Crystal structure of 2,5-dimethylanilinium cyclohexaphosphate octahydrate, $(C_8H_{12}N)_6O_{18}P_6 \cdot 8H_2O$

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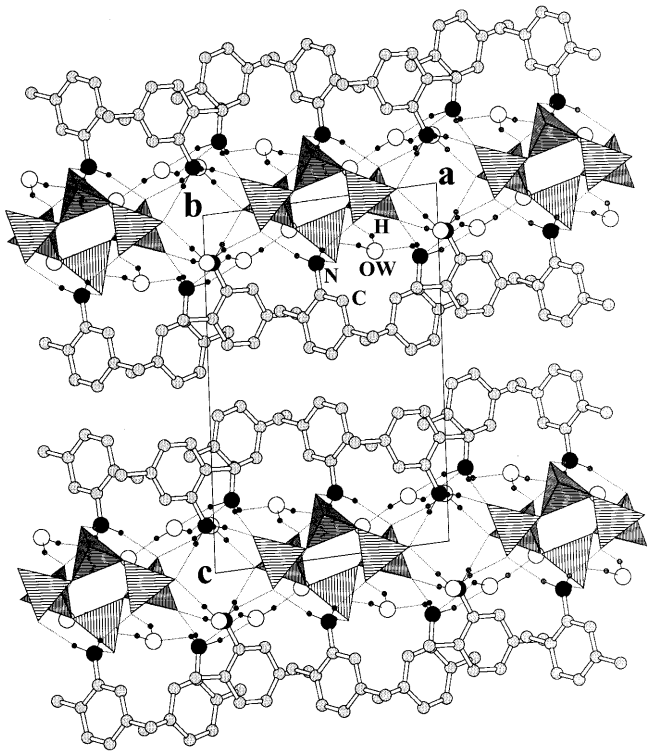

Abstract

$C_{48}H_{88}N_6O_{26}P_6$, triclinic, $P\bar{1}$ (No. 2), a = 10.759(3) Å, b = 10.351(9) Å, c = 15.914(6) Å, α = 99.82(6)°, β = 98.02(2)°, γ = 75.96(5)°, V = 1685.2 Å^3, Z = 1, $R_{gt}(F)$ = 0.060, $wR_{ref}(F)$ = 0.060, T = 296 K.

Source of material

The compound $(C_8H_{12}N)_6P_6O_{18} \cdot 8H_2O$ was prepared by an acid - base reaction. An aqueous solution of cyclohexaphosphoric acid was first obtained by passing a solution of $Li_6P_6O_{18}$ through an ion-exchange resin (amberlite IR 120) in its *H*-State. The lithium salt was prepared according to the process described by Schülke and Kayser [1]. Distilled $(CH_3)_2C_6H_3NH_2$ was added drop by drop to the $H_6P_6O_{18}$ solution, with continuous stirring until the solution exhibits a light greenish colour. The resulting solution was slowly evaporated at room temperature for several days, giving transparent thin single crystals, stable under normal conditions of temperature and humidity.

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Discussion

The projection of the crystal structure of $(C_8H_{12}N)_6O_{18}P_6 \cdot 8H_2O$ along the *b* axis shows the organization of the different components into two entities. The first one includes the inorganic components (P_6O_{18} ring, NH_3 groups and water molecules). The second one is consisted of the organic groups. The inorganic entities form parallel layers around the planes z = 0. Between these layers, the organic groups are located establishing H-bonds via their NH_3 groups with P_6O_{18} rings and water molecules. In this atomic arrangement, there are three independent organic groups having correct values of bond distances and angles. The hydrogen bonds at which they participate explains the stability of the three-dimensional network of the studied crystal structure.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.15 × 0.35 × 0.60 mm
Wavelength:	Ag K_α radiation (0.5608 Å)
μ:	1.30 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius MACH3, $\omega/2\theta$
$2\theta_{max}$:	49.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10909, 10535
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4249
$N(param)_{refined}$:	388
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*	0.9074	0.6629	0.1645	0.042
H(2)	2*i*	0.8889	0.5000	0.1667	0.042
H(3)	2*i*	1.0110	0.5934	0.1878	0.042
H(4)	2*i*	1.0549	0.4030	0.2937	0.115
H(5)	2*i*	1.1585	0.4882	0.3146	0.115
H(6)	2*i*	1.1186	0.4280	0.3869	0.115
H(7)	2*i*	1.0368	0.5925	0.4772	0.042
H(8)	2*i*	0.8586	0.8108	0.5183	0.042
H(9)	2*i*	0.6615	0.9431	0.4673	0.104
H(10)	2*i*	0.6951	1.0114	0.3965	0.104
H(11)	2*i*	0.6004	0.9160	0.3736	0.104
H(12)	2*i*	0.7501	0.7860	0.2578	0.042
H(13)	2*i*	0.5030	1.1934	0.1430	0.042
H(14)	2*i*	0.3940	1.2165	0.1484	0.042
H(15)	2*i*	0.5020	1.0719	0.1727	0.042
H(16)	2*i*	0.2405	1.1795	0.2694	0.076
H(17)	2*i*	0.3486	1.0500	0.2568	0.076
H(18)	2*i*	0.2938	1.0976	0.3448	0.076

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(19)	2*i*	0.3889	1.2552	0.4549	0.042
H(20)	2*i*	0.5550	1.3771	0.4865	0.042
H(21)	2*i*	0.7295	1.4421	0.4650	0.098
H(22)	2*i*	0.7798	1.3974	0.3755	0.098
H(23)	2*i*	0.6741	1.5280	0.3918	0.098
H(24)	2*i*	0.6412	1.3151	0.2477	0.042
H(25)	2*i*	0.0785	0.9270	0.0886	0.042
H(26)	2*i*	–0.0003	1.0274	0.1296	0.042
H(27)	2*i*	–0.0474	0.9103	0.1233	0.042
H(28)	2*i*	–0.0849	1.1459	0.2645	0.099
H(29)	2*i*	–0.1604	1.0324	0.2464	0.099
H(30)	2*i*	–0.1288	1.0969	0.3400	0.099
H(31)	2*i*	0.0534	0.9718	0.4258	0.042

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(32)	2*i*	0.2430	0.8195	0.4339	0.042
H(33)	2*i*	0.4036	0.6478	0.3669	0.120
H(34)	2*i*	0.3743	0.5867	0.2723	0.120
H(35)	2*i*	0.4499	0.7001	0.2937	0.120
H(36)	2*i*	0.2295	0.7815	0.1614	0.042
H(37)	2*i*	0.7177	0.3821	0.1220	0.042
H(38)	2*i*	0.6596	0.5385	0.1438	0.042
H(39)	2*i*	0.0832	1.2192	0.1047	0.042
H(40)	2*i*	–0.0511	1.2520	0.0884	0.042
H(41)	2*i*	0.1024	0.5465	0.1256	0.203
H(42)	2*i*	0.2444	0.4693	0.1188	0.203
H(43)	2*i*	0.3379	0.4448	0.0227	0.187
H(44)	2*i*	0.4482	0.5130	0.0670	0.187

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*	0.52584(7)	0.80582(7)	0.10980(4)	0.0367(4)	0.0349(3)	0.0478(4)	–0.0051(3)	–0.0028(3)	0.0001(3)
P(2)	2*i*	0.30241(6)	0.86612(7)	–0.01769(4)	0.0330(3)	0.0408(4)	0.0364(3)	–0.0113(3)	0.0018(2)	–0.0030(2)
P(3)	2*i*	0.26652(7)	1.15388(7)	–0.02428(4)	0.0392(4)	0.0357(3)	0.0393(3)	–0.0076(3)	–0.0009(3)	–0.0007(3)
O(1)	2*i*	0.5286(3)	0.6687(2)	0.1219(2)	0.117(2)	0.043(1)	0.113(2)	–0.022(1)	–0.016(2)	0.015(1)
O(2)	2*i*	0.5613(2)	0.9028(2)	0.1829(1)	0.052(1)	0.049(1)	0.0382(9)	–0.0106(9)	–0.0010(8)	–0.0006(7)
O(3)	2*i*	0.3868(2)	0.8733(2)	0.0712(1)	0.039(1)	0.082(2)	0.042(1)	0.007(1)	–0.0045(8)	–0.0055(9)
O(4)	2*i*	0.1698(2)	0.8811(3)	–0.0015(1)	0.040(1)	0.132(2)	0.074(1)	–0.033(1)	0.011(1)	–0.032(1)
O(5)	2*i*	0.3626(2)	0.7557(2)	–0.0806(1)	0.055(1)	0.042(1)	0.051(1)	–0.0201(9)	0.0129(8)	–0.0110(8)
O(6)	2*i*	0.3195(3)	0.9974(2)	–0.0488(1)	0.160(3)	0.039(1)	0.065(1)	–0.004(1)	0.046(2)	0.005(1)
O(7)	2*i*	0.1640(2)	1.1997(2)	–0.0901(1)	0.045(1)	0.076(2)	0.055(1)	–0.019(1)	–0.0076(8)	0.021(1)
O(8)	2*i*	0.3915(2)	1.2033(3)	–0.0329(1)	0.045(1)	0.098(2)	0.057(1)	–0.029(1)	0.0138(9)	–0.032(1)
O(9)	2*i*	0.2429(2)	1.1890(2)	0.0668(1)	0.044(1)	0.056(1)	0.045(1)	–0.0062(9)	0.0040(8)	–0.0038(8)
O(10)	2*i*	0.7286(2)	0.4645(2)	0.1657(2)	0.071(2)	0.048(1)	0.118(2)	–0.025(1)	–0.008(1)	–0.008(1)
O(11)	2*i*	0.0111(2)	1.2169(3)	0.1339(2)	0.054(1)	0.097(2)	0.081(2)	–0.016(1)	0.008(1)	0.011(1)
O(12)	2*i*	0.1664(4)	0.4689(4)	0.1413(3)	0.152(4)	0.109(3)	0.279(5)	–0.066(3)	0.141(4)	–0.072(3)
O(13)	2*i*	0.3662(4)	0.4999(4)	0.0734(3)	0.141(3)	0.129(3)	0.204(4)	–0.085(3)	0.103(3)	–0.092(3)
N(1)	2*i*	0.9196(2)	0.5965(2)	0.1979(2)	0.054(2)	0.051(1)	0.059(1)	–0.019(1)	0.004(1)	0.007(1)
N(2)	2*i*	0.4762(2)	1.1705(2)	0.1781(1)	0.041(1)	0.041(1)	0.0312(9)	–0.0116(9)	0.0031(8)	–0.0037(8)
N(3)	2*i*	0.0336(2)	0.9403(3)	0.1364(1)	0.038(1)	0.070(2)	0.049(1)	–0.020(1)	0.0006(9)	0.010(1)
C(1)	2*i*	0.9073(3)	0.6521(3)	0.2881(2)	0.048(2)	0.046(2)	0.052(1)	–0.020(1)	–0.004(1)	0.007(1)
C(2)	2*i*	0.9882(3)	0.5891(3)	0.3530(2)	0.046(2)	0.053(2)	0.078(2)	–0.014(1)	–0.009(1)	0.021(2)
C(3)	2*i*	0.9652(4)	0.6482(4)	0.4363(2)	0.067(2)	0.075(2)	0.058(2)	–0.024(2)	–0.018(2)	0.025(2)
C(4)	2*i*	0.8685(4)	0.7573(4)	0.4543(2)	0.076(2)	0.065(2)	0.050(2)	–0.027(2)	–0.001(1)	0.009(1)
C(5)	2*i*	0.7869(3)	0.8144(3)	0.3904(2)	0.062(2)	0.056(2)	0.058(2)	–0.015(2)	0.003(1)	0.008(1)
C(6)	2*i*	0.8094(3)	0.7598(3)	0.3059(2)	0.055(2)	0.054(2)	0.050(2)	–0.011(1)	–0.011(1)	0.014(1)
C(7)	2*i*	1.0907(4)	0.4654(5)	0.3345(3)	0.081(3)	0.082(3)	0.112(3)	0.010(2)	–0.008(2)	0.019(2)
C(8)	2*i*	0.6762(4)	0.9326(4)	0.4094(3)	0.101(3)	0.083(3)	0.081(2)	0.003(2)	0.019(2)	0.006(2)
C(9)	2*i*	0.4901(2)	1.2318(2)	0.2674(1)	0.038(1)	0.033(1)	0.033(1)	–0.003(1)	0.0019(9)	–0.0027(9)
C(10)	2*i*	0.4137(3)	1.2065(3)	0.3237(2)	0.048(2)	0.038(1)	0.047(1)	–0.005(1)	0.009(1)	0.005(1)
C(11)	2*i*	0.4367(3)	1.2633(3)	0.4091(2)	0.079(2)	0.051(2)	0.038(1)	–0.008(2)	0.017(1)	0.001(1)
C(12)	2*i*	0.5298(3)	1.3358(3)	0.4345(2)	0.071(2)	0.056(2)	0.036(1)	–0.004(2)	–0.003(1)	–0.008(1)
C(13)	2*i*	0.6043(3)	1.3596(3)	0.3777(2)	0.056(2)	0.049(2)	0.047(1)	–0.008(1)	–0.007(1)	–0.008(1)
C(14)	2*i*	0.5826(3)	1.3054(3)	0.2922(2)	0.040(1)	0.044(1)	0.045(1)	–0.012(1)	0.003(1)	–0.005(1)
C(15)	2*i*	0.3155(3)	1.1257(4)	0.2962(2)	0.069(2)	0.068(2)	0.071(2)	–0.033(2)	0.028(2)	–0.005(2)
C(16)	2*i*	0.7064(4)	1.4382(4)	0.4046(2)	0.071(2)	0.094(3)	0.084(2)	–0.038(2)	–0.004(2)	–0.031(2)
C(17)	2*i*	0.0906(3)	0.9029(3)	0.2206(2)	0.045(2)	0.055(2)	0.050(1)	–0.026(1)	0.008(1)	–0.003(1)
C(18)	2*i*	0.0254(3)	0.9679(3)	0.2916(2)	0.065(2)	0.058(2)	0.063(2)	–0.025(2)	0.022(2)	–0.004(1)
C(19)	2*i*	0.0855(4)	0.9286(4)	0.3695(2)	0.098(3)	0.069(2)	0.050(2)	–0.035(2)	0.023(2)	–0.003(2)
C(20)	2*i*	0.1983(4)	0.8325(4)	0.3751(2)	0.096(3)	0.087(3)	0.046(2)	–0.035(2)	–0.000(2)	0.014(2)
C(21)	2*i*	0.2578(4)	0.7703(4)	0.3040(2)	0.072(2)	0.070(2)	0.061(2)	–0.018(2)	0.000(2)	0.017(2)
C(22)	2*i*	0.2009(3)	0.8088(3)	0.2250(2)	0.049(2)	0.057(2)	0.049(2)	–0.018(1)	0.004(1)	0.005(1)
C(23)	2*i*	–0.0982(4)	1.0708(4)	0.2843(3)	0.077(3)	0.090(3)	0.094(3)	0.001(2)	0.034(2)	0.007(2)
C(24)	2*i*	0.3844(5)	0.6670(5)	0.3096(3)	0.102(4)	0.126(4)	0.090(3)	0.021(3)	0.005(2)	0.047(3)

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