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Crystal structure of 1,1′-(1,4-butanediyl)bis(imidazolium) 1,4-butylenediphosphonate — 1,4-butylenediphosphonic acid — water (1:1:2), $[C_{10}H_{16}N_4][C_4H_{10}O_6P_2] \cdot C_4H_{12}O_6P_2 \cdot 2H_2O$

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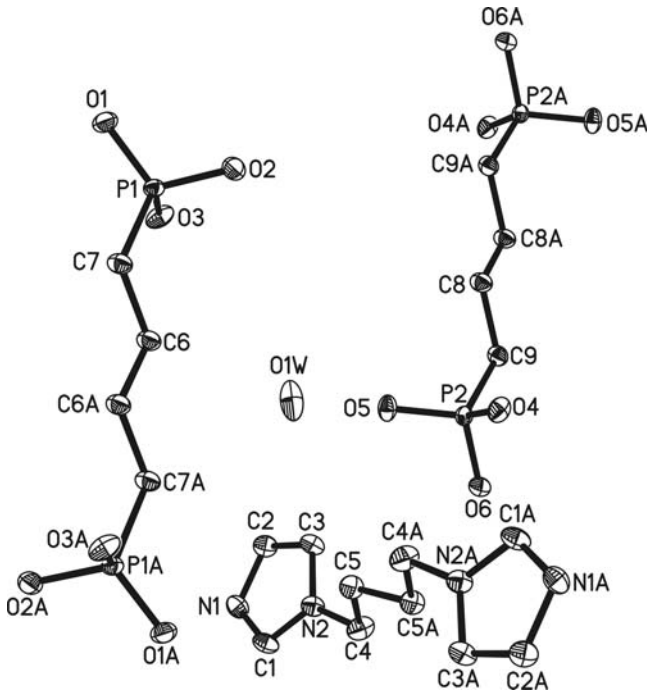

Abstract

$C_{18}H_{42}N_4O_{14}P_4$, triclinic, $P\overline{1}$ (no. 2), a = 8.420(5) Å, b = 9.005(5) Å, c = 10.608(5) Å, α = 69.768(5)°, β = 75.004(5)°, γ = 88.638(5)°, V = 727.1 Å^3, Z = 1, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.114, T = 293 K.

Source of material

All chemicals and solvents were purchased from commercial sources and used without further purification except the 1,4-butylenediphosphonic acid, which was prepared according to the reported method [1] and the 1,1′-(1,4-butanediyl)bis(imidazole), which was prepared according to [2]. A mixture of 1,4-butylenediphosphonic acid (0.06 g, 0.3 mmol) and 1,1′-(1,4-butanediyl)-bis(imidazole) (0.17 g, 0.9 mmol) was added to 25 mL methanol with stirring and slight heating. The solution was filtered into a flask, and the filtrate was left at room temperature for a week, colorless block-shaped crystals of the title compounds were obtained in 46 % yield.

Experimental details

All hydrogen in the structure were generated geometrically with d(C—H) = 0.93 - 0.97 Å, d(N—H) = 0.86 Å, d(O—H) = 0.82 Å and U_{iso}(H) = 1.2 U_{eq}(C,N), U_{iso}(H) = 1.5 U_{eq}(O). The hydrogen atoms of water molecules were located from difference Fourier maps and refined with d(O—H) = 0.869 - 0.894 Å, and U_{iso}(H) = 1.5 U_{eq}(O).

Discussion

Organophosphonates have attracted much attention because of their potential applications, such as ion exchanges, porous materials, catalysts, small molecular sensors, and non-linear optics. In supramolecular architectures, many novel two-dimensional layers and three-dimensional frameworks have been designed and produced based on hydrogen bonds [3].

The unit cell of the title crystal structure contains one 1,1′-(1,4-butanediyl)bis(imidazolium) cation, one 1,4-butylenediphosphonate anion, one 1,4-butylenediphosphonic acid molecule and two lattice water molecules. There are intermolecular hydrogen-bonding interactions between the O atoms of phosphonate and lattice water and N atoms of 1,1′-(1,4-butanediyl)bis(imidazolium), which further link the discrete species in the title crystal structure to a 3D supramolecular framework.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.16 × 0.19 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	3.31 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5219, 2975
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2361
$N(param)_{refined}$:	182
Programs:	SHELXS-97 SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.8654	0.1441	0.4824	0.043
H(2)	2*i*	0.6060	–0.0123	0.2869	0.043
H(3)	2*i*	0.5177	0.2490	0.2825	0.042
H(4A)	2*i*	0.6974	0.4866	0.3206	0.049
H(4B)	2*i*	0.7612	0.4157	0.4543	0.049
H(5A)	2*i*	0.4971	0.3437	0.5949	0.041
H(5B)	2*i*	0.4250	0.3858	0.4653	0.041
H(6A)	2*i*	0.0316	–0.0764	0.9010	0.036
H(6B)	2*i*	–0.0180	0.0990	0.8690	0.036
H(7A)	2*i*	–0.2757	0.0062	1.0319	0.037
H(7B)	2*i*	–0.2261	–0.1682	1.0594	0.037

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(8A)	2*i*	–0.0517	0.5724	0.1017	0.038
H(8B)	2*i*	–0.0058	0.3956	0.1365	0.038
H(9A)	2*i*	0.2194	0.6630	–0.0224	0.036
H(9B)	2*i*	0.2683	0.4893	–0.0031	0.036
H(1A)	2*i*	0.8177	–0.0662	0.4098	0.042

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	–0.2559	0.1518	0.7394	0.055
H(3A)	2*i*	–0.0897	–0.0698	0.7220	0.055
H(6)	2*i*	0.4713	0.6711	0.0910	0.050
H(1WA)	2*i*	0.1218	0.3250	0.4859	0.087
H(1WB)	2*i*	0.0113	0.2973	0.6192	0.087

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*	–0.30465(6)	–0.07469(6)	0.85666(6)	0.0227(3)	0.0246(3)	0.0273(3)	0.0005(2)	–0.0091(2)	–0.0116(2)
P(2)	2*i*	0.25784(6)	0.54198(6)	0.19547(5)	0.0259(3)	0.0209(3)	0.0236(3)	0.0023(2)	–0.0056(2)	–0.0083(2)
C(1)	2*i*	0.7902(3)	0.1381(3)	0.4334(3)	0.030(1)	0.048(1)	0.039(1)	0.014(1)	–0.015(1)	–0.023(1)
C(2)	2*i*	0.6463(3)	0.0524(3)	0.3252(2)	0.038(1)	0.042(1)	0.034(1)	0.005(1)	–0.011(1)	–0.020(1)
C(3)	2*i*	0.5980(3)	0.1954(3)	0.3228(2)	0.033(1)	0.044(1)	0.034(1)	0.010(1)	–0.015(1)	–0.018(1)
C(4)	2*i*	0.6770(3)	0.4023(3)	0.4109(3)	0.034(1)	0.039(1)	0.056(2)	0.005(1)	–0.008(1)	–0.027(1)
C(5)	2*i*	0.5102(3)	0.4160(3)	0.5001(3)	0.036(1)	0.034(1)	0.037(1)	0.006(1)	–0.010(1)	–0.017(1)
C(6)	2*i*	–0.0327(2)	–0.0051(3)	0.9413(2)	0.027(1)	0.039(1)	0.026(1)	0.0018(9)	–0.0103(9)	–0.011(1)
C(7)	2*i*	–0.2135(3)	–0.0634(3)	0.9883(2)	0.028(1)	0.039(1)	0.029(1)	0.0027(9)	–0.0117(9)	–0.014(1)
C(8)	2*i*	0.0190(3)	0.5011(3)	0.0663(2)	0.028(1)	0.040(1)	0.027(1)	0.002(1)	–0.0090(9)	–0.009(1)
C(9)	2*i*	0.1985(3)	0.5540(3)	0.0417(2)	0.027(1)	0.035(1)	0.025(1)	0.0026(9)	–0.0055(9)	–0.006(1)
N(1)	2*i*	0.7661(2)	0.0191(2)	0.3943(2)	0.036(1)	0.036(1)	0.035(1)	0.0137(8)	–0.0111(8)	–0.0154(9)
N(2)	2*i*	0.6892(2)	0.2485(2)	0.3912(2)	0.0284(9)	0.036(1)	0.035(1)	0.0074(8)	–0.0092(8)	–0.0181(9)
O(1)	2*i*	–0.4581(2)	–0.1812(2)	0.9182(2)	0.0284(8)	0.0392(9)	0.0347(9)	–0.0058(7)	–0.0086(7)	–0.0126(8)
O(2)	2*i*	–0.3414(2)	0.0941(2)	0.7729(2)	0.0437(9)	0.0262(8)	0.048(1)	0.0032(7)	–0.0259(8)	–0.0123(8)
O(3)	2*i*	–0.1723(2)	–0.1314(2)	0.7539(2)	0.0288(8)	0.0385(9)	0.045(1)	–0.0049(7)	0.0001(7)	–0.0247(8)
O(4)	2*i*	0.1745(2)	0.6620(2)	0.2555(2)	0.0340(8)	0.0266(8)	0.044(1)	0.0035(6)	–0.0030(7)	–0.0179(7)
O(5)	2*i*	0.2236(2)	0.3747(2)	0.2980(2)	0.052(1)	0.0231(8)	0.0260(8)	0.0017(7)	–0.0104(7)	–0.0096(7)
O(6)	2*i*	0.4494(2)	0.5802(2)	0.1473(2)	0.0265(8)	0.0357(9)	0.0373(9)	0.0042(6)	–0.0106(7)	–0.0104(7)
O(1W)	2*i*	0.0778(2)	0.2510(2)	0.5693(2)	0.066(1)	0.044(1)	0.038(1)	0.0251(9)	0.0115(9)	–0.0031(9)

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