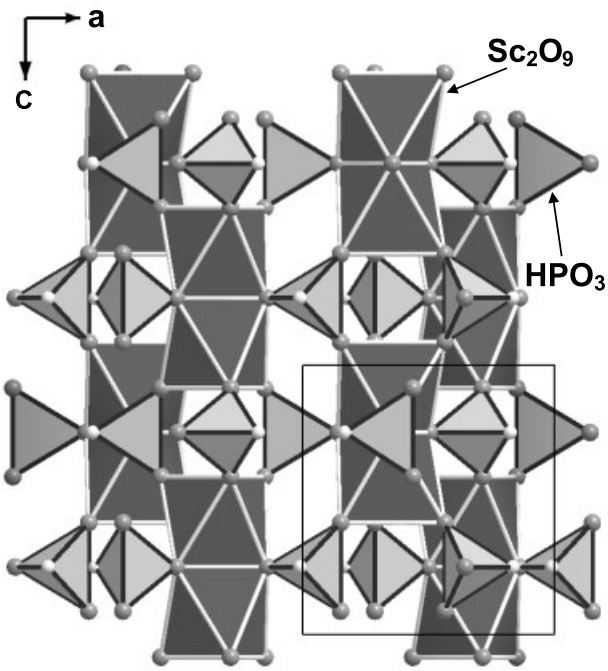
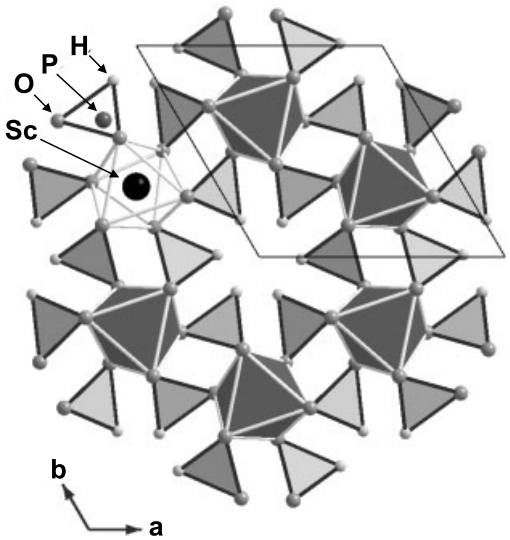
Crystal structure of discandium tris-monohydrogenphosphate(III), $Sc_2(HPO_3)_3$

B. Ewald, Yu. Prots and R. Kniep*

Max-Planck-Institut für Chemische Physik fester Stoffe, Nöthnitzer Str. 40, D-01187 Dresden, Germany

Received August 12, 2003, accepted and available on-line October 5, 2003; CSD-No. 409724





Abstract

$H_3O_9P_3Sc_2$, hexagonal, $P6_3/m$ (No. 176), a = 8.3055(7) Å, c = 7.697(1) Å, V = 459.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.028, $wR_{ref}(F^2)$ = 0.061, T = 293 K.

* Correspondence author (e-mail: kniep@cpfs.mpg.de)

Source of material

Single crystals of the title compound were obtained as by-product during investigations of the system Na–Sc–B–P–O(-H) under mild hydrothermal conditions. According to a molar ratio of 4:1:2, a mixture of 1.000 g (6.61 mmol) $ScCl_3$ (Alfa-Aesar, 99.9%), 0.630 g (1.65 mmol) $Na_2B_4O_7 \cdot 10H_2O$ (Riedel-de-Haën, 99%) and 0.350 g (3.30 mmol) $NaH_2PO_2 \cdot H_2O$ (Alfa Aesar) was treated hydrothermally at T = 443 K. The reaction was carried out under autogenous pressure in a 20 ml teflon autoclave filled with 7 ml water according to a filling degree of 30%. After a reaction time of 58 days the product was washed with water and dried at 333 K in air. The crystals obtained are of hexagonal bipyramidal shape and up to 60 μm in length. Lattice parameters were refined from 2740 reflections obtained during the data collection.

Experimental details

The position of the hydrogen atom was determined from difference Fourier maps and was refined maintaining its U_{iso} parameter constant.

Discussion

Although scandium phosphates(III) have already been investigated [1-5], the crystal structure of $Sc_2(HPO_3)_3$ has not been reported so far. Scandium hydrogenphosphate(III) is isostructural with the known compounds $M^{III}(HPO_3)_3$ (M = Fe [6], Al and Ga [7]). The structural building units consist of HPO_3 tetrahedra and Sc_2O_9 dimers built from distorted ScO_6 octahedra sharing a common face (figure, top). Every oxygen of the octahedral dimers belongs to one phosphate(III) group which links three adjacent Sc_2O_9 units (figure, bottom). The crystal structure contains infinite channels running along [001]. The protons of the phosphate(III) groups point towards the channels' center. Interatomic distances are in usual limits observed for for this class of compounds: d(P—O) = 1.507(1) Å and 1.536(2) Å, d(Sc—O) = 2.020(1) Å and 2.182(1) Å, d(P—H) = 1.36(3) Å.

Table 1. Data collection and handling.

Crystal:	colorless hexagonal bipyramid, size 0.06 × 0.06 × 0.06 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	20.20 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7 Mercury CCD; 375 images, $\Delta\varphi$ = 0.8°; 60 images, χ = –90°, $\Delta\omega$ = 0.8°
$2\theta_{max}$:	64.26°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4307, 576
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 504
$N(param)_{refined}$:	27
Programs:	SHELXL-97 [8], DIAMOND [9]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	6*h*	0.011(4)	0.178(4)	1/4	0.021

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sc(1)	4*f*	1/3	2/3	0.04587(7)	0.0180(2)	U_{11}	0.0081(2)	0.00899(8)	0	0
P(1)	6*h*	0.35183(8)	0.04198(8)	1/4	0.0136(2)	0.0160(3)	0.0117(3)	0.0071(2)	0	0
O(1)	12*i*	0.4367(2)	0.8497(2)	0.0858(2)	0.0260(6)	0.0239(6)	0.0197(6)	0.0112(5)	0.0053(5)	0.0079(5)
O(2)	6*h*	0.1407((2)	0.4969(2)	1/4	0.0164(7)	0.0211(6)	0.0112(7)	0.0051(6)	0	0

References

1. Petru, F.; Muck, A.: Über das Scandiumphosphit. Z. Anorg. Allg. Chem. **352** (1967) 330-332.
2. Kommissarova, L. N.; Melnicov, P. P.; Chernova, N. A.: On Scandium Phosphites. J. Less-Common Met. **13** (1967) 253-262.
3. Petru, F.; Muck, A.: Chromatographische Untersuchung von Produkten der Oxydation von Scandiumphosphit $Sc_2(HPO_3)_3$ durch Luftsauerstoff und von Produkten seiner thermischen Zersetzung im Vakuum. Z. Chem. **9** (1969) 38-39.
4. Petru, F.; Muck, A.: Beiträge zur Chemie der selteneren Elemente LXXVII., Coll. Czechoslov. Chem. Comm. **36** (1971) 3774-3779.
5. Ebert, M.; Kavan, L.: Scandium, Yttrium and Lanthanum Phosphites. J. Less-Common Met. **81** (1981) 55-60.
6. Sghyar, P. M.; Durand, J.; Cot, L.; Rafiq, M.: Structure du Phosphite de Fer: $Fe_2(PO_3H)_3$. Acta Crystallogr. C**47** (1991) 2515-2517.
7. Morris, R. E.; Attfield, M. P.; Cheetham, A. K.: Synthesis and Structures of Two Isostructural Phosphites, $Al_2(HPO_3)_3$ and $Ga_2(HPO_3)_3$. Acta Crystallogr. C**50** (1994) 473-476.
8. Sheldrick, G. M.: SHELXL-97. Program for the refinement of crystal structures. University of Göttingen, Germany 1997.
9. Brandenburg, K.: Diamond, Version 2.0f. Crystal Impact GbR, Bonn, Germany 1998.