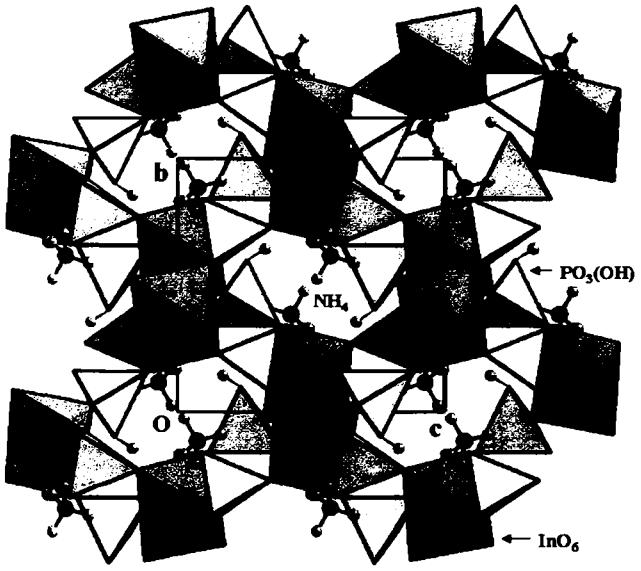
Crystal structure of ammonium indium di(hydrogenmonophosphate), $(NH_4)In[PO_3(OH)]_2$

S.-Y. Mao[I], M.-R. Li[I], J.-X. Mi[I], H.-H. Chen[I], J.-F. Deng[I] and J.-T. Zhao*,[II]

[I] Xiamen University, College of Chemistry and Chemical Engineering, Xiamen 361005, P. R. China
[II] Chinese Academy of Sciences, Shanghai Institute of Ceramics, State Key Laboratory of High Performance Ceramics and Superfine Microstructure,1295 Dingxi Road, Shanghai 200050, P. R. China

Received April 4, 2002, accepted and available on-line July 3, 2002; CSD-No. 409621



Abstract

$H_6InNO_8P_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.6004(4)$ Å, $b = 8.2820(4)$ Å, $c = 9.6693(3)$ Å, $\beta = 116.205(4)°$, $V = 689.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.040$, $wR_{ref}(F^2) = 0.104$, $T = 295$ K.

Source of material

$(NH_4)In[P_2O_6(OH)_2]$ was synthesized under mild hydrothermal conditions. The reactions were carried out with mixtures of $InCl_3$, $LiCl{\cdot}H_2O$, $NaBO_2{\cdot}4H_2O$ and $(NH_4)H_2PO_4$ with the molar ratio of 1:4:9:6 in aqueous solution. The mixture was sealed in 20 ml glass tube. The pH value of the solution was about 0.2. The filling of the solution were about 30% of the glass tubes. The glass tubes were placed in an oven with subsequent heating at 413 K for 7 days. The starting materials are all of analytical grade and used without further purification.

Discussion

There was only one ammonium-indium phosphate [1] and one ammonium-indium borophospahte [2] reported up to now and the title compound is another indium phosphate with ammonium cation obtained as a by-product of new borophosphate, $NaIn[BP_2O_8(OH)]$ [3]. The title compound has a new structure type which is characterized by isolated InO_6 octahedra sharing common O-corners with six isolated $PO_3(OH)$ tetrahedra to form a three-dimensional network structure. Every $PO_3(OH)$ tetrahedron shares common O-corners with three InO_6 octahedra besides the terminal OH group. In the structure, there are eight-membered rings formed by four InO_6 octahedra and four hydrogenphosphate tetrahedra. Ammonium groups are distributed within the open elliptical channels running along the a axis. In InO_6 octahedron the In—O bond distances range from 2.107 Å to 2.168 Å. Bond lengths and angles of hydrogenphosphate $PO_3(OH)$ tetrahedra are similar to other phosphates.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.07 × 0.15 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	39.05 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	56.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1764, 1666
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1596
$N(param)_{refined}$:	110
Programs:	SHELXL-97 [4], DIAMOND [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.9924	0.1696	0.0049	0.05
H(2)	4e	0.2644	0.6439	0.6704	0.05
H(3)	4e	0.6525	0.4748	0.5241	0.05
H(4)	4e	0.5792	0.3318	0.5218	0.05
H(5)	4e	0.6630	0.4119	0.6613	0.05
H(6)	4e	0.7198	0.2953	0.5660	0.05

* Correspondence author (e-mail: jtzhao@mail.sic.ac.cn)

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}
In(1)	4e	0.24676(3)	0.12325(3)	0.51223(3)	0.0064(2)	0.0098(2)	0.0088(2)	0.00006(9)	0.0047(2)	–0.00039(9)
P(1)	4e	0.4144(1)	0.4323(1)	0.7720(1)	0.0077(5)	0.0105(5)	0.0086(5)	–0.0019(4)	0.0052(4)	–0.0017(4)
P(2)	4e	0.0137(1)	0.3213(1)	0.1824(1)	0.0059(5)	0.0103(5)	0.0093(5)	0.0008(4)	0.0044(4)	0.0015(4)
O(1)	4e	0.8596(4)	0.4120(4)	0.1269(4)	0.007(1)	0.020(2)	0.017(2)	0.002(1)	0.006(1)	0.006(1)
O(2)	4e	0.1330(4)	0.4164(4)	0.1544(4)	0.011(1)	0.016(2)	0.016(2)	–0.002(1)	0.010(1)	–0.001(1)
O(3)	4e	0.3253(4)	0.5985(4)	0.7382(4)	0.020(2)	0.013(2)	0.024(2)	0.005(1)	0.014(2)	0.004(1)
O(4)	4e	0.3561(4)	0.3419(4)	0.6186(4)	0.016(2)	0.016(2)	0.011(2)	–0.003(1)	0.007(1)	–0.004(1)
O(5)	4e	0.5832(4)	0.4729(4)	0.8240(4)	0.011(1)	0.020(2)	0.016(2)	–0.003(1)	0.007(1)	–0.006(1)
O(6)	4e	0.3856(4)	0.3405(4)	0.8934(4)	0.015(2)	0.017(2)	0.012(2)	0.004(1)	0.010(1)	0.005(1)
O(7)	4e	0.0632(4)	0.2709(4)	0.3507(4)	0.012(1)	0.016(2)	0.011(1)	0.003(1)	0.008(1)	0.005(1)
O(8)	4e	0.9793(4)	0.1578(4)	0.0873(4)	0.024(2)	0.015(2)	0.020(2)	–0.004(1)	0.014(2)	–0.005(1)
N(1)	4e	0.6561(5)	0.3845(5)	0.5735(6)	0.020(2)	0.026(2)	0.020(2)	0.006(2)	0.011(2)	0.003(2)

Acknowledgments. This project was supported by the Fund for Distinguished Young Scholars from the NNSF of China, the Fund for University Key Teachers from MOE of China, and the Fund from the State Key Laboratory for Crystal Materials at Shandong University. JTZ is indebted to the technical support of graph-making, word-processing, printing and copying provided by the MPI CPfS, Dresden, Germany.

References

1. Mao, S.-Y.; Li, M.-R.; Huang, Y.-X.; Mi, J.-X.; Chen, H.-H.; Wei, Z.-B.; Zhao, J.-T.: Hydrothermal synthesis and crystal structure of the first ammonium indium(III) phosphate $(NH_4)In(OH)PO_4$ with spiral chains of $InO_4(OH)_2$. J. Solid State Chem. **165** (2002) 209-213.
2. Mi, J.-X.; Li, M.-R.; Mao, S.-Y.; Huang, Y.-X.; Wei, Z.-B.; Zhao, J.-T.; Kniep, R.: Crystal structure of ammonium indium (monophosphate-hydrogenmonoborate-monophosphate),$(NH_4)In[BP_2O_8(OH)]$. Z. Kristallogr. NCS **217** (2002) 5-6.
3. Li, M.-R.: Synthesis and crystal structural chemistry of new indium borophosphates, MSc thesis, Xiamen University, 2002.
4. Sheldrick, G. M.: SHELXL-97. Program for refining crystal structures. University of Göttingen, Germany 1997.
5. Brandenburg, K.: Diamond Version 2.1e, Crystal Impact GbR, Bonn, Germany 1996-2001.