A RARE-ALKALI BIOTITE FROM KINGS MOUNTAIN, NORTH CAROLINA¹

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Several years ago, after Judge Harry E. Way of Custer, South Dakota, had spectroscopically detected the rare-alkali metals in a deep-brown mica from a pegmatite containing pollucite and lithium minerals, in Tin Mountain, 7 miles west of Custer, another brown mica was collected, which had developed notably in mica schist at its contact with a similar mass of pegmatite about one half mile east of Tin Mountain. J. J. Fahey of the United States Geolgoical Survey analyzed the mica, and it proved to contain the rare-alkali metals⁴ and to be considerably different from any mica theretofore described. Although the cesium-bearing minerals before known (pollucite, lepidolite, and beryl) had come from the zone of highest temperature in the pegmatite, the brown mica was from the zone of lowest temperature.

The occurrence naturally suggested that where dark mica was found developed at the border of a pegmatite, especially one carrying lithium minerals, it should be examined for the rare-alkali metals. As had been found by Judge Way, spectroscopic tests on the biotite from Tin Mountain gave strong lithium and rubidium lines, and faint cesium lines. Lithium lines were shown in a biotite from the border of the Morefield pegmatite, a mile south of Winterham, Virginia, but rubidium and cesium were not detected. Similarly placed dark micas from Newry and Hodgeon Hill, near Buckfield, Maine, gave negative results. They should be retested. Tests by Dr. Charles E. White on a shiny dark mica from the Chestnut Flat pegmatite near Spruce Pine, North Carolina, gave strong lithium and weaker cesium lines. Biotite from the Corner Rock pegmatite near Craggy Gardens, North Carolina, was found by Morris Slavin of the Bureau of Mines to give strong lithium lines.

When opportunity was recently had to examine spodumene pegmatites enclosed in mica schists on the W. A. Ware farm, about $2\frac{1}{2}$ miles southwest of Kings Mountain, North Carolina, a lenticular mass of bright brownish mica 4 inches thick, 8 inches long, and 6 inches broad was found. It was on the surface close to the edge of the pegmatite. It is intergrown with pegmatite of border phase (See Fig. 1), and undoubtedly

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⁴ Hess, Frank L., and Fahey, J. J., Cesium biotite from Custer County, South Dakota: *Am. Mineral.*, vol. 17, pp. 173–176, 1932. came from the schist at the contact. None has been found at any place in the pegmatite. Keith and Sterrett⁵ noticed such segregations and said: "The reaction of wall rock and dike is also evident by the unusual coarseness of the mica schist of the wall rock in many places."

There is some white mica in the mass which is easily separated, and in this R. C. Wells, on testing with a pocket spectroscope, found no lithium or rare-alkali metal lines.



FIG. 1. Intergrowth of rare-alkali biotite with pegmatitite, apparently from edge of dike, Ware farm, Kings Mountain, N.C.

In sections under the microscope the brown mica is found to be remarkably pure, except for numerous microscopic prisms of apatite which pierce the mica plates. It does not enclose zircon and shows no halos as does the mica from the vicinty of Tin Mountain; and although both carry considerable titanium, in neither has rutile been found in the plates.

⁵ Keith, Arthur, and Sterrett, Douglas B. Gaffney, Kings Mountain, South Carolina-North Carolina, U. S. Geol. Survey Atlas No. 222, 1931, p. 11.

1041

The optical properties, determined by Miss Jewell J. Glass of the U. S. Geological Survey, are as follows:

Optically negative, acute bisectrix X is sensibly normal to the basal cleavage (001). The optical angle (2V) is very small, 0° to 5°. The indices of refraction are: $\alpha = 1.555$, $\beta = 1.589$, $\gamma = 1.590$. Color dark brown. Luster bronzy. Pleochroism along X, pale pinkish brown; Y and Z dark brown to dark reddish brown. Absorption strong, Y and Z>X.

The pegmatite with which the mica is associated is rather remarkable. Dark minerals are exceedingly scarce—even black tourmaline is rare. No massive segregations of mica, feldspar, or quartz; no graphic granite, and almost no beryl were found. The pegmatite was formed in several stages. A comparatively fine-grained mass of quartz and microcline, lean in muscovite, represents the first stage. It was roughly sheeted and long flat bodies of white microcline were introduced, free from perthitic structure, and as much as 2 or 3 feet thick. Sheeting took place again, the feldspar was broken, and later solutions replaced the corners with finegrained albite, quartz, muscovite, and spodumene. Spodumene seems to make up 15 or 20 per cent of much of the mass. Some cassiterite is present, mostly in greisenized parts of the pegmatite. Graton⁶ reported finding lithiophyllite and lepidolite, but, excepting the mica described here, spodumene is the only mineral carrying a rare-alkali metal that was found in several visits to the deposits. The pegmatites are from 3 to more than 100 feet broad, extend for $4\frac{1}{2}$ miles southwest of Kings Mountain and toward the northeast are known at a number of places between Kings Mountain and Lincolnton.7

CHEMICAL ANALYSIS

As the analysis of the mica involved the use of some new methods, a brief outline of the analytical procedure seems desirable.

Silica and fluorine in a gram sample were separated as described by Hoffman and Lundell,⁸ involving fusion with sodium carbonate, leaching of the melt with water, and the separation of the silica by two precipitations with zinc oxide. Two fusions and leachings were made to insure complete removal of fluorine. Silica was determined on the combined residues and zinc precipitates by the usual method.

⁶ Graton, Louis C., Reconnaissance of some gold and tin deposits of the southern Appalachians: U. S. Geol. Survey, Bull. 293, p. 38, 1906.

⁷ For a more detailed description of the pegmatites see: Hess, Frank L., Lithium in North Carolina: *Eng. and Min. Jour.*, vol. 137, pp. 339-342, July 1936.

⁸ Hoffman, J. I., and Lundell, G. E. F., Determination of fluorine and of silica in glasses and enamels containing fluorine: *Bur. Standards Jour. Research*, vol. 3, p. 581, 1929. The fluorine was determined nephelometrically. The extracted fluoride solution, free from silica, was made up to a definite volume with a sodium chloride concentration of 55 grams per liter. In a 25 milliliter aliquot of this solution the fluorine was precipitated as colloidal calcium fluoride, the solubility being reduced by alcohol, and gelatin serving as a stabilizer. A standard of known fluoride content was precipitated at the same time and the two opalescent solutions compared in a nephelometer to give a preliminary figure for fluorine, and from this the unknown and standard were adjusted to within 5 per cent of equality and new aliquots precipitated to obtain a final reading. The nephelometric procedure for fluorine has been shown⁹ to be accurate and dependable.

The rare alkalies were determined by methods described by Wells and Stevens.¹⁰ The alkalies in a 0.5 gram sample were extracted by the J. Lawrence Smith procedure, and potassium, rubidium, and cesium separated as chloroplatinates insoluble in 80 per cent alcohol. From the soluble portion, containing sodium and lithium, platinum was removed with formic acid and sodium and lithium separated by the Palkin method,¹¹ based on the insolubility of sodium chloride in an alcohol-ether solution. The sodium and lithium were then separately weighed as sulphates.

The insoluble chloroplatinates of potassium, rubidium, and cesium were treated with formic acid to remove platinum, the resulting chlorides evaporated to dryness and a number of extractions made with alcohol saturated with hydrogen chloride (10 ml. solvent used) to remove all of the rubidium and cesium. In the first two extracts cesium was removed by precipitating potassium and rubidium with an alcoholic ammonium sulphate reagent. The cesium was then weighted as sulphate and the rubidium in the first two alcohol-hydrochloric acid extracts obtained by difference. From the additional extracts rubidium was obtained by converting to chloroplatinates and extracting with 15 per cent alcohol, the potassium salt being soluble.

This description gives the method of analysis in brief. Details of procedure may be found in the articles cited.

Results of the analysis of the brown mica from Kings Mountain and a corrected analysis of the biotite from the edge of the pegmatite near Tin Mountain, South Dakota, are given below.

⁹ Stevens, Rollin E., Nephelometric determination of fluorine: *Ind. Eng. Chem., Anal. Ed.*, vol. 8, pp. 248–252, 1936.

¹⁰ Wells, Roger C., and Stevens, Rollin E., Determination of the common and rare alkalies in mineral analysis: *Ind. Eng. Chem.*, Anal. Ed., vol. 6, pp. 439–442, 1934.

¹¹ Palkin, S., Separation of lithium from the other alkali metals: *Jour. Am. Chem. Soc.*, vol. **38**, p. 2326, 1916.

1043

		1.	2.
	SiO ₂	42.02	39.97
	Al_2O_3	18.75	17.51
	Cr_2O_3		none
	Fe_2O_3	0.66	2.26
	FeO	8.29	14.81
	MnO	0.27	0.22
	MgO	9.55	8.45
	CaO	0.93	none
	Li_2O	1.20	0.65
	Na_2O	0.73	0.45
	$K_{2}O$	8.54	8.48
	Rb_2O	1.85	1.48
	Cs_2O	0.47	1.12
	$H_{2}O - 110^{\circ}C.$	0.16	0.32
	H ₂ O+110°C.	2.44	2.48
	TiO ₂	1.35	2.64
	F	4.34	3.17
		101.55	101.01
	Less $O = F_2$	1,83	1.34
	8	99.72	99.67

ANALYSES OF BIOTITE

1. From Kings Mt., N.C., analysis by R. E. Stevens.

2. From near Tin Mt., S. D., analysis by J. J. Fahey with redeterminations of lithium, rubidium, and cesium by R. C. Wells and R. E. Stevens.

The two micas have a very similar composition. The most marked differences are in the iron and silica. The mica from the light-colored pegmatite of North Carolina, as might perhaps be expected, carries much less iron than that from the more highly colored rocks of the Black Hills, and it makes up the difference in silica. The alkali oxides amount to 12.79 per cent in the Kings Mountain mica, and to 12.18 per cent in the Custer mica, a ratio of 1.05 to 1; the molecular ratio rises to 1.18 to 1, owing to the large quantities of the lighter alkali metals in the southern mica. Each carries less than half the magnesia of an ordinary phlogopite; the iron, even in the South Dakota mica, is low for lepidomelane and both seem to fit best among the biotites. Rare-alkali micas of this kind seem generally to have a high fluorine content, and conversely those low in fluorine do not contain rare alkalies. A black biotite of similar occurrence, collected by W. T. Schaller at Ridgway, Va., contained only 1.40 per cent fluorine and no rare alkalies.