

## A LITHIOPHOSPHATE OCCURRENCE IN NORTH CAROLINA

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Lithiophosphate,  $\text{Li}_3\text{PO}_4$ , was first described by Matias and Bondareva (1957) in masses up to  $5 \times 9$  cm occurring as a hydrothermal replacement of montebrasite in a Kola pegmatite, USSR. Fisher (1958) made an X-ray diffraction powder pattern of synthetic  $\text{Li}_3\text{PO}_4$  as early as 1950 in anticipation of finding the material in pegmatites, but Matias and Bondareva were the first to report such an occurrence.

The Foote Mineral Company spodumene mine at Kings Mountain, Cleveland County, North Carolina is the site of the second occurrence of lithiophosphate. All of the specimens examined by the author were collected by Mr. Jack Eaker of Kings Mountain who generously supplied the samples used in this study. The Foote mine is rapidly becoming one of the most interesting mineral localities in the United States. More than 80 species have been identified by the author in the last three years. Included are several new minerals, some half-dozen species which formerly were one-locality minerals, and very fine crystals of such uncommon species of fairfieldite, eucryptite, bikitaite, roscherite, eosphorite, milarite and lithiophilite.

Lithiophosphate occurs as colorless crystals more than 1 cm in length and in colorless to white to buff-colored masses exceeding 2 cm in one dimension. It is found on quartz and albite crystal druses which line open fissures in massive albite-microcline-quartz-spodumene-muscovite pegmatite, suggesting a late origin. One mass of lithiophosphate is in contact with rhodochrosite but the relative age of the two minerals is not clear. Rhodochrosite appears to be one of the latest phases to form at the Foote mine.

The crystals are limpid and their faces well-developed. They are elongated parallel to  $b$  and superficially resemble topaz in habit. Observed forms are  $\{110\}$ ,  $\{101\}$ ,  $\{010\}$ ,  $\{013\}$ , and  $\{001\}$ .  $\{110\}$  and  $\{101\}$  are the most prominently developed forms. Lithiophosphate has perfect  $\{010\}$  cleavage. A distinct  $\{110\}$  cleavage or parting generates a diamond-shaped cross-hatching through the (001) plane and leads to the breaking out of pointed chips along the sharp edges, producing a saw-tooth effect. No striations were observed on the crystals but etching is present on some.

The optical properties and specific gravity of lithiophosphate from the Foote mine are given in Table 1 and compared with those of the Kola material and synthetic  $\text{Li}_3\text{PO}_4$  (Swanson *et al.*, 1965).

TABLE 1. LITHIOPHOSPHASE OPTICAL PROPERTIES AND SPECIFIC GRAVITY

	(1) Foote mine, USA	(2) Kola, USSR	(3) Synthetic $\text{Li}_3\text{PO}_4$
$\alpha$	1.553(1)	1.550(2)	1.550
$\beta$	1.558(1)	1.557(2)	n.d.
$\gamma$	1.566(1)	1.567(2)	1.538 <sup>b</sup>
$2V$	large, $\sim 80^\circ$	$69^\circ$	large
Sign	(+)	(+)	(-) <sup>b</sup>
Orientation $Y = a, Z = b$		—	—
$\rho$	2.478 <sup>a</sup>	2.46	2.479(calc)

<sup>a</sup> Berman balance, this study.

<sup>b</sup> Highly suspect due to poor quality of the crystal; H. E. Swanson, pers. commun. (1969).

(1) Optics by Peter B. Leavens, Na light at  $27^\circ\text{C}$ ., pers. commun. (1968).

(2) Matias and Bondareva (1957).

(3) Swanson *et al.* (1965).

A wet chemical analysis was not performed because semiquantitative spectrochemical analysis showed only Li and P as major constituents. Ti and Si are the most abundant trace constituents in concentrations of  $0.05 \pm 0.025$  and  $0.02 \pm 0.01$  percent respectively. The sum of all the trace elements analysed for is less than 0.1 percent indicating that the mineral is near 99.9 percent  $\text{Li}_3\text{PO}_4$ .

Power diffraction data are not presented herein because they match perfectly those of synthetic  $\text{Li}_3\text{PO}_4$  (low form) as reported by Swanson *et al.* (1965). Swanson *et al.* (1964) described a high form of  $\text{Li}_3\text{PO}_4$  which was produced by heating the low form to  $800^\circ\text{C}$  for 15 minutes. Crystals of lithiophosphate from the Foote mine treated similarly underwent a transformation to a phase which, on the basis of the powder pattern, is identical to the high form of synthetic lithiophosphate. The cell parameters of synthetic  $\text{Li}_3\text{PO}_4$  published by Zambonini and Laves (1932) are those of the high form. The structure of the high form was determined by Zemann (1960). Zambonini and Malossi (1931) reported a specific gravity of 2.456 for synthetic high  $\text{Li}_3\text{PO}_4$  and Zemann (1960) and Swanson *et al.* (1964) gave calculated densities from X-ray measurements of 2.42 and 2.439  $\text{g}/\text{cm}^3$  respectively. The failure of early workers to recognize the existence of these polymorphs will lead to unfortunate confusion in referencing.

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THE AMERICAN MINERALOGIST, VOL. 54, SEPTEMBER–OCTOBER, 1969

DIFFERENTIAL THERMAL ANALYSIS OF HIGH-  
ALUMINA ALLOPHANE

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ABSTRACT

A differential thermalgram of high-alumina allophane ( $\text{SiO}_2 \cdot 2\text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O}$ ) shows an endothermal peak at  $90^\circ\text{C}$  representing loss of water; there is no evidence of  $\text{OH}^-$ . Amounts of fluxing ions were apparently insufficient to suppress a high-temperature exothermal reaction at  $990^\circ\text{C}$ , tentatively ascribed to formation of an Al-Si spinel.

Since publication of the article on high-alumina allophane (Snetsinger, 1967), several inquiries have been received regarding availability of differential thermal analytical data on this material. A thermalgram has subsequently been obtained, done on a scant milligram of pure allophane using a microholder unit manufactured by the Robert L. Stone Company (Tracor Instruments) of Austin, Texas; the sample was run in a combined cup-and-differential thermocouple. Referring to Figure 1, the strong endothermal peak at about  $90^\circ\text{C}$  probably represents loss of absorbed water, while the small exothermal effect at  $340^\circ \text{ca.}$  may be due to (1) oxidation of a very small amount of organic material or (2) oxidation of a trace of ferrous iron. Irregularities in the baseline occur above  $400^\circ\text{C}$ ; these are due to electronic noise. The exothermal peak at  $990^\circ\text{C}$  is tentatively ascribed to formation of an Al-Si spinel. Amounts of ferrous iron and organic material in the high-alumina allophane are insufficient to have suppressed any dehydroxylation endotherm in the  $330^\circ\text{C}$  range, and no other dehydroxylation peaks occur. This is in agreement with the lack of infrared evidence for presence of hydroxyl (Snetsinger, 1967).

Presence or absence of an exothermal peak above  $900^\circ\text{C}$  in allophane has been of interest to several investigators. Fieldes (1955) classified as "allophane A" ones that yielded exothermal peaks near  $900^\circ\text{C}$ , whereas his "allophane B" lacked such peaks. Campbell *et al.* (1968) found that the height of high-temperature exothermal peaks of allophanes increases as particle size of allophane decreases; organic matter apparently en-