Crystal structure of 1,6-hexanediammonium tetra(phenylammonium) cyclohexaphosphate hexahydrate, $[(C_6H_{12}(NH_3)_2)(C_6H_5NH_3)_4]P_6O_{18} \cdot 6H_2O$

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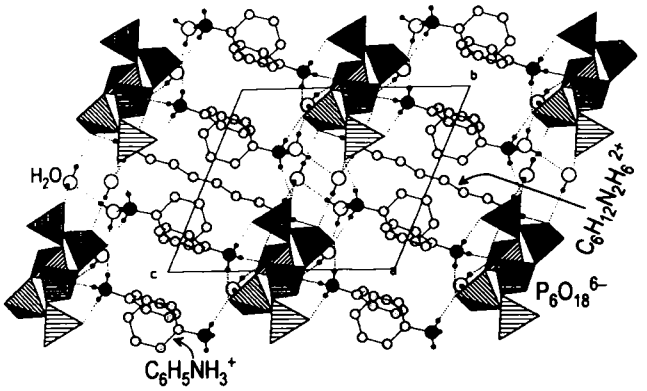

Abstract

$C_{30}H_{62}N_6O_{24}P_6$, triclinic, $P\bar{1}$ (No. 2), $a = 10.082(2)$ Å, $b = 10.637(2)$ Å, $c = 12.262(2)$ Å, $\alpha = 111.94(2)°$, $\beta = 97.06(2)°$, $\gamma = 94.41(2)°$, $V = 1199.8$ Å^3, $Z = 1$, $R_{gt}(F) = 0.040$, $wR_{ref}(F) = 0.057$, $T = 296$ K.

Source of material

The title compound was synthesized by the reaction of cyclohexaphosphoric acid on an aqueous solution of 1,6-diaminohexane and aniline prepared in molar ratio $1,6\text{-}NH_2C_6H_{12}NH_2$ / $C_6H_5NH_2 = 1/4$. The acid was produced from $Li_6P_6O_{18}$ [1] solution through the use of ion-exchange resins Amberlite IR120. Single crystals appeared as square prisms after some days of evaporation of the solution at room temperature.

Discussion

The atomic arrangement, depicted in the figure (projection along *a* axis), can be described as layers of $P_6O_{18}^{6-}$ groups. Between these layers, we find the organic entities: the hexane diammonium groups are outstretched in the *c* direction between two layers and the phenylammonium groups are perpendicular to the inorganic layers. These entities establish hydrogen bonds to interconnect the different layers. Inside such a layer, the phosphoric ring has internal symmetry $\bar{1}$. It develops around inversion centre located at (1/2,0,1/2) and is built up by only three independent PO_4 tetrahedra.

Table 1. Data collection and handling.

Crystal:	pink prism, size 0.22 × 0.24 × 0.35 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	3.11 cm^{-1}
Diffractometer, scan mode:	CAD4, $\omega/2\theta$
$2\theta_{max}$:	59.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7246, 6946
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\,\sigma(I_{obs})$, 4423
$N(param)_{refined}$:	422
Programs:	SIR92 [2], teXsan [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	0.563(2)	0.569(2)	–0.312(2)	0.039(5)
H(2)	2*i*	0.695(3)	0.650(3)	–0.313(2)	0.067(8)
H(3)	2*i*	0.557(2)	0.712(2)	–0.286(2)	0.055(7)
H(4)	2*i*	0.571(3)	0.495(2)	–0.155(2)	0.056(7)
H(5)	2*i*	0.623(3)	0.553(2)	0.044(2)	0.059(7)
H(6)	2*i*	0.717(3)	0.799(3)	0.180(2)	0.078(9)
H(7)	2*i*	0.754(4)	0.968(3)	0.099(3)	0.11(1)
H(8)	2*i*	0.701(3)	0.884(3)	–0.119(3)	0.09(1)
H(9)	2*i*	0.406(3)	0.858(2)	0.257(2)	0.052(7)
H(10)	2*i*	0.296(3)	0.925(3)	0.299(2)	0.080(9)
H(11)	2*i*	0.406(3)	0.990(3)	0.260(2)	0.066(8)
H(12)	2*i*	0.434(4)	0.794(4)	0.048(3)	0.12(1)
H(13)	2*i*	0.332(3)	0.715(3)	–0.144(3)	0.10(1)
H(14)	2*i*	0.108(3)	0.732(3)	–0.200(2)	0.076(9)
H(15)	2*i*	–0.002(3)	0.846(3)	–0.027(2)	0.067(8)
H(16)	2*i*	0.106(3)	0.920(3)	0.156(3)	0.09(1)
H(17)	2*i*	–0.028(2)	0.747(2)	0.384(2)	0.051(7)
H(18)	2*i*	–0.048(2)	0.618(2)	0.391(2)	0.052(7)
H(19)	2*i*	0.078(3)	0.722(2)	0.465(2)	0.059(7)
H(20)	2*i*	0.153(2)	0.676(2)	0.282(2)	0.052(7)
H(21)	2*i*	0.112(2)	0.544(2)	0.285(2)	0.041(6)
H(22)	2*i*	–0.041(2)	0.671(2)	0.167(2)	0.048(6)
H(23)	2*i*	–0.096(2)	0.526(2)	0.166(2)	0.047(6)
H(24)	2*i*	0.080(2)	0.434(2)	0.064(2)	0.036(5)
H(25)	2*i*	0.128(2)	0.582(2)	0.057(2)	0.041(6)
H(26)	2*i*	–0.206(2)	0.415(2)	0.408(2)	0.051(7)
H(27)	2*i*	–0.266(3)	0.519(3)	0.401(2)	0.057(7)
H(28)	2*i*	0.111(3)	0.254(3)	0.334(3)	0.080(9)
H(29)	2*i*	0.155(3)	0.382(3)	0.420(2)	0.071(8)
H(30)	2*i*	0.939(4)	1.103(4)	0.329(3)	0.10(1)
H(31)	2*i*	1.034(4)	1.039(4)	0.342(3)	0.11(1)

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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}
P(1)	2*i*	0.64150(4)	1.16433(4)	0.40967(3)	0.0227(2)	0.0234(1)	0.0276(2)	0.0019(1)	-0.0014(1)	0.0106(1)
P(2)	2*i*	0.75036(4)	0.94391(4)	0.46477(4)	0.0257(2)	0.0254(2)	0.0296(2)	0.0045(1)	0.0015(1)	0.0114(1)
P(3)	2*i*	0.52570(4)	0.72694(4)	0.40271(4)	0.0288(2)	0.0234(2)	0.0304(2)	0.0030(1)	0.0026(1)	0.0102(1)
O(1)	2*i*	0.5281(1)	1.1537(1)	0.3165(1)	0.0305(6)	0.0353(5)	0.0344(5)	0.0021(4)	-0.0068(4)	0.0164(4)
O(2)	2*i*	0.7738(1)	1.2364(1)	0.4137(1)	0.0318(6)	0.0455(6)	0.0502(6)	-0.0086(5)	-0.0039(5)	0.0268(5)
O(3)	2*i*	0.6559(2)	1.0135(1)	0.3957(1)	0.0550(8)	0.0260(5)	0.0543(7)	-0.0013(5)	-0.0234(6)	0.0167(5)
O(4)	2*i*	0.8334(2)	0.8640(2)	0.3832(1)	0.0416(7)	0.085(1)	0.0398(7)	0.0277(7)	0.0101(6)	0.0110(7)
O(5)	2*i*	0.8130(1)	1.0448(1)	0.5836(1)	0.0306(6)	0.0302(5)	0.0363(6)	-0.0002(4)	-0.0040(5)	0.0092(4)
O(6)	2*i*	0.6384(2)	0.8475(1)	0.4883(1)	0.0561(8)	0.0375(6)	0.0324(6)	-0.0174(6)	-0.0015(5)	0.0137(4)
O(7)	2*i*	0.4976(2)	0.7289(1)	0.2832(1)	0.0622(8)	0.0306(5)	0.0272(5)	0.0012(6)	0.0026(5)	0.0069(4)
O(8)	2*i*	0.5569(2)	0.6000(1)	0.4151(1)	0.0446(7)	0.0295(5)	0.0698(8)	-0.0006(5)	-0.0088(6)	0.0239(5)
O(9)	2*i*	0.3964(1)	0.7704(2)	0.4620(1)	0.0350(7)	0.100(1)	0.0294(6)	0.0224(7)	0.0007(5)	0.0076(7)
O(10)	2*i*	-0.1977(2)	0.5003(2)	0.4087(2)	0.0403(7)	0.0394(6)	0.081(1)	0.0027(6)	0.0106(7)	0.0268(6)
O(11)	2*i*	0.1453(2)	0.3363(2)	0.3462(2)	0.0556(9)	0.072(1)	0.0574(8)	-0.0020(8)	0.0067(7)	0.0301(7)
O(12)	2*i*	1.0006(2)	1.0963(2)	0.3210(2)	0.0530(9)	0.0687(9)	0.078(1)	0.0162(8)	0.0178(8)	0.0401(7)
N(1)	2*i*	0.6062(2)	0.6495(2)	-0.2788(1)	0.0349(7)	0.0321(6)	0.0427(7)	0.0011(6)	-0.0045(6)	0.0186(5)
N(2)	2*i*	0.3492(2)	0.9106(2)	0.2420(1)	0.0391(8)	0.0345(7)	0.0339(7)	-0.0013(6)	-0.0019(6)	0.0101(6)
N(3)	2*i*	0.0159(2)	0.6822(2)	0.3859(1)	0.0349(7)	0.0343(6)	0.0286(6)	0.0013(6)	0.0018(5)	0.0096(5)
C(1)	2*i*	0.6361(2)	0.6850(2)	-0.1513(2)	0.0293(8)	0.0319(7)	0.0388(8)	0.0009(6)	-0.0021(6)	0.0145(6)
C(2)	2*i*	0.6139(2)	0.5891(2)	-0.1039(2)	0.051(1)	0.0343(8)	0.0442(9)	0.0045(7)	0.0103(8)	0.0164(7)
C(3)	2*i*	0.6475(3)	0.6267(2)	0.0176(2)	0.061(1)	0.057(1)	0.0466(9)	0.012(1)	0.0137(9)	0.0266(8)
C(4)	2*i*	0.7013(2)	0.7563(3)	0.0904(2)	0.038(1)	0.070(1)	0.043(1)	0.0095(9)	0.0024(8)	0.0150(9)
C(5)	2*i*	0.7244(3)	0.8501(3)	0.0419(2)	0.083(2)	0.056(1)	0.056(1)	-0.019(1)	-0.017(1)	0.012(1)
C(6)	2*i*	0.6914(3)	0.8153(2)	-0.0791(2)	0.090(2)	0.043(1)	0.060(1)	-0.026(1)	-0.021(1)	0.0227(9)
C(7)	2*i*	0.2793(2)	0.8616(2)	0.1192(2)	0.0334(8)	0.0320(7)	0.0329(7)	0.0015(6)	-0.0012(6)	0.0092(6)
C(8)	2*i*	0.3471(3)	0.8002(3)	0.0289(2)	0.042(1)	0.118(2)	0.043(1)	0.031(1)	0.0011(9)	0.009(1)
C(9)	2*i*	0.2815(3)	0.7552(4)	-0.0878(2)	0.068(2)	0.139(3)	0.036(1)	0.033(2)	0.005(1)	0.007(1)
C(10)	2*i*	0.1516(3)	0.7717(3)	-0.1119(2)	0.067(2)	0.070(1)	0.044(1)	0.008(1)	-0.013(1)	0.017(1)
C(11)	2*i*	0.0848(2)	0.8316(3)	-0.0214(2)	0.041(1)	0.059(1)	0.069(1)	0.0040(9)	-0.016(1)	0.022(1)
C(12)	2*i*	0.1477(2)	0.8775(2)	0.0956(2)	0.0346(9)	0.050(1)	0.053(1)	0.0072(8)	0.0050(8)	0.0162(8)
C(13)	2*i*	0.0810(2)	0.6226(2)	0.2796(2)	0.0319(8)	0.0352(8)	0.0313(7)	0.0037(7)	0.0027(6)	0.0072(6)
C(14)	2*i*	-0.0161(2)	0.5889(2)	0.1664(2)	0.0345(8)	0.0436(9)	0.0308(7)	0.0061(7)	0.0019(6)	0.0099(6)
C(15)	2*i*	0.0482(2)	0.5180(2)	0.0580(2)	0.0370(9)	0.0416(9)	0.0331(8)	0.0039(7)	0.0041(7)	0.0088(7)

References

1. Schulke, U.; Kayser, R.: Zur thermischen Dehydratisierung von Lithium-dihydrogenphosphat, -hydrogendiphosphat, und -cyclophosphat Hydraten. Z. Anorg. Allg. Chem. **531** (1985) 167-175.
2. Altomare, A.; Cascarano, G.; Giacovazzo C.; Guagliardi, A.: Complection and refinement of crystal structures with SIR92. J. Appl. Crystallogr. **26** (1993) 343-350.
3. teXsan for Windows version 1.03. Single Crystal Structure Analysis Software. Version 1.03. Molecular Structure Corporation (MSC), 3200 Research Forest Drive, The Woodlands, TX 77381, USA 1997.