

many specimens, and to Mrs. Richard L. Armstrong, Yale University, for the spectrographic analysis of wurtzite.

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THE AMERICAN MINERALOGIST, VOL. 47, JULY-AUGUST, 1962

THE DETERMINATION OF DIOCTAHEDRAL MICA AND POTASSIUM  
FELDSPAR IN SUBMICROSCOPIC GRAIN SIZES

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## INTRODUCTION

The method reported here has been used successfully as a means of quantitatively determining illite and potassium feldspars in the size range  $<10 \mu$  and  $<2 \mu$ ESD.<sup>1</sup> The method was developed using ground muscovite standards. Because of the structural and chemical similarity between muscovite and illite, it was assumed that the technique was applicable to illite-potassium feldspar separations. The term "dioctahedral mica" will be used here to describe micas of either illitic or muscovitic composition.

The method is based upon the differential solubility of potassium feldspar and dioctahedral mica in HCl after heating to 825° C. Heating muscovite to 800° C. for several hours causes a loss of hydroxyl water<sup>2</sup> (Roy, 1949). This change in composition renders the muscovite susceptible to attack by hot, concentrated HCl. Muscovite so treated yields an insoluble residue that (1) shows a weight loss of about 50%, (2) gives no characteristic 10-angstrom  $\alpha$ -ray diffraction maximum, and (3) contains less than 10% of the potassium present in the untreated sample. Potassium feldspars are chemically unaffected by this treatment.

The procedure is useful in the following applications:

- (1) Separating potassium feldspar from dioctahedral mica.

<sup>1</sup> Equivalent spherical diameter.

<sup>2</sup> There is some evidence that dehydroxylation of muscovite is incomplete at 800° C. Higher temperatures were not used, however, because of the possible formation of other potassium-bearing phases.

- (2) Quantitatively determining potassium feldspar and dioctahedral mica when these are the only potassium-bearing minerals present.

#### PROCEDURE

The sample containing dioctahedral mica, potassium feldspar, and other non-potassium minerals is split into two portions. One portion is analyzed quantitatively for  $K_2O$ . At least 100 mg of the other portion is weighed, placed in a crucible, and heated to  $825^\circ C.$  for two hours. About one hour is required for the furnace to reach  $825 C.$ , therefore the total heating time is approximately three hours. After cooling, the sample is transferred to a beaker containing 100 to 200 ml of concentrated HCl. The mixture is boiled for two hours.<sup>1</sup> After cooling, the suspension is centrifuged and washed with water five times. Care must be taken to ensure quantitative retention of the residue during washing. The residue is dried at  $100^\circ C.$ , weighed, and analyzed for  $K_2O$ .

The per cent  $K_2O$  due to dioctahedral mica and the per cent  $K_2O$  due to potassium feldspar may be calculated according to the following expressions:

$$\text{Mica } K_2O = \frac{K_2O \times g - K_2O\Delta \times g\Delta}{g}$$

and

$$\text{K-Spar } K_2O = K_2O - \text{Mica } K_2O$$

where  $g$  is the weight of sample before treatment,  $g\Delta$  is the weight after treatment,  $K_2O$  is the  $K_2O$  content before treatment, and  $K_2O\Delta$  is the  $K_2O$  content after treatment.

To convert these  $K_2O$  values to per cent dioctahedral mica and per cent potassium feldspar, it is necessary to assume nominal values for the potassium contents of these minerals. Uncertainties will be associated with the selection of these values. However,  $x$ -ray diffraction studies of the micas and feldspars can provide information that will greatly reduce errors in the selection of nominal values for the potassium contents of these minerals. In any event,  $x$ -ray diffraction studies will be necessary to ascertain the absence of potassium-bearing phases other than dioctahedral mica and potassium feldspar.

#### EXPERIMENTAL RESULTS

The following experiment was designed to test the efficiency of the method in destroying dioctahedral mica while having potassium feldspar unaffected.

<sup>1</sup> The acid treatment in terms of duration, concentration, and amount of acid used could probably be varied without affecting the efficiency of the method. A severe treatment was used here to ensure complete destruction of the mica.

Crystals of microcline, quartz, and muscovite were ground<sup>1</sup> to a fine powder. The <10  $\mu$ ESD fraction of the muscovite was separated by settling in water. Two standards were prepared that contained respectively 50% quartz–50% muscovite and 50% quartz–50% feldspar. The <10  $\mu$ ESD fraction of the muscovite was used to prepare the quartz-muscovite standard. Quartz was added to serve as a carrier that would be inert under the conditions of the treatment. Portions of these two standards were analyzed quantitatively for K<sub>2</sub>O using an internally standardized x-ray fluorescence technique that has a coefficient of variability of  $\pm 2\%$ . Weighed portions of each of the standards were then treated according to the technique set forth above. After treatment, the two

TABLE 1. POTASSIUM CONTENTS OF STANDARDS BEFORE AND AFTER TREATMENT

Standard	%K <sub>2</sub> O	Weight Before Treatment	Weight After Treatment	%K <sub>2</sub> O After Treatment
Quartz-Feldspar	6.50	0.516g	0.501g	6.50
Quartz-Muscovite	5.15	0.517g	0.378g	0.45

standards were weighed and again analyzed for K<sub>2</sub>O. The data of the experiment are shown in Table 1.

An x-ray diffraction pattern was prepared from a randomly oriented aggregate of the treated quartz-muscovite standard. The pattern showed none of the characteristic muscovite diffraction effects. However, a weak, broad, symmetrical maximum at  $d = 10 \text{ \AA}$  was observed that extended from about  $d = 8 \text{ \AA}$  to  $d = 12 \text{ \AA}$ . This diffraction effect was taken to indicate the presence of small amounts of a badly distorted ten-angstrom structure.

The data of Table 1 show that the potassium content of potassium feldspar is unaffected by the treatment. Muscovite, on the other hand, shows a loss of more than 90% of its original potassium. Actually the muscovite loss was greater than indicated by the data of Table 1 because the muscovite-quartz mixture lost weight during the treatment. The following calculations provide a figure for the exact amount of muscovite destroyed:

The original mixture of 50% quartz and 50% muscovite contained 5.15% K<sub>2</sub>O, therefore the pure muscovite contained 10.30% K<sub>2</sub>O. The quartz-muscovite standard contained  $5.15 \times 0.517 / 100 = 0.0267\text{g K}_2\text{O}$ . After treatment, the same standard contained  $0.45 \times$

<sup>1</sup> Muscovite is very difficult to grind by normal methods. The authors found that the most satisfactory technique consists of filing the edge of a muscovite "book" with a metal file. The strokes should be parallel to the cleavage planes.

$0.378/100=0.0017\text{g K}_2\text{O}$ . The  $\text{K}_2\text{O}$  values can now be expressed in terms of grams of muscovite as follows:

$$\frac{0.0267\text{g}}{10.30\% \text{K}_2\text{O}} \times 100 = 0.259\text{g muscovite before treatment}$$

and

$$\frac{0.0017\text{g}}{10.30\% \text{K}_2\text{O}} \times 100 = 0.017\text{g muscovite after treatment}$$

Therefore  $(0.017/0.259) \times 100 = 6.6\%$  of the original muscovite was left after treatment.

These calculations show that treatment of a mixture of muscovite and feldspar would have resulted in a separation of better than 90%, inasmuch as the experimental data show that potassium feldspar was unaffected chemically.

#### CONCLUSIONS

The treatment described here is capable of selectively removing dioctahedral mica from admixture with potassium feldspar. The efficiency of separation is approximately 90%. The method can be used for the quantitative determination of dioctahedral micas and potassium feldspars provided that (1) accurate nominal potassium contents can be assumed for these minerals, and (2) these are the only potassium-bearing phases present.

#### ACKNOWLEDGMENTS

The authors gratefully acknowledge the support of the National Science Foundation.

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THE AMERICAN MINERALOGIST, VOL. 47, JULY-AUGUST, 1962

#### BROMYRITE NEAR EAGLE, COLORADO

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In June 1960, several samples of a soft silvery green mineral resembling cerargyrite were collected from fracture coatings in the breast of a short horizontal adit located on Horse Mountain about eight miles south of the town of Eagle, Eagle County, Colorado, in the Brush Creek Mining Dis-