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Literatur

BERGMANN, E. D. (1968). *Chem. Rev.* **68**, 41.
GOMPPER, R. & KUTTER, E. (1965). *Chem. Ber.* **98**, 2825.

HAFNER, K., SCHULZ, G. & WAGNER, K. (1964). *Liebigs Ann.* **678**, 39.
HARTKE, K. (1964). *Tetrahedron Letters*, **38**, 2737.
HARTKE, K., SCHMIDT, E., CASTILLO, M. & BARTULIN, J. (1966). *Chem. Ber.* **99**, 3268.
NORMAN, N. & POST, B. (1961). *Acta Cryst.* **14**, 503.
SALAMON, G. (1969). Dissertation Marburg.

Acta Cryst. (1969). **B25**, 993

On the crystal structure of leucophanite. By E. CANNILLO, G. GIUSEPPETTI and V. TAZZOLI, *Centro di Cristallografia del C.N.R., Sez. VI, Istituto di Mineralogia dell'Università di Pavia, Italy*

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The space group and the structure parameters of leucophanite given in a previous paper are confirmed.

In a previous paper (Cannillo, Giuseppetti & Tazzoli, 1967) the space group $P2_12_12_1$ was assumed for the determination of the crystal structure of leucophanite. Some features of the measured intensities led to the conclusion that the true space group would have been $P1$.

In order to resolve the question, the collection of the intensities has been repeated and extended to one half of the reciprocal sphere for Cu $K\alpha$ radiation. The refinement with the least-squares method has been continued in the space group $P1$.

However, the atomic shifts from orthorhombic symmetry were not significant and the variations in the thermal parameters did not suggest another kind of order for the calcium and sodium atoms.

At this stage the suspicion arose that some double reflexion effects were responsible for the 'anomalies' in the

measured intensities. Precession pictures taken with Mo $K\alpha$ radiation did not show the previously observed 'anomalies', thus confirming the suspicion; in particular, the three reflexions $h00$ (h odd), inconsistent with the orthorhombic space group, did not appear and the sensible differences between some intensities which would be equivalent in the orthorhombic space group, were not now observed.

Thus the space group of leucophanite is, without any doubt, $P2_12_12_1$, and the structure parameters published in the cited paper are substantially confirmed.

Reference

CANNILLO, E., GIUSEPPETTI, G. & TAZZOLI, V. (1967). *Acta Cryst.* **23**, 255.

Acta Cryst. (1969). **B25**, 993

A comment on the synthesis and lattice constants of transition metal thionibates with berthierite (FeSb_2S_4) structure. By B. VAN LAAR, *Reactor Centrum Nederland, Petten, The Netherlands* and D.J.W. IJDO, *Laboratory of Inorganic Chemistry, University of Leiden, The Netherlands*

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Transition metal thionibates ($M\text{Nb}_2\text{S}_4$ where $M = \text{Mn, Fe, Co, Ni}$ or Cu) have been indexed on the basis of a hexagonal unit cell. The results are much more satisfactory than those from earlier indexing on the basis of an orthorhombic unit cell.

Eibschütz, Hermon & Shtrikman (1967) describe the preparation and X-ray patterns of the compounds $M\text{Nb}_2\text{S}_4$ ($M = \text{Mn, Fe, Co, Ni, and Cu}$). The X-ray powder photographs of these compounds were indexed on the basis of an orthorhombic unit cell having about the same lattice constants as those published for berthierite (FeSb_2S_4) by Buerger & Hahn (1955). Eibschütz *et al.* consider this as evidence that the compounds $M\text{Nb}_2\text{S}_4$ are isostructural with FeSb_2S_4 .

These conclusions do not seem to be completely justified. If $M\text{Nb}_2\text{S}_4$ is isostructural with FeSb_2S_4 , it would be expected that their X-ray patterns should be very similar, because the scattering powers of Nb and Sb differ by only twenty per cent. Comparing the data of Eibschütz *et al.* with the diagram of FeSb_2S_4 published by Buerger (1936), it is easily seen that this is not at all the case.

This discrepancy can be resolved by indexing the published data on the basis of hexagonal unit cells. The lattice