Crystal structure of bis(3,5-dimethylanilinium) dihydrogenodiphosphate, $(C_8H_{12}N)_2H_2P_2O_7$

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Abstract

$C_{16}H_{26}N_2O_7P_2$, monoclinic, $C12/c1$ (No. 15), $a = 32.53(1)$ Å, $b = 8.415(2)$ Å, $c = 7.792(4)$ Å, $\beta = 92.18(3)^\circ$, $V = 2131.4$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.071$, $wR_{\text{ref}}(F) = 0.077$, $T = 296$ K.

Source of material

The diphosphoric acid was prepared by passing a concentrated solution of sodium diphosphate $Na_4P_2O_7$ through an ion exchange resin (Amberlite IR 120) in its H-state. The diphosphoric acid was then neutralized with 3,5-dimethylaniline at low temperature by mixing them in stoichiometric proportions. The resulting solution was slowly evaporated at room temperature until the formation of crystals of $(C_8H_{12}N)_2H_2P_2O_7$.

Experimental details

The slightly high value of R is due to the bad quality of the crystal.

Discussion

The crystal structure of the title compound can be described as a succession of thick layers perpendicular to the a axis at $x = 0$ and $x = 1/2$. These layers are formed by phosphoric $H_2P_2O_7^{2-}$ and organic 3,5- $(CH_3)_2C_6H_4NH_3^+$ entities. Each $H_2P_2O_7^{2-}$ anion is connected to two adjacent neighbor diphosphate groups by strong hydrogen bonds building corrugated ribbons parallel to the c direction. These ribbons are interconnected by the H-bonds created by ammonium groups of organic cations. The $H_2P_2O_7$ entity has a two-fold internal symmetry. The bridging oxygen atom is located on the two-fold axis. The empirical formula of the title compound is similar to that of another published diphosphate [1]. However, the two compounds are

completely different. Their organic molecules (3,5-dimethylanilinium for one and 2-phenylethylammonium for the other) and their crystallographic data are not comparable.

Table 1. Data collection and handling.

Crystal:	colorless prism, size $0.15 \times 0.20 \times 0.35$ mm
Wavelength:	Mo $K\alpha$ radiation (0.7107 Å)
μ :	2.41 cm^{-1}
Diffractometer, scan mode:	MACH3, $\omega/2\theta$
$2\theta_{\text{max}}$:	59.92°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3739, 3280
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2193
$N(\text{param})_{\text{refined}}$:	123
Programs:	teXsan [2], SIR92 [3]

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

Atom	Site	x	y	z	U_{iso}
H(1)	8f	0.4702	1.0248	0.8473	0.047
H(2)	8f	0.4516	0.6990	0.3002	0.058
H(3)	8f	0.4325	0.6064	0.1690	0.044
H(4)	8f	0.4397	0.5251	0.3464	0.057
H(5)	8f	0.4106	0.7648	0.5749	0.034
H(6)	8f	0.3688	0.9181	0.7657	0.133
H(7)	8f	0.3269	0.9571	0.7160	0.132
H(8)	8f	0.3379	0.8074	0.7991	0.144
H(9)	8f	0.2917	0.7775	0.4962	0.070
H(10)	8f	0.2641	0.7270	0.1803	0.164
H(11)	8f	0.2706	0.5514	0.2383	0.166
H(12)	8f	0.2884	0.6006	0.0751	0.144
H(13)	8f	0.3614	0.5865	0.1309	0.050

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
P(1)	8 <i>f</i>	0.47650(3)	0.7779(1)	0.9070(2)	0.0357(6)	0.0223(5)	0.0264(5)	−0.0009(6)	0.0008(4)	−0.0006(6)
O(1)	8 <i>f</i>	0.45777(9)	0.9354(4)	0.8363(4)	0.041(2)	0.019(2)	0.051(2)	0.003(1)	−0.009(2)	−0.005(2)
O(2)	8 <i>f</i>	0.50699(9)	0.8069(4)	1.0501(4)	0.049(2)	0.025(2)	0.032(2)	0.005(1)	−0.005(1)	−0.003(1)
O(3)	4 <i>e</i>	1/2	0.7016(5)	3/4	0.060(3)	0.025(3)	0.038(2)	0	0.018(2)	0
O(4)	8 <i>f</i>	0.44302(9)	0.6636(4)	0.9428(4)	0.041(2)	0.038(2)	0.037(2)	−0.010(1)	0.007(1)	−0.001(2)
N(1)	8 <i>f</i>	0.4333(1)	0.6220(5)	0.2893(5)	0.030(2)	0.030(2)	0.036(2)	−0.002(2)	0.002(2)	−0.001(2)
C(1)	8 <i>f</i>	0.3927(1)	0.6724(5)	0.3468(6)	0.036(3)	0.026(2)	0.041(3)	−0.001(2)	0.004(2)	0.002(2)
C(2)	8 <i>f</i>	0.3899(1)	0.7474(6)	0.4987(6)	0.043(3)	0.050(4)	0.040(3)	−0.002(2)	0.003(2)	−0.007(3)
C(3)	8 <i>f</i>	0.3513(2)	0.7891(8)	0.5581(7)	0.050(3)	0.069(4)	0.058(3)	0.000(3)	0.014(3)	−0.014(4)
C(4)	8 <i>f</i>	0.3171(1)	0.7513(8)	0.4541(8)	0.038(3)	0.071(5)	0.075(4)	0.011(3)	0.013(3)	−0.014(4)
C(5)	8 <i>f</i>	0.3199(2)	0.6753(8)	0.3009(8)	0.033(3)	0.094(5)	0.070(4)	−0.002(3)	−0.007(3)	−0.010(4)
C(6)	8 <i>f</i>	0.3586(2)	0.6332(7)	0.2426(7)	0.036(3)	0.073(4)	0.049(3)	0.010(3)	−0.004(2)	−0.014(3)
C(7)	8 <i>f</i>	0.3479(2)	0.875(1)	0.725(1)	0.063(5)	0.162(9)	0.106(6)	0.008(5)	0.014(4)	−0.072(6)
C(8)	8 <i>f</i>	0.2822(2)	0.633(1)	0.184(1)	0.049(4)	0.20(1)	0.126(7)	0.017(5)	−0.025(4)	−0.069(7)

References

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