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BARYLITE FROM SEAL LAKE, LABRADOR



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MINERAL SCIENCES DIVISION

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BARYLITE FROM SEAL LAKE, LABRADOR

by

E. H. Nickel* and D. J. Charette*

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SUMMARY OF RESULTS

The occurrence of barylite, the principal beryllium mineral in ore samples from the Seal Lake, Labrador deposit of Rio Tinto Canadian Exploration Limited, is described. A chemical analysis of the barylite shows that it contains 15.00% BeO, and that its composition conforms fairly closely to the theoretical formula BaBe₂Si₂O₇. A selective staining method employing hydrofluoric acid and sodium or potassium rhodizonate has been developed for the barylite.

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INTRODUCTION

In August, 1960, officials of Rio Tinto Canadian Exploration Limited, Toronto, Ontario, provided on request, a suite of samples from the company's beryllium deposit at Seal Lake, Labrador. The samples were selected by R. A. Dujardin, company geologist, with a view to providing a representative selection of material for mineralogical study. A preliminary report on the mineralogy of the samples was issued early in 1961 (1) and, since that time, the mineralogical investigation has been continued.

In the preliminary report it was noted that barylite was the only beryllium mineral recognized at the time. Since then eudidymite has been discovered in the ore, and it appears likely that other beryllium minerals may be found. Indications are, however, that barylite greatly predominates in quantity over all other beryllium minerals in the material examined. Because of its importance, efforts were made to obtain a reliable chemical analysis for the mineral and, in addition, to develop a diagnostic stain which would be specific for barylite in this deposit.

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As this phase of the work was nearing completion, it came to the attention of the first author that a paper on the barylite from the Seal Lake deposit had been submitted to The American Mineralogist by Heinrich and Deane (2). On reading a draft copy of this paper kindly provided by Dr. Heinrich, it was evident that some of the results of our investigation supplemented those of Heinrich and Deane. It was consequently decided to publish our information in the same issue of The American Mineralogist, if possible. To this end, a paper (3) was prepared, and submitted to the editor of that journal. The paper consisted essentially of the portions of this report entitled "Chemical Analysis" and "Staining Method".

The chemical analyses were made by one of the authors (D.J.C.), while the other author (E.H.N.) is responsible for the remainder of this report.

OCCURRENCE

Most of the samples consist of syenitic gneiss composed predominantly of white albite and black sodiumiron amphibole. A number of accessory minerals have been found, some of which are very rare, including some that have so far defied identification. These will be discussed in a later report. The barylite generally occurs as relatively small disseminated grains (Figures 1 and 2), although in one of the samples examined (TR-16) the barylite is particularly abundant and comprises a significant proportion of the sample. In this sample the barylite varies from disseminated prismatic grains to massive aggregates (Figure 3), and appears to correspond to the "vein barylite" described by Heinrich and Deane (2).

The barylite for analysis was concentrated from Sample No. TR-16 because of its abundance in this sample. This barylite is light pink in contrast to the white barylite in the other samples. However, since the X-ray powder diffraction patterns show no discernable differences and the optical properties of the barylite from TR-16 are similar to those of the barylite in the other samples, it may be inferred that their chemical compositions are similar, and that the pink colour of the analyzed barylite is in all likelihood due to the presence of a minor impurity, possibly iron.

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Figure 1 - Colour photomicrograph of thin section stained with potassium rhodizonate. Feldspar is white; amphibole is black; barylite is stained brickred.

Ultropak illumination. Magnification 40X.



Figure 2 - Colour Photomicrograph of -35+48 Mesh crushed sample stained with potassium rhodizonate, showing composite nature of the grains at this size. Feldspar is white; amphibole is black; barylite is stained brick-red. Ultropak illumination. Magnification 40X.



Figure 3 - Photomicrograph of thin section of Sample TR-16, stained with potassium rhodizonate. The barylite grains, which are actually stained brick-red, appear in the photomicrograph as shades of grey. The disseminated barylite exhibits prismatic development, whereas some of the barylite in the massive portion is more equidimensional. The white areas are quartz.

Transmitted illumination. Magnification 65X.

X-RAY DIFFRACTION ANALYSIS

The X-ray powder diffraction data for the analyzed barylite are shown in Table 1.

The powder pattern was obtained from a 114.6 mm Debye-Scherrer camera, using filtered cobalt radiation. Intensities were estimated visually. Our data are in good agreement with those of Heinrich and Deane (personal communication) on their sample of barylite from Seal Lake and on a barylite sample from Langban, Sweden.

TABLE 1

			· · · · · · · · · · · · · · · · · · ·
Int. (Est.)	d(A)	Int. (Est.)	d(A)
7	5.84	2	2.08
3	4.52	2	2.03
3	4.32	1	1.948
9	3.38	2	1.892
4	3.25	4	1.851
8	3.05	2	1.739
8	2.98	1	1.705
10	2.92	6	1.687
1	2.61	1	1.671
4	2.53	2	1.621
4	2.45	2	1,588
2	2.40	1	1,566
5	2.34	4	1,527
2	2.26	2	1.490
6	2.21	1	1.459
4	2,17	1	1.445
2	2.13	2	1.436
1	2.11		

X-ray Powder Diffraction Data Barvlite

CHEMICAL ANALYSIS

The barylite was concentrated from sample No. TR-16 by pulverizing and centrifuging repeatedly in Clerici solution diluted to a specific gravity of 3.90, which is slightly below that of the barylite. The lighter minerals, principally quartz, floated in this liquid, leaving a heavy concentrate consisting of nearly pure barylite.

The chemical analysis of the barylite is shown in Table 2. When this analysis is compared with the theoretical composition of pure barylite $(BaBe_2Si_2O_7)$ it is seen that the analyzed sample contains a small excess of SiO₂ and a deficiency of BeO and BaO. This can be explained by the presence of about 2 per cent quartz in the analyzed barylite.

TABLE 2

	Barylite,	BaBe2Si207
	Seal Lake, Labrador	(Carc.)
BeO	15.00%	15.46%
BaO	46.12	47.40
SiO2	38,52	37,14
Fe ₂ 03	0.08	hand Mall
A1 ₂ 0 ₃	0.04	Bagin Rank
CaO*	0.15	940 SVD
н20	0.11	odas tran
	100.02	100.00

Chemical Analysis of Barylite

*Includes minor amount MgO.

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The analysis recalculated to molecular proportions is shown in Table 3. This also shows the deficiency of BeO and BaO and the excess of SiO_2 with respect to pure $BaBe_2Si_2O_7$. However, the mole ratio BeO:BaO is 1.93:0.97 = 2:1, which corresponds to that of pure barylite.

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The specific gravity of the barylite, determined by pycnometer, is 3.96. This is in good agreement with the calculated value of 3.98, based on the chemical analysis and accepting the unit cell parameters given by J. C. Smith (4). The calculated specific gravity of pure $BaBe_2Si_2O_7$ is 4.00.

STAINING METHOD

Since it is very difficult to distinguish barylite from the other white minerals in the rock, an attempt was made to find a suitable diagnostic stain for barylite. A number of reagents were investigated, particularly those likely to be specific for beryllium or barium. The most successful reagents were found to be sodium rhodizonate and potassium rhodizonate, both of which react with barium. The use of sodium rhodizonate as a spot test for barium has been described by Feigl and Suter (5), and Feigl (6). In staining barylite, the potassium rhodizonate was found to be equally as effective as the corresponding sodium salt.

ΤA	BI	Æ	3
ın	D1	-11-	J

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Oxide	Analyzed Wt. %	Mol. Wt.	Mol. Props.	Mol. Props. x 3.2233*	$\frac{\text{Theoretical}}{\text{BaBe}_2\text{Si}_2\text{O}_7}$
BeO	15.00	25.01	0.5998	1,93	2.00
BaO	46.12	153.36	0.3007	0.97	1.00
SiO ₂	38,52	60.09	0.6410	2.07	2.00
Fe ₂ 03	0.08	159.70	0.0005)		
A1203	0.04	101.96	0.0004	0,01	
CaO	0,15	56.08	0.0027)		
Н ₂ О	0.11	18.02	0.0061	0,02	
	100.02		1,5512	5.00	5.00

Molecular Proportions in Barylite

*This factor brings the total mol. props. to 5,00,

1 9 1 The staining method involves a two-step procedure--etching followed by staining. The details are as follows:

Reagents

Etching Reagent: Concentrated hydrofluoric acid (HF), 48% solution.

Staining Reagent: 0.2% aqueous solution of sodium rhodizonate or potassium rhodizonate. The solution of either salt is prepared with cold water shortly before use, as it deteriorates rather rapidly. In practice, a solution prepared in the morning may be used for the remainder of the day, but is ineffective the following day.

Procedure

Rock slabs and thin sections: Suspend sample over hot fuming HF for 1 minute; rinse with water; immerse in staining solution for 2 to 5 minutes; rinse with water; dry, if necessary, but without heating.

<u>Crushed samples</u>: Immerse the sample in cold HF in a platinum or "Teflon" crucible for 1 to 5 minutes, depending on particle size (excessive etching will decompose finely pulverized barylite completely); decant HF and rinse with water; apply a staining solution for 2 to 5 minutes; decant and rinse with water; dry, if necessary, but without heating. - 11 -

Results

If the above procedure is followed, the barylite is selectively stained to a brick-red colour (Figures 1 and 2). The contacts between the barylite grains and the other minerals are sharply defined, and there is no appreciable tendency for the colour to run. At room temperature the colour persists for a long time, although over a period of months it becomes somewhat lighter in shade. In stained sections examined after six months, the colour was found to have changed from its original bright brick-red to a reddish-orange shade. Heating, even to relatively moderate temperatures, causes the stain to change to a dark chocolate-brown, so that heating should be avoided when drying the stained sample.

Some grains of barylite stain more deeply than others, even within the same sample (see Figure 3). This is apparently due to variations in the degree of attack by the hydrofluoric acid, which, in turn, appears to depend on such factors as surface preparation, degree of fracturing or cleavage, and crystallographic orientation.

In general, since the staining reaction depends on the presence of barium ions, other barium-bearing minerals, if present, might also be expected to be stained. The staining procedure was tried on barite, one of the most common barium minerals. It resisted staining, however, because of its insolubility in HF. With respect to interference by other elements, Feigl (6) notes that "the rhodizonate test is recommended only for the detection of barium...when bivalent heavy metals are known to be absent" (p. 216). Feigl and Suter (5), in their investigations of the effects of sodium rhodizonate on a number of elements, showed that in a neutral solution both barium and strontium react to form a red-brown colour, and that a few other ions (mercury, cadmium, bismuth and copper) produce other shades of red. Poluektov (7) reported that in a weakly acid medium the rare earth elements react with potassium and sodium rhodizonates to form a red-violet colour. Therefore, in applying this staining method to barylite the possibility of these interferences must be considered.

In the Seal Lake beryllium-bearing assemblage, however, the only mineral, other than barylite, affected by the stain appears to be a metamict rare-earth mineral that has not yet been fully identified. It changes from its original tan colour to a darker brown, a colour that is quite distinct from that of the stained barylite. The barylite appears to be the only mineral in the assemblage that produces the characteristic brick-red stain when the described procedure is followed. It may be concluded, therefore, that the barylite in the Seal Lake mineral assemblage is selectively stained by this procedure.

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The authors are grateful to officials of Rio Tinto Canadian Exploration Limited for providing the samples for this investigation and for arranging discussions with members of their geological staff. Dr. A. A. Levinson of Gulf Research and Development Company, formerly with the Texas Division of the Dow Chemical Company, merits our appreciation for discussing the staining method with us and drawing our attention to the papers by Poluektov (7) and Heinrich and Deane (2). The authors are also grateful to Dr. E. Wm. Heinrich and Mr. R. W. Deane for providing a copy of their manuscript in advance of its publication.

Dr. J. A. Soles of the Mineral Processing Division of the Mines Branch made the colour photomicrographs, prints of which are included in this report. Miss Margaret Jost, chemistry student at the University of Ottawa assisted in developing the staining method during her period of employment as student assistant at the Mines Branch in the summer of 1961.

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