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Standard X-ray Diffraction Powder Patterns

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National Bureau of Standards Monograph 25 — Section 4

Issued June 28, 1966

For sale by the Superintendent of Documents, U.S. Government Printing Office
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Lithium Phosphate, low form (lithiophosphate), Li_3PO_4 (orthorhombic) revised

This compound was described in earlier work [1] from this laboratory. Recently the structure of the low form was determined by Keffer, Mighell, Mauer, Swanson, and Block [2]. The following pattern is intended to replace the earlier one in Monograph 25, Section 3.

Powder data card. No. 12–230, Fisher [3] 1958.

Additional published patterns. Matias and Bondareva [4] 1957 and National Bureau of Standards [1] 1964.

NBS sample. The sample of lithium phosphate was obtained from the City Chemical Co., New York, N.Y. Spectrographic analysis showed the following major impurities: 0.1 to 1.0 percent calcium; 0.01 to 0.1 percent each of aluminum, sodium, and strontium; and 0.001 to 0.01 percent each of barium, iron, magnesium, and silicon.

The sample was colorless. Two indices of refraction are $N_\alpha=1.550$ and $N_\gamma=1.538$. It is optically negative with a large 2V. N_β could not be determined because of the crystal shape.

The d-values of the three strongest lines are 3.973, 3.797, and 2.640 Å.

Structural data. Keffer, Swanson, Mighell, Mauer, and Block [2] in 1964 determined that low form lithium phosphate has the space group C_{2v}^7–$Pmn2_1$ (No. 31) and $2(Li_3PO_4)$ per unit cell.

hkl	Internal Standard Ag, $a=4.0861$ Å; temp. 25 °C Cu λ 1.5405 Å	
	d	*I*
	Å	
010	5. 232	34
110	3. 973	100
101	3. 797	98
011	3. 554	56
111	3. 071	26
200	3. 059	
210	2. 640	64
020	2. 616	36
002	2. 423	47
211	2. 318	31
021	2. 303	
012	2. 199	<1
121	2. 155	4
112	2. 070	3
310, 202	1. 899	1
301	1. 879	2
221	1. 839	4
212	1. 785	14
311	1. 769	9
122	1. 7074	1
130	1. 6777	2
031	1. 6415	1
320	1. 6083	3
131	1. 5855	2
103	1. 5616	4
013	1. 5431	3
400	1. 5287	11
230	1. 5152	16
113	1. 4959	1
410	1. 4675	1
411	1. 4043	2
213	1. 3776	14
123	1. 3409	2
420	1. 3203	2
040	1. 3078	<1
402	1. 2931	4
232	1. 2848	8
331	1. 2788	4
303	1. 2656	2
412	1. 2550	1
223	1. 2533	1
240	1. 2027	<1
510	1. 1909	2
501	1. 1860	2
241	1. 1676	3
422	1. 1591	2
430	1. 1498	<1
323	1. 1394	<1
340	1. 1014	2

References

[1] H. E. Swanson, M. C. Morris, E. H. Evans, and L. Ulmer, Standard X-ray Diffraction Powder Patterns, NBS Mono. 25, **Sec. 3,** 38 (1964).

[2] C. Keffer, A. Mighell, F. Mauer, H. Swanson, and S. Block, The crystal structure of low temperature lithium phosphate (to be published in Inorg. Chem.).

[3] D. J. Fisher, Note on lithiophosphate, Am. Mineralogist **43,** 761–2 (1958).

[4] V. V. Matias and A. M. Bondareva, Lithiophosphate, a new mineral, Dokl. Akad. Nauk SSSR 112, 124–6 (1957); an English abstract exists in Am. Mineralogist **42,** 585 (1957).

[5] F. Zambonini and F. Laves, Über die Kristallstruktur des Li_3PO_4 und seine Beziehung Zum Strukturtyp des Olivin, Z. Krist. **83,** 26–28 (1932).

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Lattice Constants

		a	b	c
		Å	Å	Å
	Zambonini and Laves [5]	6. 08	10. 28	4. 87
1932	National Bureau of Standards,			
1965	sample at 25 °C	6. 1155 ±0. 0004	5. 2340 ±0. 0005	4. 8452 ±0. 0005

The density of lithium phosphate, low form, calculated from the NBS lattice constants is 2.479 g/cm³ at 25 °C.

Lithium Sulfate Monohydrate, $Li_2SO_4 \cdot H_2O$ (monoclinic)

Powder data card. No. 1–0425, Hanawalt, Rinn, and Frevel [1] 1938.

Additional published patterns. None.

NBS sample. The sample of lithium sulfate monohydrate was prepared at NBS from lithium metal and sulfuric acid, using enough heat to fume off the excess acid. Spectrographic analysis showed the following major impurities: 0.01 to 0.1 percent each of silver, calcium, sodium, and tungsten; and 0.001 to 0.01 percent each of aluminum, barium, iron, potassium, magnesium, silicon, and strontium.

The sample was colorless and optically negative. The indices of refraction are N_α=1.460, N_β= 1.477, and N_γ=1.487; 2V is large.

The *d*-values of the three strongest lines are 5.084, 4.133, and 3.559 Å.

Structural data. Ziegler [2] in 1934 determined that lithium sulfate monohydrate has the space group C_2^2—$P2_1$ (No. 4) with $2(Li_2SO_4 \cdot H_2O)$ per unit cell.

Lattice Constants

		a	b	c	β
		Å	Å	Å	
1934	Ziegler [2]	5. 44	4. 84	8. 16	107°35′
1952	Bechmann [3]	5. 45	4. 87	8. 18	107°18′
1954	Larson and Helmholz [4]	5. 430	4. 836	8. 140	107°14′
1965	National Bureau of Standards, sample at 25 °C	5. 4518 ±0. 0005	4. 8707 ±0. 0004	8. 175 ±0. 001	107°19. 8′ ±0. 4′

The density of lithium sulfate monohydrate calculated from the NBS lattice constants is 1.939 g/cm³ at 25° C.

References

[1] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by x-ray diffraction, Anal. Chem. **10,** 457–512 (1938).

[2] G. E. Ziegler, The crystal structure of lithium sulphate monohydrate, $Li_2SO_4 \cdot H_2O$, Z. Krist. **89,** 456–459 (1934).

[3] R. Bechmann, Elastic and piezoelectric coefficients of lithium sulphate monohydrate, Proc. Phys. Soc. **65B,** 375–77 (1952).

[4] A. C. Larson and L. Helmholz, Redetermination of the crystal structure of lithium sulfate monohydrate, $Li_2SO_4 \cdot H_2O$, J. Chem. Phys. **22,** 2049–2050 (1954).