Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Lithium samarium polyphosphate, $LiSm(PO_3)_4$

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Received 19 October 2009; accepted 30 December 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(Sm–O) = 0.003 Å; R factor = 0.019; wR factor = 0.048; data-to-parameter ratio = 10.2.

The mixed-metal rare-earth polyphosphate $LiSm(PO_3)_4$ consists of a three-dimensional framework in which zigzag $[(PO_3)_n]^{n-}$ chains with a periodicity of four PO_4 tetrahedra are connected through Li^+ and Sm^{3+} ions (both with 2. symmetry).

Related literature

For the structures, properties and applications of condensed alkaline metal–rare earth polyphosphates with the general formula $MLn(PO_3)_4$ (M = alkali metal, Ln = rare earth metal), see: Ferid *et al.* (1984); Ettis *et al.* (2003); Parreu *et al.* (2007); Zhu *et al.* (2007); Ben Zarkouna *et al.* (2007).

Experimental

Crystal data

$LiSm(PO_3)_4$
M_r = 473.17
Monoclinic, $C2/c$
a = 16.379 (2) Å
b = 7.0499 (9) Å
c = 9.6936 (12) Å
β = 126.138 (2)°
V = 903.96 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.27 mm^{-1}
T = 298 K
0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997) T_{min} = 0.439, T_{max} = 1.000
2405 measured reflections
854 independent reflections
843 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.048
S = 1.09
854 reflections
84 parameters
$\Delta\rho_{max}$ = 1.02 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2086).

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supplementary materials

Acta Cryst. (2010). E**66**, i3 [**doi:10.1107/S1600536809055822**]

Lithium samarium polyphosphate, $LiSm(PO_3)_4$

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Comment

Interest in alkali-metal rare-earth polyphosphates stems from their physical properties, such as high luminescence efficiency (Ettis *et al.*, 2003; Parreu *et al.*, 2007; Zhu *et al.*, 2007). The compound $LiSm(PO_3)_4$ has been reported but only unit cell parameters have been refined from powder X-ray diffraction data (Ferid *et al.*, 1984). The single-crystal structure determination performed here confirms that it is isotypic with $LiLn(PO_3)_4$ (Ln = Y, La, Nd, Eu, Gd, Tb, Dy, Er, Yb) (Ben Zarkouna *et al.*, 2007). The structure features two P sites (Fig. 1) centred within PO_4 tetrahedra, which share common corners (O2 or O6) to form infinite zigzag chains $(PO_3)_n^{n-}$ that are aligned parallel to the *b*-direction and are linked together by four-coordinate Li^+ and eight-coordinate Sm^{3+} ions (Fig. 2).

Experimental

Finely ground reagents Li_2CO_3, Sm_2O_3, and $NH_4H_2PO_4$ were mixed in a molar ratio of Li:Sm:P = 7:1:10, placed in a Pt crucible, and heated at 673 K for 4 h. The mixture was reground and heated at 1073 K for 20 h, cooled to 873 K at a rate of 4 K h^{-1}, and then quenched to room temperature. A few yellow prism-shaped crystals of the title compound were obtained.

Refinement

The highest peak and the deepest hole in the difference electron density map are located 0.92 Å and 1.11 Å, respectively, from Sm1.

Figures

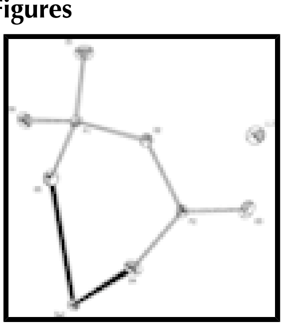

Fig. 1. Part of the structure of $LiSm(PO_3)_4$ showing the labelling of all atoms. Displacement ellipsoids are drawn at the 50% probability level.

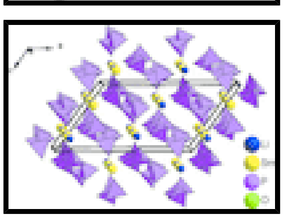

Fig. 2. Projection of the structure of $LiSm(PO_3)_4$ down the *b* axis.

lithium samarium polyphosphate

Crystal data

$LiSm(PO_3)_4$	$F(000) = 884$
$M_r = 473.17$	$D_x = 3.477$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 487 reflections
$a = 16.379$ (2) Å	$\theta = 2.1–23.0°$
$b = 7.0499$ (9) Å	$\mu = 7.27$ mm^{-1}
$c = 9.6936$ (12) Å	$T = 298$ K
$\beta = 126.138$ (2)°	Prism, yellow
$V = 903.96$ (19) Å^3	0.20 × 0.15 × 0.05 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	854 independent reflections
Radiation source: fine-focus sealed tube	843 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.023$
φ and ω scans	$\theta_{max} = 25.7°$, $\theta_{min} = 3.1°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$h = -20 \rightarrow 19$
$T_{min} = 0.439$, $T_{max} = 1.000$	$k = -8 \rightarrow 8$
2405 measured reflections	$l = -10 \rightarrow 11$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.019$	$w = 1/[\sigma^2(F_o^2) + (0.0274P)^2 + 6.0112P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.048$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.09$	$\Delta\rho_{max} = 1.02$ e Å^{-3}
854 reflections	$\Delta\rho_{min} = -0.71$ e Å^{-3}
84 parameters	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.0069 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Li1	0.5000	0.2975 (12)	0.7500	0.014 (2)
Sm1	0.5000	0.20102 (3)	0.2500	0.00541 (15)
P1	0.36163 (7)	0.55515 (13)	0.33744 (11)	0.0057 (2)
P2	0.35188 (7)	0.15529 (14)	0.40335 (12)	0.0056 (2)
O1	0.3857 (2)	0.7182 (4)	0.4524 (4)	0.0117 (6)
O2	0.3410 (2)	0.3787 (4)	0.4149 (3)	0.0094 (5)
O3	0.4267 (2)	0.0930 (4)	0.5830 (3)	0.0104 (5)
O4	0.3705 (2)	0.1147 (4)	0.2737 (3)	0.0104 (5)
O5	0.43430 (19)	0.5038 (4)	0.2978 (3)	0.0094 (5)
O6	0.25564 (19)	0.5836 (4)	0.1557 (3)	0.0091 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Li1	0.018 (5)	0.011 (5)	0.016 (5)	0.000	0.011 (5)	0.000
Sm1	0.00650 (19)	0.00499 (19)	0.00591 (19)	0.000	0.00431 (14)	0.000
P1	0.0060 (4)	0.0046 (4)	0.0068 (5)	0.0004 (3)	0.0040 (4)	0.0004 (3)
P2	0.0059 (4)	0.0053 (4)	0.0062 (5)	−0.0005 (3)	0.0040 (4)	0.0004 (4)
O1	0.0130 (14)	0.0093 (14)	0.0112 (15)	−0.0007 (10)	0.0063 (13)	−0.0026 (10)
O2	0.0167 (13)	0.0052 (13)	0.0133 (13)	0.0000 (10)	0.0126 (12)	0.0009 (10)
O3	0.0108 (13)	0.0076 (12)	0.0094 (13)	0.0003 (10)	0.0041 (11)	0.0016 (10)
O4	0.0127 (13)	0.0110 (13)	0.0126 (13)	−0.0019 (11)	0.0102 (11)	−0.0026 (11)
O5	0.0076 (12)	0.0107 (13)	0.0111 (13)	0.0012 (10)	0.0061 (11)	0.0021 (10)
O6	0.0083 (12)	0.0125 (13)	0.0073 (12)	0.0027 (10)	0.0049 (11)	0.0012 (10)

Geometric parameters (Å, °)

Li1—O3	1.962 (7)	Sm1—O5^{iv}	2.553 (3)
Li1—O3^{i}	1.962 (7)	P1—O1	1.483 (3)
Li1—O5^{ii}	1.980 (7)	P1—O5	1.495 (3)
Li1—O5^{iii}	1.980 (7)	P1—O2	1.590 (3)
Li1—P2	2.927 (3)	P1—O6	1.597 (3)
Li1—P2^{i}	2.927 (3)	P1—Li1^{iii}	3.033 (3)
Li1—P1^{ii}	3.033 (3)	P2—O4	1.485 (3)
Li1—P1^{iii}	3.033 (3)	P2—O3	1.487 (3)
Sm1—O4	2.345 (3)	P2—O6^{viii}	1.580 (3)

Sm1—O4^{iv}	2.345 (3)	P2—O2	1.596 (3)
Sm1—O1^{iii}	2.405 (3)	O1—Sm1^{iii}	2.405 (3)
Sm1—O1^{v}	2.405 (3)	O3—Sm1^{vii}	2.463 (3)
Sm1—O3^{vi}	2.463 (3)	O5—Li1^{iii}	1.980 (7)
Sm1—O3^{vii}	2.463 (3)	O6—P2^{ix}	1.580 (3)
Sm1—O5	2.553 (3)		
O3—Li1—O3^{i}	85.4 (4)	O3^{vi}—Sm1—O3^{vii}	65.38 (12)
O3—Li1—O5^{ii}	124.08 (11)	O4—Sm1—O5	72.34 (9)
O3^{i}—Li1—O5^{ii}	118.63 (11)	O4^{iv}—Sm1—O5	137.49 (9)
O3—Li1—O5^{iii}	118.63 (11)	O1^{iii}—Sm1—O5	72.38 (9)
O3^{i}—Li1—O5^{iii}	124.08 (11)	O1^{v}—Sm1—O5	84.63 (9)
O5^{ii}—Li1—O5^{iii}	90.0 (4)	O3^{vi}—Sm1—O5	136.79 (8)
O3—Li1—P2	27.29 (8)	O3^{vii}—Sm1—O5	132.74 (9)
O3^{i}—Li1—P2	112.7 (3)	O4—Sm1—O5^{iv}	137.49 (9)
O5^{ii}—Li1—P2	108.29 (11)	O4^{iv}—Sm1—O5^{iv}	72.34 (9)
O5^{iii}—Li1—P2	99.83 (10)	O1^{iii}—Sm1—O5^{iv}	84.63 (9)
O3—Li1—P2^{i}	112.7 (3)	O1^{v}—Sm1—O5^{iv}	72.38 (9)
O3^{i}—Li1—P2^{i}	27.29 (8)	O3^{vi}—Sm1—O5^{iv}	132.74 (9)
O5^{ii}—Li1—P2^{i}	99.83 (10)	O3^{vii}—Sm1—O5^{iv}	136.79 (8)
O5^{iii}—Li1—P2^{i}	108.29 (11)	O5—Sm1—O5^{iv}	66.51 (12)
P2—Li1—P2^{i}	139.9 (3)	O4—Sm1—Li1^{vii}	74.97 (7)
O3—Li1—P1^{ii}	106.74 (11)	O4^{iv}—Sm1—Li1^{vii}	74.97 (7)
O3^{i}—Li1—P1^{ii}	102.43 (10)	O1^{iii}—Sm1—Li1^{vii}	103.71 (6)
O5^{ii}—Li1—P1^{ii}	25.02 (8)	O1^{v}—Sm1—Li1^{vii}	103.71 (6)
O5^{iii}—Li1—P1^{ii}	114.9 (3)	O3^{vi}—Sm1—Li1^{vii}	32.69 (6)
P2—Li1—P1^{ii}	100.84 (3)	O3^{vii}—Sm1—Li1^{vii}	32.69 (6)
P2^{i}—Li1—P1^{ii}	92.67 (3)	O5—Sm1—Li1^{vii}	146.74 (6)
O3—Li1—P1^{iii}	102.43 (10)	O5^{iv}—Sm1—Li1^{vii}	146.74 (6)
O3^{i}—Li1—P1^{iii}	106.74 (11)	O4—Sm1—Li1^{iii}	105.03 (7)
O5^{ii}—Li1—P1^{iii}	114.9 (3)	O4^{iv}—Sm1—Li1^{iii}	105.03 (7)
O5^{iii}—Li1—P1^{iii}	25.02 (8)	O1^{iii}—Sm1—Li1^{iii}	76.29 (6)
P2—Li1—P1^{iii}	92.67 (3)	O1^{v}—Sm1—Li1^{iii}	76.29 (6)
P2^{i}—Li1—P1^{iii}	100.84 (3)	O3^{vi}—Sm1—Li1^{iii}	147.31 (6)
P1^{ii}—Li1—P1^{iii}	139.9 (3)	O3^{vii}—Sm1—Li1^{iii}	147.31 (6)
O3—Li1—Sm1^{vii}	42.70 (19)	O5—Sm1—Li1^{iii}	33.26 (6)
O3^{i}—Li1—Sm1^{vii}	42.70 (19)	O5^{iv}—Sm1—Li1^{iii}	33.26 (6)
O5^{ii}—Li1—Sm1^{vii}	135.01 (19)	Li1^{vii}—Sm1—Li1^{iii}	180.000 (1)
O5^{iii}—Li1—Sm1^{vii}	135.01 (19)	O1—P1—O5	118.76 (17)
P2—Li1—Sm1^{vii}	69.97 (16)	O1—P1—O2	106.74 (15)
P2^{i}—Li1—Sm1^{vii}	69.97 (16)	O5—P1—O2	110.85 (15)
P1^{ii}—Li1—Sm1^{vii}	110.03 (16)	O1—P1—O6	111.48 (16)

P1^{iii}—Li1—Sm1^{vii}	110.03 (16)	O5—P1—O6	105.01 (14)
O3—Li1—Sm1^{iii}	137.30 (19)	O2—P1—O6	102.92 (15)
O3^{i}—Li1—Sm1^{iii}	137.30 (19)	O1—P1—Li1^{iii}	91.88 (18)
O5^{ii}—Li1—Sm1^{iii}	44.99 (19)	O2—P1—Li1^{iii}	143.38 (18)
O5^{iii}—Li1—Sm1^{iii}	44.99 (19)	O6—P1—Li1^{iii}	98.86 (11)
P2—Li1—Sm1^{iii}	110.03 (16)	O4—P2—O3	119.71 (16)
P2^{i}—Li1—Sm1^{iii}	110.03 (16)	O4—P2—O6^{viii}	111.89 (15)
P1^{ii}—Li1—Sm1^{iii}	69.97 (16)	O3—P2—O6^{viii}	107.63 (15)
P1^{iii}—Li1—Sm1^{iii}	69.97 (16)	O4—P2—O2	109.68 (15)
Sm1^{vii}—Li1—Sm1^{iii}	180.0	O3—P2—O2	104.96 (15)
O4—Sm1—O4^{iv}	149.93 (13)	O6^{viii}—P2—O2	101.19 (15)
O4—Sm1—O1^{iii}	93.04 (10)	O4—P2—Li1	126.36 (11)
O4^{iv}—Sm1—O1^{iii}	94.01 (10)	O6^{viii}—P2—Li1	121.18 (11)
O4—Sm1—O1^{v}	94.01 (10)	O2—P2—Li1	68.45 (19)
O4^{iv}—Sm1—O1^{v}	93.04 (10)	P1—O1—Sm1^{iii}	139.38 (17)
O1^{iii}—Sm1—O1^{v}	152.59 (13)	P1—O2—P2	132.45 (17)
O4—Sm1—O3^{vi}	74.20 (9)	P2—O3—Li1	115.5 (2)
O4^{iv}—Sm1—O3^{vi}	80.54 (9)	P2—O3—Sm1^{vii}	139.82 (16)
O1^{iii}—Sm1—O3^{vi}	136.12 (9)	Li1—O3—Sm1^{vii}	104.6 (2)
O1^{v}—Sm1—O3^{vi}	71.22 (9)	P2—O4—Sm1	132.98 (16)
O4—Sm1—O3^{vii}	80.54 (9)	P1—O5—Li1^{iii}	120.9 (2)
O4^{iv}—Sm1—O3^{vii}	74.20 (9)	P1—O5—Sm1	137.19 (16)
O1^{iii}—Sm1—O3^{vii}	71.22 (9)	Li1^{iii}—O5—Sm1	101.8 (2)
O1^{v}—Sm1—O3^{vii}	136.12 (9)	P2^{ix}—O6—P1	133.96 (18)

Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2; (ii) *x*, −*y*+1, *z*+1/2; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*+1, *y*, −*z*+1/2; (v) *x*, −*y*+1, *z*−1/2; (vi) *x*, −*y*, *z*−1/2; (vii) −*x*+1, −*y*, −*z*+1; (viii) −*x*+1/2, *y*−1/2, −*z*+1/2; (ix) −*x*+1/2, *y*+1/2, −*z*+1/2.

Fig. 1

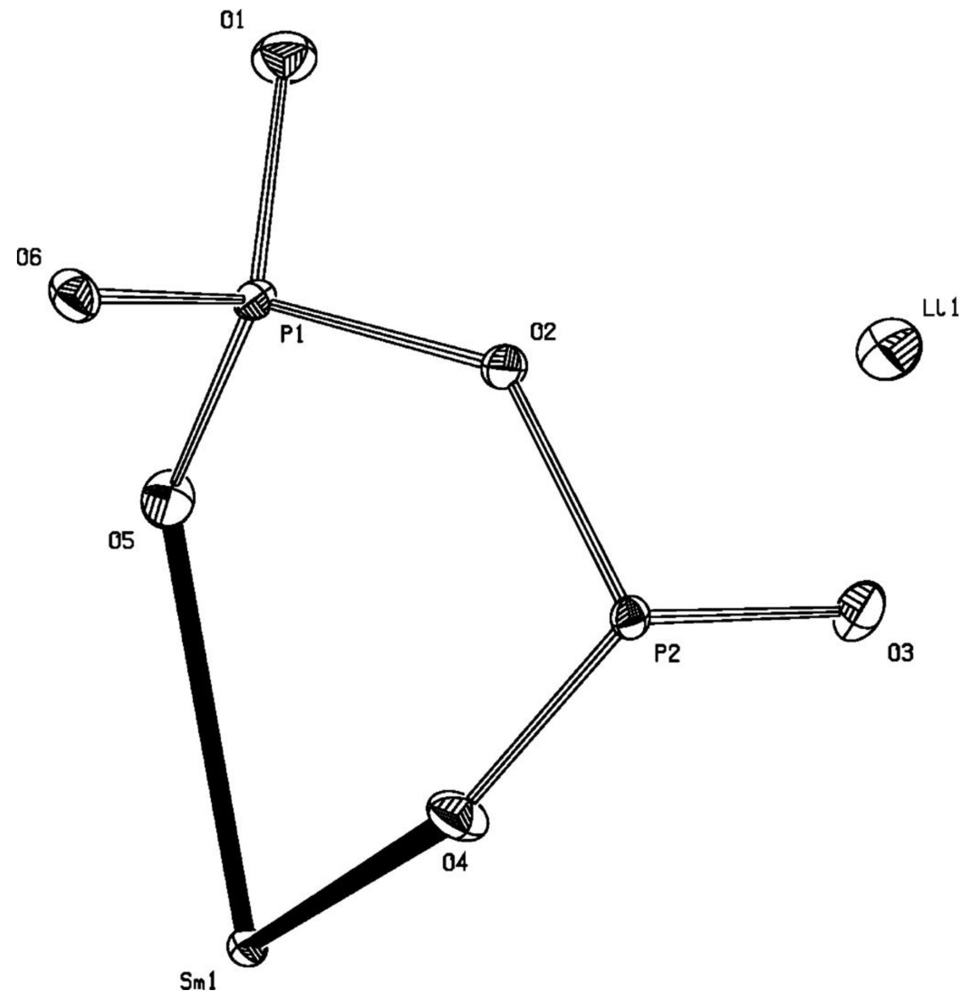

Fig. 2