Crystal structure of 3,4-dimethyl-phenylammonium cyclotetraphosphate, $(C_8H_{12}N)_4(P_4O_{12})$

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Abstract

$C_{32}H_{48}N_4O_{12}P_4$, triclinic, $P\bar{1}$ (No. 2), $a = 7.982(5)$ Å, $b = 7.652(2)$ Å, $c = 17.154(2)$ Å, $\alpha = 93.83(2)^\circ$, $\beta = 91.93(4)^\circ$, $\gamma = 63.56(4)^\circ$, $V = 936.0$ Å³, $Z = 1$, $R_{gt}(F) = 0.035$, $wR_{eff}(F) = 0.043$, $T = 296$ K.

Source of material

Crystals of the title compound were prepared by adding slowly an ethanolic solution of 3,4-xylidine to an aqueous solution of cyclotetraphosphoric acid kept at low temperature (275 K). After some days of evaporation, large prisms of $(C_8H_{12}N)_4P_4O_{12}$ appear in the solution.

Discussion

A detailed fragment of the structure is shown in the figure (upper part, asymmetric unit with atom labels). The atomic arrangement is typical for a layered structure. Layers including the P_4O_{12} ring anions spread on the (a, b) planes (figure below). Between these layers, we find the organic cations $(C_8H_{12}N)^+$ oriented in the b direction. These entities establish hydrogen bonds to interconnect the different P_4O_{12} rings and so contribute to the intralayer cohesion in the network.

Table 1. Data collection and handling.

Crystal:	colourless prism, size $0.09 \times 0.14 \times 0.70$ mm
Wavelength:	Ag $K\alpha$ radiation (0.5608 Å)
μ :	1.45 cm^{-1}
Diffractometer, scan mode:	CAD4, $\omega/2\theta$
$2\theta_{\max}$:	49.92°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	6862, 6669
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 3 \sigma(I_{\text{obs}})$, 4937
$N(\text{param})_{\text{refined}}$:	331
Programs:	teXsan [1], SIR92 [2]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	2i	0.368(2)	−0.198(2)	0.0963(7)	0.042(3)
H(2)	2i	0.292(2)	0.011(2)	0.0990(7)	0.043(3)
H(3)	2i	0.480(2)	−0.090(2)	0.1093(7)	0.050(4)
H(4)	2i	0.116(1)	−0.121(2)	0.1933(6)	0.035(3)
H(5)	2i	−0.063(2)	−0.131(2)	0.297(1)	0.084(5)
H(6)	2i	0.019(2)	−0.198(2)	0.376(1)	0.086(5)
H(7)	2i	−0.093(2)	0.014(3)	0.367(1)	0.114(7)
H(8)	2i	0.232(3)	−0.128(3)	0.471(1)	0.119(7)

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(9)	2i	0.133(3)	0.099(3)	0.463(1)	0.118(7)
H(10)	2i	0.322(3)	0.010(3)	0.478(1)	0.123(8)
H(11)	2i	0.511(2)	-0.005(2)	0.3687(8)	0.063(4)
H(12)	2i	0.569(2)	-0.038(2)	0.2391(7)	0.046(3)
H(13)	2i	0.839(2)	0.255(2)	0.0757(7)	0.049(4)
H(14)	2i	0.627(2)	0.375(2)	0.0841(7)	0.049(4)
H(15)	2i	0.714(1)	0.171(2)	0.1006(7)	0.037(3)
H(16)	2i	0.512(1)	0.340(2)	0.2236(6)	0.033(3)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(17)	2i	0.372(2)	0.403(2)	0.349(1)	0.095(6)
H(18)	2i	0.534(2)	0.344(2)	0.4169(9)	0.079(5)
H(19)	2i	0.428(2)	0.571(2)	0.4078(9)	0.084(5)
H(20)	2i	0.800(2)	0.422(3)	0.455(1)	0.089(6)
H(21)	2i	0.692(3)	0.639(3)	0.442(1)	0.117(7)
H(22)	2i	0.913(3)	0.523(3)	0.437(1)	0.136(8)
H(23)	2i	1.024(2)	0.455(2)	0.3014(7)	0.052(4)
H(24)	2i	0.986(1)	0.364(1)	0.1740(6)	0.028(3)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	2i	-0.13758(3)	-0.23573(3)	0.04037(1)	0.01474(7)	0.01538(8)	0.0238(1)	-0.00544(6)	0.00121(7)	-0.00169(7)
P(2)	2i	0.24281(3)	-0.52846(3)	0.07359(1)	0.01524(8)	0.01847(8)	0.0241(1)	-0.00744(6)	-0.00209(7)	0.00108(7)
O(1)	2i	-0.06967(8)	-0.17625(8)	-0.02752(4)	0.0225(2)	0.0254(3)	0.0296(3)	-0.0120(2)	0.0005(2)	0.0041(2)
O(2)	2i	-0.22051(8)	-0.38705(8)	0.01302(4)	0.0233(2)	0.0216(2)	0.0249(3)	-0.0128(2)	0.0009(2)	-0.0018(2)
O(3)	2i	-0.27905(8)	-0.08214(9)	0.09277(4)	0.0213(3)	0.0209(3)	0.0387(4)	-0.0059(2)	0.0089(2)	-0.0087(3)
O(4)	2i	0.03246(8)	-0.36891(9)	0.09429(4)	0.0164(2)	0.0269(3)	0.0234(3)	-0.0057(2)	-0.0007(2)	0.0009(2)
O(5)	2i	0.30139(9)	-0.68141(9)	0.12982(4)	0.0301(3)	0.0260(3)	0.0333(3)	-0.0118(2)	-0.0102(3)	0.0095(3)
O(6)	2i	0.35797(8)	-0.42358(9)	0.06314(4)	0.0217(2)	0.0277(3)	0.0337(3)	-0.0151(2)	0.0009(2)	-0.0021(2)
N(1)	2i	0.3759(1)	-0.0893(1)	0.12104(5)	0.0263(3)	0.0217(3)	0.0304(4)	-0.0094(2)	0.0011(3)	0.0029(3)
N(2)	2i	0.7281(1)	0.2845(1)	0.10533(5)	0.0231(3)	0.0252(3)	0.0270(4)	-0.0108(2)	-0.0005(3)	0.0029(3)
C(1)	2i	0.3457(1)	-0.0772(1)	0.20504(5)	0.0305(4)	0.0211(3)	0.0286(4)	-0.0106(3)	0.0001(3)	0.0033(3)
C(2)	2i	0.1941(1)	-0.0987(1)	0.23115(6)	0.0314(4)	0.0284(4)	0.0355(5)	-0.0130(3)	0.0013(4)	0.0016(4)
C(3)	2i	0.1565(2)	-0.0799(2)	0.31071(7)	0.0425(5)	0.0295(4)	0.0409(6)	-0.0121(4)	0.0107(4)	0.0037(4)
C(4)	2i	0.2736(2)	-0.0398(2)	0.36330(7)	0.0647(7)	0.0361(5)	0.0307(5)	-0.0189(5)	0.0048(5)	0.0015(4)
C(5)	2i	0.4273(2)	-0.0246(2)	0.33519(7)	0.0664(7)	0.0548(6)	0.0368(6)	-0.0351(5)	-0.0128(5)	0.0037(5)
C(6)	2i	0.4643(2)	-0.0429(2)	0.25604(7)	0.0437(5)	0.0463(5)	0.0380(5)	-0.0276(4)	-0.0053(4)	0.0066(4)
C(7)	2i	-0.0119(2)	-0.0987(2)	0.3374(1)	0.0570(7)	0.0658(8)	0.0642(9)	-0.0275(6)	0.0227(6)	0.0066(7)
C(8)	2i	0.2364(3)	-0.0126(3)	0.45029(9)	0.108(1)	0.0706(9)	0.0342(7)	-0.0341(8)	0.0120(8)	-0.0038(6)
C(9)	2i	0.7460(1)	0.3446(1)	0.18680(5)	0.0234(3)	0.0220(3)	0.0265(4)	-0.0091(3)	-0.0025(3)	0.0029(3)
C(10)	2i	0.6116(1)	0.3651(1)	0.24005(6)	0.0264(4)	0.0307(4)	0.0300(4)	-0.0127(3)	-0.0022(3)	0.0027(3)
C(11)	2i	0.6270(1)	0.4195(1)	0.31777(6)	0.0395(5)	0.0317(4)	0.0293(5)	-0.0133(4)	0.0006(4)	0.0027(4)
C(12)	2i	0.7797(2)	0.4544(2)	0.34162(6)	0.0545(6)	0.0361(5)	0.0330(5)	-0.0210(4)	-0.0120(5)	0.0010(4)
C(13)	2i	0.9116(2)	0.4323(2)	0.28660(7)	0.0426(5)	0.0448(5)	0.0488(6)	-0.0269(4)	-0.0141(5)	0.0022(5)
C(14)	2i	0.8977(1)	0.3772(1)	0.20920(7)	0.0284(4)	0.0340(4)	0.0404(5)	-0.0170(3)	-0.0019(4)	0.0024(4)
C(15)	2i	0.4799(2)	0.4376(2)	0.37491(8)	0.0652(7)	0.0556(7)	0.0378(6)	-0.0242(5)	0.0170(5)	-0.0015(5)
C(16)	2i	0.7997(3)	0.5173(3)	0.42516(8)	0.098(1)	0.0765(9)	0.0376(7)	-0.0448(7)	-0.0175(7)	-0.0049(6)

References

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