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4-[2-(Hydrogen phosphonato)-2-hydroxy-2-phosphonoethyl]pyridinium

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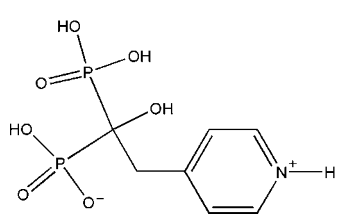
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.117; data-to-parameter ratio = 14.6.

The title compound, $C_7H_{11}NO_7P_2$, exists as a zwitterion in which the positive charge resides on the protonated pyridyl N atom and the negative charge on one of the two phosphate groups. In the crystal, adjacent molcules are linked by O—H···O and N—H···O hydrogen bonds into a three-dimensional network.

Related literature

For metal complexes of phosphonic acids, see: Ma *et al.* (2008, 2009).



Experimental

Crystal data

$C_7H_{11}NO_7P_2$
M_r = 283.11
Monoclinic, $P2_1/n$
a = 10.083 (2) Å
b = 9.4713 (19) Å
c = 11.708 (2) Å
β = 94.87 (3)°
V = 1114.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 293 K
0.3 × 0.25 × 0.2 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.880, T_{max} = 0.92
11137 measured reflections
2548 independent reflections
2093 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.117
S = 1.02
2548 reflections
174 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.81 (2)	2.06 (2)	2.816 (3)	156 (3)
O4—H4A···O7^{i}	0.81 (2)	1.77 (2)	2.574 (3)	176 (4)
O5—H5A···O2^{i}	0.82 (2)	1.66 (2)	2.477 (3)	175 (4)
N1—H2A···O7^{ii}	0.82 (2)	1.93 (2)	2.741 (3)	171 (4)
O6—H6A···O3^{iii}	0.81 (4)	1.67 (4)	2.476 (3)	175 (4)

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x+1, y, z$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5136).

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supporting information

Acta Cryst. (2011). E**67**, o1025 [doi:10.1107/S1600536811011408]

4-[2-(Hydrogen phosphonato)-2-hydroxy-2-phosphonoethyl]pyridinium

Feng-Lei Wang, Rong-Xin Yuan and Ji-Min Xie

S1. Comment

Metal diphosphonates have been extensively studied for the diversity structures. Monomeric, dimeric, polymeric, two-dimensional layers to three-dimensional frameworks are all featured among these complexs Ma *et al.*, 2008; Ma *et al.*, 2009). Because diphosphonates have capabilities to stabilize transition metals in a wide range of oxidation states. The title compound was synthesized and characterized by X-ray crystal structure analysis.

There are some intermolecular hydrogen bonds in the structure of the title compound (Fig. 1 and Table 1). The intermolecular hydrogen bonds [O1—H1A···O2^{i}, O5—H5A···O2^{i} and O4—H4A···O7^{i}; symmetry code: -x+3/2, y-1/2, -z+1/2] bridge the molecules through head-to-tail into a one-dmensional chain. These chains are linked to two-dimensional structure through hydrogen bonds [N1—H2A···O7^{ii}; symmetry code: x+1, y, z]. The hydrogen bonds [O6—H6A···O3^{iii}; symmetry code: x-1/2, -y+1/2, z-1/2] connected the layers into a three-dimensional network (shown in Fig. 2).

S2. Experimental

The title compound was synthesized by reaction of 4-pyridine acetic acid hydrochloride (0.520 g, 3 mmol) and phosphite (0.711 g, 8.7 mmol) in chlorobenzene (15 mL). The solution was stirred while phosphorus trichloride (0.64 g, 4.7 mmol) was added drop by drop and the temperature should control between 110-120 ^{o}C with vigorous stirring for 4 hours. The crude product was concentrated in vacuo. White block crystals formed in high yield by recrystallization.

S3. Refinement

Carbon-bond H atoms were positioned geometrically (C—H = 0.93 Å), and were included in the refinement in the riding mode approximation, with U_{iso}(H) = 1.2U_{eq}(C). H atoms bound to O and N atoms were located in a difference Fourier map and refined with restraints [N—H and O—H = 0.82 (1) Å, with U_{iso}(H) values fixed at 1.5U_{eq}(N) and 1.5U_{eq}(O)].

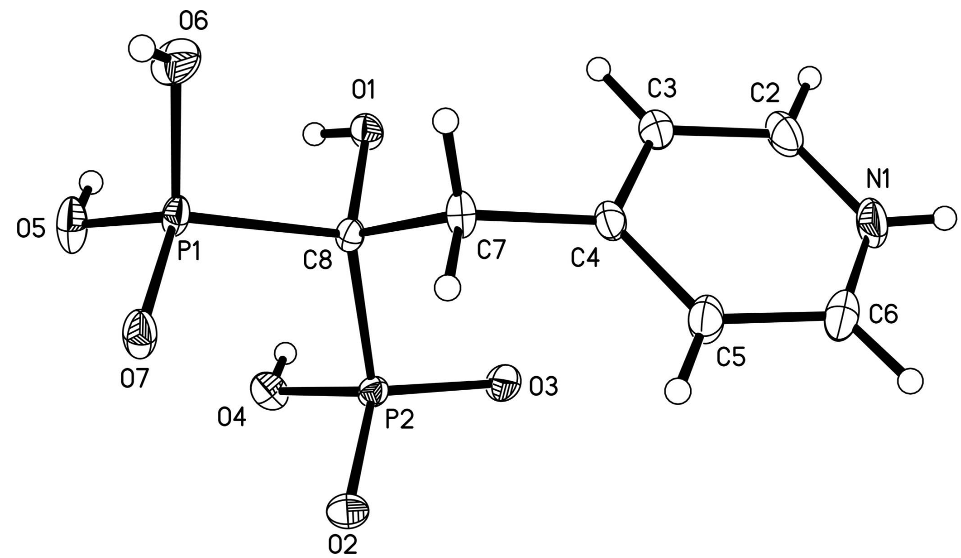

Figure 1

A view of the compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level. [Symmetry code A = 1-*x*, *y*, 1/2-*z*].

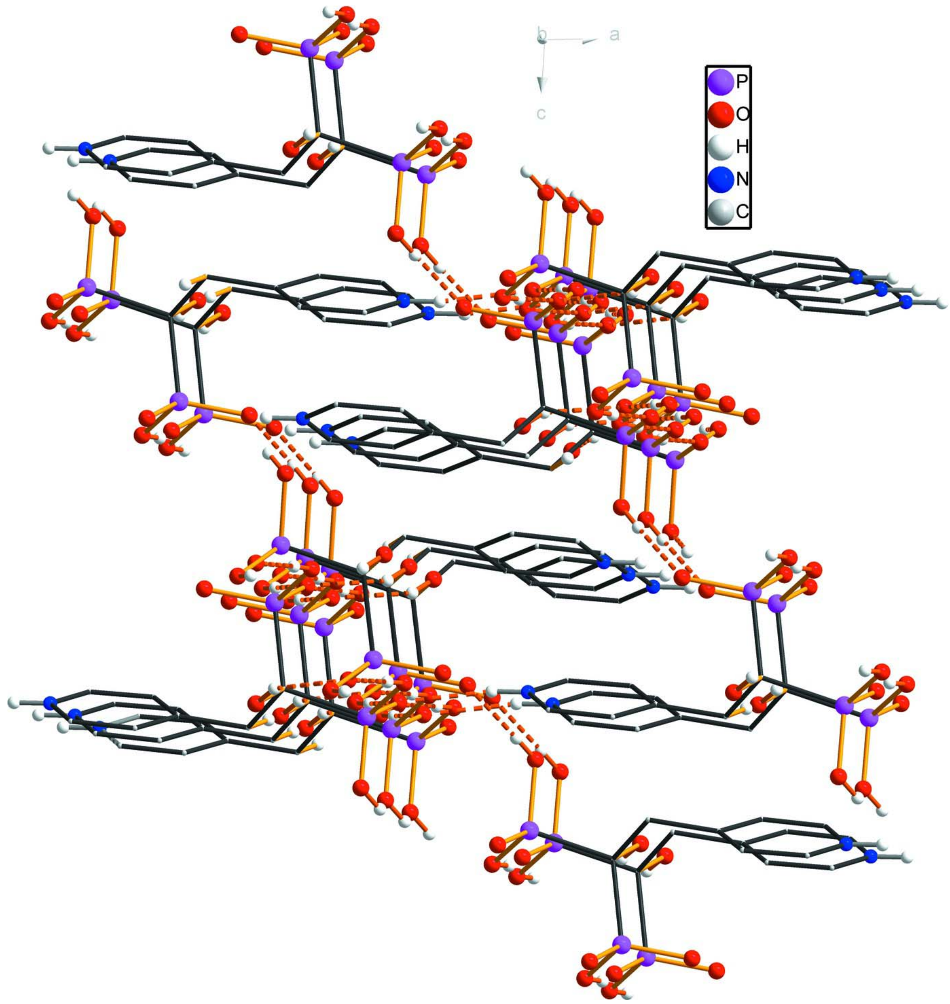

Figure 2

Hydrogen bonds connected three-dimensional structure of the title compound (Carbon-bond H atoms were omitted for clarity).

4-[2-(Hydrogen phosphonato)-2-hydroxy-2-phosphonoethyl]pyridinium

Crystal data

$C_7H_{11}NO_7P_2$
$M_r = 283.11$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.083$ (2) Å
$b = 9.4713$ (19) Å
$c = 11.708$ (2) Å
$\beta = 94.87$ (3)°
$V = 1114.1$ (4) Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.688$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10067 reflections
$\theta = 3.4$–27.6°
$\mu = 0.42$ mm^{-1}
$T = 293$ K
Block, colorless
0.3 × 0.25 × 0.2 mm

Data collection

Rigaku Mercury diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
dtfind.ref scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.880, T_{max} = 0.92
11137 measured reflections
2548 independent reflections
2093 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 27.5°, θ_{min} = 3.3°
$h = -13 \rightarrow 13$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.117
S = 1.02
2548 reflections
174 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 1.150P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.66199 (6)	0.19546 (7)	0.09909 (6)	0.01961 (18)
P2	0.87076 (6)	0.20407 (7)	0.30954 (6)	0.01825 (18)
O1	0.88791 (18)	0.0416 (2)	0.12393 (16)	0.0229 (4)
H1A	0.846 (3)	−0.021 (3)	0.151 (3)	0.041 (10)*
O2	0.81307 (19)	0.3435 (2)	0.34177 (16)	0.0277 (4)
O3	1.01876 (18)	0.1862 (2)	0.33689 (16)	0.0275 (5)
O4	0.79155 (19)	0.0847 (2)	0.36597 (16)	0.0258 (4)
H4A	0.827 (4)	0.008 (3)	0.370 (3)	0.059 (12)*
O5	0.58172 (19)	0.0799 (2)	0.1556 (2)	0.0327 (5)
H5A	0.613 (4)	0.000 (2)	0.157 (3)	0.069 (14)*
O6	0.6632 (2)	0.1660 (2)	−0.02975 (18)	0.0341 (5)
H6A	0.619 (4)	0.218 (4)	−0.072 (3)	0.059 (13)*
O7	0.60449 (17)	0.33631 (19)	0.12698 (17)	0.0269 (5)
N1	1.3345 (2)	0.3264 (3)	0.1400 (2)	0.0362 (6)
H2A	1.4152 (19)	0.334 (4)	0.143 (3)	0.058 (12)*

C2	1.2820 (3)	0.2028 (3)	0.1039 (3)	0.0331 (7)
H2	1.3374	0.1275	0.0894	0.040*
C3	1.1465 (3)	0.1875 (3)	0.0883 (2)	0.0279 (6)
H3	1.1097	0.1020	0.0625	0.033*
C4	1.0641 (3)	0.2998 (3)	0.1109 (2)	0.0221 (6)
C5	1.1232 (3)	0.4260 (3)	0.1492 (3)	0.0334 (7)
H5	1.0705	0.5024	0.1662	0.040*
C6	1.2600 (3)	0.4372 (4)	0.1618 (3)	0.0413 (8)
H6	1.2999	0.5220	0.1855	0.050*
C7	0.9147 (2)	0.2907 (3)	0.0868 (2)	0.0234 (6)
H7A	0.8954	0.2725	0.0055	0.028*
H7B	0.8777	0.3826	0.1023	0.028*
C8	0.8396 (2)	0.1788 (2)	0.1534 (2)	0.0166 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0118 (3)	0.0169 (3)	0.0293 (4)	0.0002 (3)	−0.0028 (2)	−0.0001 (3)
P2	0.0170 (3)	0.0162 (3)	0.0213 (3)	0.0004 (3)	−0.0001 (2)	−0.0024 (3)
O1	0.0193 (9)	0.0172 (10)	0.0326 (10)	0.0020 (8)	0.0050 (8)	−0.0043 (8)
O2	0.0302 (10)	0.0183 (10)	0.0349 (11)	0.0020 (8)	0.0048 (8)	−0.0068 (8)
O3	0.0193 (10)	0.0355 (11)	0.0265 (10)	0.0036 (8)	−0.0060 (7)	−0.0050 (9)
O4	0.0287 (11)	0.0186 (10)	0.0310 (10)	0.0019 (8)	0.0082 (8)	0.0040 (8)
O5	0.0167 (10)	0.0234 (12)	0.0577 (14)	−0.0027 (8)	0.0011 (9)	0.0076 (10)
O6	0.0316 (11)	0.0390 (13)	0.0294 (11)	0.0093 (10)	−0.0116 (9)	−0.0025 (10)
O7	0.0161 (9)	0.0177 (10)	0.0466 (12)	0.0012 (8)	0.0018 (8)	−0.0016 (8)
N1	0.0154 (12)	0.0549 (18)	0.0386 (15)	−0.0072 (12)	0.0046 (10)	−0.0016 (13)
C2	0.0199 (14)	0.0435 (19)	0.0368 (16)	0.0047 (13)	0.0074 (12)	0.0006 (14)
C3	0.0225 (14)	0.0300 (16)	0.0319 (15)	−0.0022 (12)	0.0060 (11)	−0.0039 (12)
C4	0.0180 (13)	0.0260 (14)	0.0229 (13)	−0.0027 (11)	0.0051 (10)	0.0022 (11)
C5	0.0240 (15)	0.0265 (16)	0.0507 (19)	−0.0058 (12)	0.0094 (13)	−0.0057 (14)
C6	0.0262 (16)	0.043 (2)	0.055 (2)	−0.0144 (15)	0.0074 (14)	−0.0112 (16)
C7	0.0144 (12)	0.0248 (14)	0.0311 (14)	−0.0015 (11)	0.0021 (10)	0.0059 (12)
C8	0.0129 (11)	0.0129 (12)	0.0236 (12)	0.0017 (9)	−0.0004 (9)	−0.0007 (10)

Geometric parameters (Å, º)

P1—O7	1.5015 (19)	N1—C2	1.339 (4)
P1—O6	1.535 (2)	N1—H2A	0.815 (19)
P1—O5	1.543 (2)	C2—C3	1.371 (4)
P1—C8	1.855 (2)	C2—H2	0.9300
P2—O2	1.5038 (19)	C3—C4	1.388 (4)
P2—O3	1.5090 (19)	C3—H3	0.9300
P2—O4	1.563 (2)	C4—C5	1.392 (4)
P2—C8	1.844 (3)	C4—C7	1.512 (3)
O1—C8	1.440 (3)	C5—C6	1.379 (4)
O1—H1A	0.806 (18)	C5—H5	0.9300
O4—H4A	0.811 (18)	C6—H6	0.9300

O5—H5A	0.821 (19)	C7—C8	1.551 (3)
O6—H6A	0.81 (4)	C7—H7A	0.9700
N1—C6	1.328 (4)	C7—H7B	0.9700

O7—P1—O6	114.23 (12)	C2—C3—H3	120.0
O7—P1—O5	108.10 (12)	C4—C3—H3	120.0
O6—P1—O5	109.93 (13)	C3—C4—C5	118.2 (2)
O7—P1—C8	112.34 (11)	C3—C4—C7	121.6 (2)
O6—P1—C8	103.49 (12)	C5—C4—C7	120.1 (2)
O5—P1—C8	108.59 (11)	C6—C5—C4	119.8 (3)
O2—P2—O3	116.23 (11)	C6—C5—H5	120.1
O2—P2—O4	107.85 (11)	C4—C5—H5	120.1
O3—P2—O4	111.15 (11)	N1—C6—C5	119.7 (3)
O2—P2—C8	108.99 (11)	N1—C6—H6	120.2
O3—P2—C8	106.16 (11)	C5—C6—H6	120.2
O4—P2—C8	105.97 (11)	C4—C7—C8	117.8 (2)
C8—O1—H1A	112 (2)	C4—C7—H7A	107.9
P2—O4—H4A	116 (3)	C8—C7—H7A	107.9
P1—O5—H5A	117 (3)	C4—C7—H7B	107.9
P1—O6—H6A	117 (3)	C8—C7—H7B	107.9
C6—N1—C2	122.5 (3)	H7A—C7—H7B	107.2
C6—N1—H2A	120 (3)	O1—C8—C7	107.8 (2)
C2—N1—H2A	117 (3)	O1—C8—P2	108.77 (16)
N1—C2—C3	119.8 (3)	C7—C8—P2	111.14 (17)
N1—C2—H2	120.1	O1—C8—P1	109.33 (16)
C3—C2—H2	120.1	C7—C8—P1	105.54 (16)
C2—C3—C4	119.9 (3)	P2—C8—P1	114.04 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{i}	0.81 (2)	2.06 (2)	2.816 (3)	156 (3)
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Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+1/2; (ii) *x*+1, *y*, *z*; (iii) *x*−1/2, −*y*+1/2, *z*−1/2.