldspar stain (solution 3), applied with a pipette, and allowed to stand for *30 to 40 seconds*. Potassium feldspar is stained a strong yellow.

(9) Finally, the section is again washed carefully and dried slowly, and a cover glass is cemented on with liquid Canada balsam. Stained sections should not be heated when applying the cover glass as they are quite fragile and will break up if the lower balsam layer becomes soft. If the cover is applied with liquid balsam (not heated), the balsam remains sticky for a few days but the sections can be used almost immediately.

The above procedure leaves potassium feldspar yellow, nepheline greenish blue, and plagioclase feldspar remains uncolored. The staining treatment obscures the optical properties of nepheline, but optical determinations can still be made on the feldspars. The colors of both stains appear to be permanent on covered thin sections; no change was observed in stained sections after three months.

For Quartz-Bearing Rocks, or for rocks which contain neither quartz nor a feldspathoid, steps (4) and (5) above are omitted. The resultant potassium feldspar stain is the same. In addition, the surface of plagioclase feldspar is roughened and its apparent relief in thin section is raised so that untwinned plagioclase can be distinguished at a glance from quartz, which remains clear.

Application of the Method to Other Minerals

The same staining procedure, omitting steps (4) and (5), offers a method for distinguishing in thin section between leucite and analcite, minerals which are not easily identified by optical methods. Under the hydrofluoric acid and sodium cobaltinitrite treatment, with timing as for the feldspars, leucite is stained yellow and analcite remains uncolored.

This procedure also distinguishes alunite from kaolin, talc, sericite and brucite. This was not tested in thin section, but on a polished surface alunite is the only one of this group of minerals to show the yellow stain. It might be expected that sericite would also be stained, since the yellow precipitate depends upon the presence of potassium. This is not the case, however, since the micas, unless altered, are not sufficiently decomposed by the hydrofluoric acid treatment to give an appreciable stain with sodium cobaltinitrite. This behavior of the micas under the staining treatment was noted by Gabriel and Cox.

Other minerals tested in the rocks were:—hornblende, pyroxene, biotite, corundum, ilmenite, magnetite and calcite. The first five of these are inappreciably affected by the combined staining procedure, and their recognition is not impaired. Magnetite is almost completely dissolved and calcite completely dissolved by the HCl treatment, but enough magnetite remains so that the grains can be measured in Rosiwal analysis.

Further possible applications of these two stains may be suggested by the following notes—(minerals not specifically tested for the present investigation). In addition to the minerals referred to, the following dissolve in hydrochloric acid with surface gelatinization:—cancrinite, chondrodite, chrysotile, melilite, olivine and the zeolites.

The following dissolve in HCl without surface gelatinization: apatite, chlorite, cordierite (partly), epidote (partly), gypsum, sodalite, vesuvianite (partly), and the calcic members of the scapolite series.

The following may be expected to be inappreciably affected by the combined staining procedure: andalusite, axinite, beryl, goethite, garnet, hematite, kyanite, ottrelite, rutile, sillimanite, staurolite, sphene, tourmaline, zircon and zoisite. Hematite and goethite are slowly attacked by HCl.

Apparatus and Reagents

Apparatus 1—A much simpler fuming box than that described by Gabriel and Cox is suitable for thin sections, polished rock chips or granular material. This can easily be made by folding one-sixteenth inch lead sheet into a rectangular box two inches by three inches in plan dimensions, and three-quarters of an inch deep. A shallow tray and stand and a flat cover for the fume box can also be made from lead sheet without special equipment (Fig. 1). In use, concentrated hydrofluoric acid is poured in the box to a depth of about one-eighth of an inch and the box is heated from below with a low bunsen flame to about 65°C.

Solution 1 (protective coating). Sheet celluloid dissolved in acetone to about the consistency of glycerine.

Solution 2 (malachite green). Prepared by dissolving one gram of malachite green in 200 cc. of distilled water.

Solution 3 (potassium stain). Concentrated sodium cobaltinitrite solution, prepared by adding 15 cc. of glacial acetic acid and 25 cc. of water to 12.5 grams of $Co(NO_3)_2 \cdot 6H_2O$ and 20 grams of $NaNO_2$. Upon standing a yellow precipitate is developed in this solution. The solution is apparently stable in contact with the precipitate and should not be filtered.

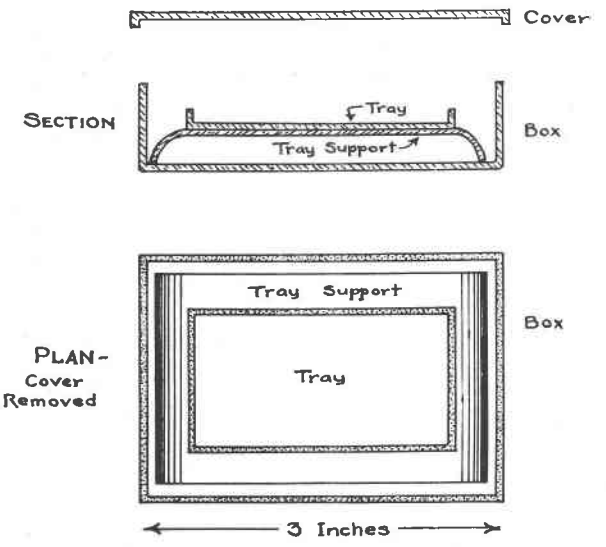

FIG. 1

FURTHER APPLICATIONS

The staining technique is considered to have its most important application in distinguishing minerals rapidly for Rosiwal analysis, but it may be found useful in some cases for mineral identification where optical methods are not entirely satisfactory, and also for facilitating study of the textural relations of rock minerals. For example, the potassium feldspar stain brings out perthitic intergrowths of the feldspars in a striking fashion. Both the gelatinization stain and the cobaltinitrite stain can be applied to polished rock chips as well as to thin sections. Rock chips should be heated, however, before being placed in the fuming box, otherwise the hydrofluoric acid will condense on the rock and the resultant yellow precipitate from the staining solution will be smeared over the surface.

It is difficult to illuminate stained polished surfaces well enough to permit these to take the place of thin sections for Rosiwal analysis. Mineral proportions can be estimated from stained polished sections, however, and the textural relations of a medium or coarse grained rock can be observed with a hand lens or binocular microscope.