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4-Cyanopyridinium dihydrogen phosphate–isonicotinonitrile–phosphoric acid (1/1/1)

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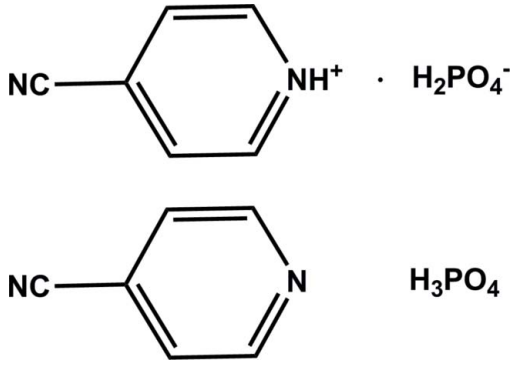
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.096; data-to-parameter ratio = 16.2.

The asymmetric unit of the title compound, $C_6H_5N_2^+\cdot H_2PO_4^-\cdot C_6H_4N_2\cdot H_3PO_4$, contains one 4-cyanopyridinium cation, one $H_2PO_4^-$ anion, one independent isonicotinonitrile molecule and one independent H_3PO_4 molecule. The dihedral angle between the mean planes of the separate protonated and unprotonated pyridine rings is 9.93 (8)°. In the crystal, N—H···O and O—H···N hydrogen bonds and weak C—H···O and C—H···N intermolecular interactions connect the organic molecules into a two-dimensional network parallel to the *ac* plane. O—H···O hydrogen-bonding interactions involving the $H_2PO_4^-$ anions and H_3PO_4 molecules provide additional support from the inorganic groups Weak π–π stacking interactions between the pyridine rings of neighbouring organic molecules [centroid–centroid distances = 3.711 (4) and 3.784 (2) Å] further link the layers into a three-dimensional network.

Related literature

For the properties of related compounds, see: Chen *et al.* (2001); Huang *et al.* (1999); Zhang *et al.* (2001). For related structures, see: Wang *et al.* (2002); Xue *et al.* (2002); Ye *et al.* (2008).



Experimental

Crystal data

$C_6H_5N_2^+\cdot H_2O_4P^-\cdot C_6H_4N_2\cdot H_3O_4P$
M_r = 404.21
Triclinic, $P\overline{1}$
a = 8.1040 (5) Å
b = 8.8872 (9) Å
c = 12.1606 (8) Å
α = 81.491 (1)°
β = 82.009 (1)°
γ = 79.133 (1)°
V = 845.07 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 173 K
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.910, T_{max} = 1.000
8963 measured reflections
3798 independent reflections
3306 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.096
S = 1.14
3798 reflections
235 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O5^{i}	0.82	1.75	2.5576 (14)	169
O4—H4···O3^{ii}	0.82	1.74	2.5611 (14)	176
O6—H6···N1^{iii}	0.82	1.86	2.6749 (17)	178
O7—H7···O1^{iv}	0.82	1.70	2.5150 (15)	173
O8—H8···O3^{ii}	0.82	1.76	2.5795 (15)	177
N3—H3···O1	0.90	1.77	2.6466 (16)	162
C1—H1*A*···O2^{v}	0.95	2.44	3.2549 (19)	144
C8—H8*A*···N2^{vi}	0.95	2.51	3.273 (2)	138
C10—H10*A*···O7^{vii}	0.95	2.31	3.1631 (19)	149
C11—H11*A*···O1^{v}	0.95	2.52	3.3321 (19)	144

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x, -y+1, -z+2$; (iii) $x-1, y+1, z$; (iv) $x, y+1, z$; (v) $-x+1, -y, -z+2$; (vi) $-x+1, -y+1, -z+1$; (vii) $x+1, y-1, z$.

Data collection: *CrystalClear* (Rigaku, 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2136).

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supporting information

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4-Cyanopyridinium dihydrogen phosphate–isonicotinonitrile–phosphoric acid (1/1/1)

Ying-Chun Wang

S1. Comment

Simple organic salts containing strong intrermolecular H-bonds have attracted attention as materials which display ferroelectric-paraelectric phase transitions (Chen *et al.*, 2001; Huang, *et al.* 1999; Zhang, *et al.* 2001). In an effort to obtain phase transition crystals of organic salts, various organic molecules have been studied with a series of new crystal materials (Wang *et al.*, 2002; Xue, *et al.* 2002; Ye *et al.*, 2008). Herewith, we present the synthesis and crystal structure of the title compound, $C_6H_5N_2^+.H_2PO_4^-.C_6H_4N_2.H_3PO_4$,(I).

The asymmetric unit of (I) is comprised of one 4-cyanopyridinium cation, one $H_2PO_4^-$ anion, one independent isonicotinonitrile molecule and one independent H_3PO_4 molecule (Fig. 1). The two separate pyridine rings in the asymmetric unit are almost planar with the largest deviation from the least-squares plane being 0.001 (1) Å and 0.003 (1) Å, respectively. The dihedral angle between the mean planes of the two separate pyridine rings is 9.93 (8)°. Bond lengths and angles in each of these units are in normal ranges.

In the crystal N—H···O and O—H···N hydrogen bonds and weak C—H···O and C—H···N intermolecular interactions bring the organic molecules into a 2D network (Fig. 2). Also, O—H···O hydrogen bonding interactions involving the $H_2PO_4^-$ anions and H_3PO_4 molecules provide additional support for the 2D network from the inorganic groups (Table 1, Fig. 3). In addition, weak π–π stacking interactions between the pyridine rings of neighbouring organic molecules further link the layers into a 3D network (Cg1···Cg2 = 3.711 (4) Å and Cg1···Cg2 = 3.784 (2) Å, where Cg1 and Cg2 are the centroids of the pyridine rings, N1/C1/C2/C3/C4/C5 and N3/C7/C8/C9/C10/C11, respectively).

S2. Experimental

Isonicotinonitrile (10 mmol and stirred at 60°C for 2 h. The precipitate was then filtrated. Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of the solution.

S3. Refinement

H2, H3, H4, H6 and H8 were refined freely. In the last stages of the refinement these atoms were restrained with N3—H3 = 0.90 (2)Å and O2—H2, O4—H4, O6–H6, O8—H8 all = 0.82 (2)Å with U_{iso}(H) = 1.2U_{eq}(N) and U_{iso}(H)=1.5U_{eq}(O). All the remaining H atoms attached to C atoms were placed in calculated positions and then refined using the riding model with C—H lengths of 0.95 Å (CH). The isotropic displcement parameers for these atoms were set to 1.2 (CH) times U_{eq} of the parent atom.

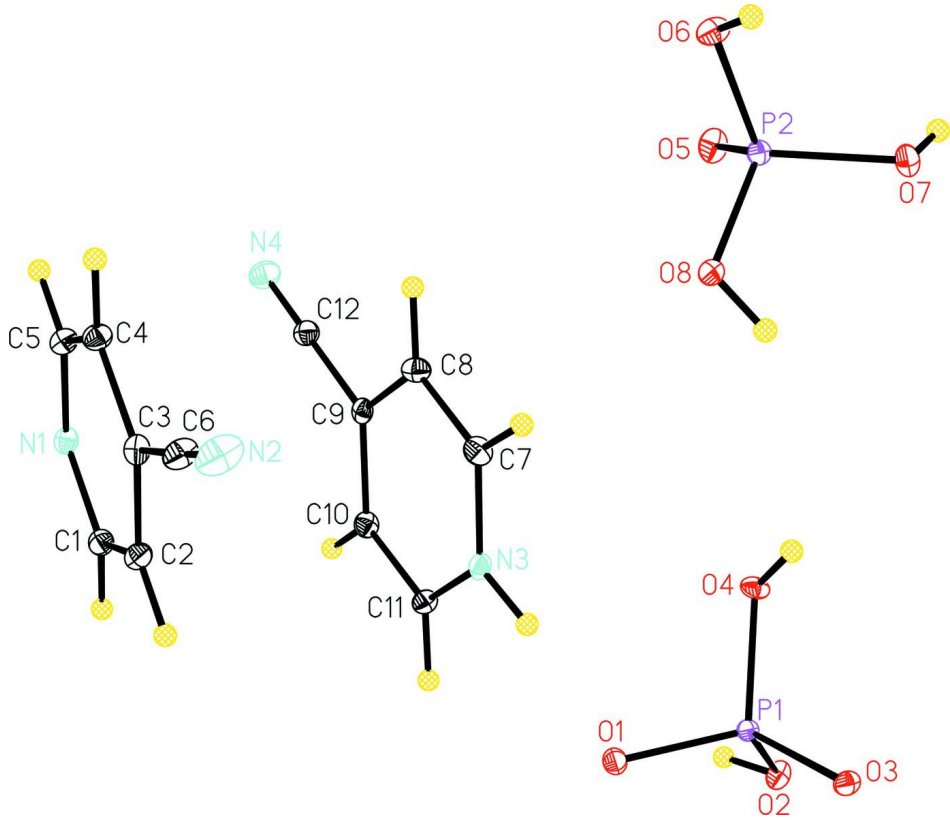

Figure 1

Molecular structure of the title compound showing the atom labeling scheme and 50% probability displacement ellipsoids for one cation-anion unit and bimolecular unit in the asymmetric unit.

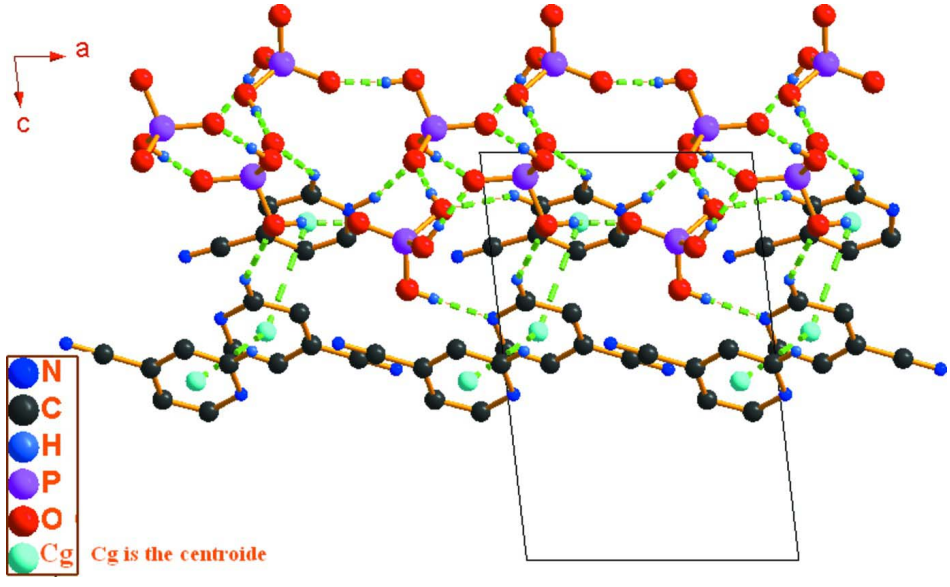

Figure 2

Crystal packing of the title compound viewed along the *b* axis showing O—H···O, O—H···N, hydrogen bonds (dotted lines), weak C—H···O, C—H···N intermolecular interactions (dotted lines) and weak π—π stacking interactions (dashed lines).

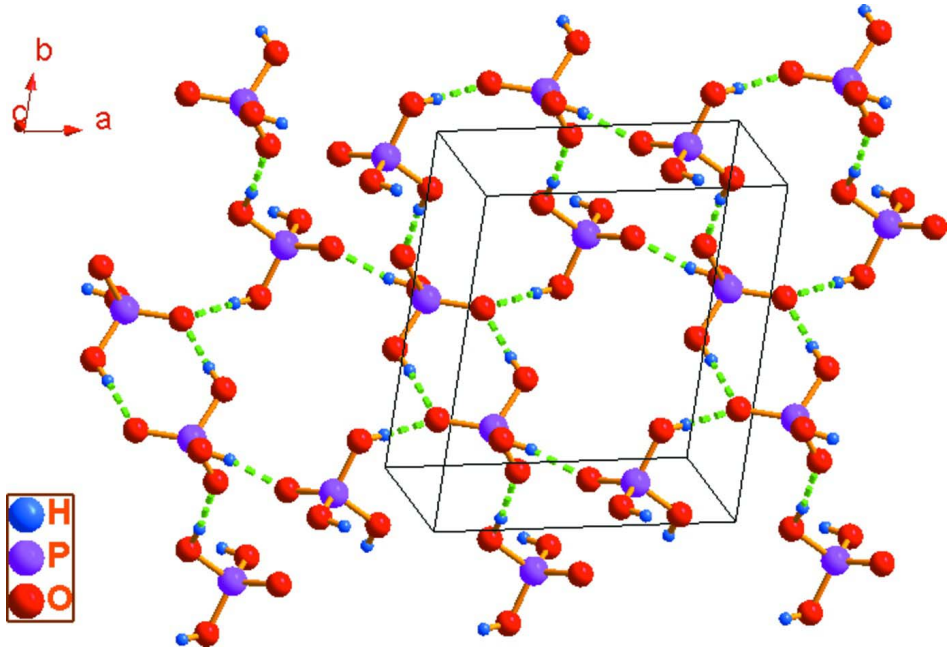

Figure 3

Crystal packing of the title compound viewed along the *c* axis showing the O—H···O hydrogen bonds (dotted line).

4-Cyanopyridinium dihydrogen phosphate–isonicotinonitrile–phosphoric acid (1/1/1)

Crystal data

$C_6H_5N_2^+·H_2O_4P^-·C_6H_4N_2·H_3O_4P$
M_r = 404.21
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.1040 (5) Å
b = 8.8872 (9) Å
c = 12.1606 (8) Å
α = 81.491 (1)°
β = 82.009 (1)°
γ = 79.133 (1)°
V = 845.07 (11) Å^3
Z = 2
$F(000)$ = 416
D_x = 1.589 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3798 reflections
θ = 2.6–27.5°
μ = 0.31 mm^{-1}
T = 173 K
Block, colorless
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD profile fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.910, T_{max} = 1.000
8963 measured reflections
3798 independent reflections
3306 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −10→10
k = −11→11
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.096$
$S = 1.14$
3798 reflections
235 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ 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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	1.02100 (16)	0.12068 (15)	0.59294 (11)	0.0141 (3)
N2	0.39938 (18)	0.32104 (18)	0.46853 (13)	0.0273 (4)
C4	0.83067 (19)	0.30110 (18)	0.48280 (13)	0.0158 (3)
H4A	0.8118	0.3903	0.4294	0.019*
C5	0.98861 (19)	0.24689 (18)	0.51769 (13)	0.0155 (3)
H5A	1.0784	0.3010	0.4873	0.019*
C6	0.5317 (2)	0.27525 (19)	0.49470 (14)	0.0188 (3)
C2	0.73050 (19)	0.09074 (18)	0.60593 (13)	0.0152 (3)
H2A	0.6429	0.0347	0.6378	0.018*
C3	0.69979 (19)	0.22094 (18)	0.52833 (13)	0.0142 (3)
C1	0.89403 (19)	0.04504 (18)	0.63541 (13)	0.0146 (3)
H1A	0.9166	−0.0442	0.6883	0.018*
N3	0.50667 (15)	0.28063 (14)	0.85938 (11)	0.0138 (3)
H3	0.4049	0.2582	0.8897	0.017*
N4	1.12529 (17)	0.40818 (17)	0.74487 (12)	0.0216 (3)
C11	0.63720 (19)	0.18015 (18)	0.89738 (13)	0.0146 (3)
H11A	0.6178	0.0886	0.9447	0.017*
C8	0.6851 (2)	0.44610 (18)	0.75916 (13)	0.0158 (3)
H8A	0.7008	0.5377	0.7107	0.019*
C9	0.82275 (18)	0.34390 (17)	0.79861 (12)	0.0128 (3)
C10	0.79957 (19)	0.20896 (18)	0.86819 (13)	0.0149 (3)
H10A	0.8932	0.1385	0.8949	0.018*
C7	0.52563 (19)	0.41132 (18)	0.79207 (13)	0.0162 (3)
H7A	0.4294	0.4798	0.7671	0.019*
C12	0.9921 (2)	0.37991 (18)	0.76837 (13)	0.0158 (3)

P1	0.15691 (4)	0.29108 (4)	1.05923 (3)	0.00975 (11)
O1	0.24327 (13)	0.17698 (12)	0.97909 (9)	0.0140 (2)
O2	0.21239 (13)	0.23350 (12)	1.17856 (9)	0.0145 (2)
H2	0.3149	0.2034	1.1716	0.022*
O3	−0.03370 (12)	0.32218 (12)	1.06993 (9)	0.0131 (2)
O4	0.22876 (13)	0.44436 (12)	1.01948 (9)	0.0140 (2)
H4	0.1635	0.5162	0.9902	0.021*
P2	0.31115 (5)	0.91637 (4)	0.78249 (3)	0.01113 (11)
O5	0.47632 (13)	0.89331 (13)	0.82709 (9)	0.0184 (3)
O6	0.32346 (13)	1.01043 (13)	0.66430 (9)	0.0163 (2)
H6	0.2317	1.0444	0.6412	0.024*
O7	0.16276 (13)	1.00119 (12)	0.85896 (9)	0.0153 (2)
H7	0.1963	1.0539	0.8984	0.023*
O8	0.24974 (14)	0.76550 (12)	0.76809 (9)	0.0167 (2)
H8	0.1813	0.7401	0.8206	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0147 (6)	0.0160 (6)	0.0114 (7)	−0.0017 (5)	−0.0025 (5)	−0.0017 (5)
N2	0.0158 (7)	0.0357 (9)	0.0274 (9)	−0.0039 (6)	−0.0062 (6)	0.0085 (7)
C4	0.0170 (7)	0.0155 (7)	0.0138 (8)	−0.0025 (6)	−0.0025 (6)	0.0015 (6)
C5	0.0148 (7)	0.0173 (8)	0.0143 (8)	−0.0042 (6)	−0.0015 (6)	−0.0006 (6)
C6	0.0162 (8)	0.0215 (8)	0.0173 (8)	−0.0039 (6)	−0.0015 (6)	0.0027 (6)
C2	0.0154 (7)	0.0172 (8)	0.0134 (8)	−0.0046 (6)	−0.0009 (6)	−0.0013 (6)
C3	0.0129 (7)	0.0169 (7)	0.0128 (8)	−0.0006 (6)	−0.0032 (6)	−0.0029 (6)
C1	0.0175 (8)	0.0144 (7)	0.0113 (7)	−0.0017 (6)	−0.0024 (6)	−0.0006 (6)
N3	0.0107 (6)	0.0164 (6)	0.0145 (7)	−0.0030 (5)	0.0010 (5)	−0.0043 (5)
N4	0.0176 (7)	0.0241 (8)	0.0239 (8)	−0.0074 (6)	−0.0024 (6)	−0.0010 (6)
C11	0.0155 (7)	0.0148 (7)	0.0130 (8)	−0.0034 (6)	−0.0005 (6)	−0.0004 (6)
C8	0.0185 (8)	0.0148 (7)	0.0149 (8)	−0.0049 (6)	−0.0039 (6)	0.0000 (6)
C9	0.0131 (7)	0.0159 (7)	0.0111 (7)	−0.0038 (6)	−0.0012 (6)	−0.0057 (6)
C10	0.0131 (7)	0.0147 (7)	0.0160 (8)	0.0004 (6)	−0.0025 (6)	−0.0013 (6)
C7	0.0147 (7)	0.0155 (7)	0.0181 (8)	−0.0001 (6)	−0.0053 (6)	−0.0013 (6)
C12	0.0178 (8)	0.0167 (8)	0.0136 (8)	−0.0044 (6)	−0.0023 (6)	−0.0019 (6)
P1	0.00801 (19)	0.00916 (19)	0.0115 (2)	−0.00048 (14)	−0.00172 (14)	−0.00001 (14)
O1	0.0128 (5)	0.0137 (5)	0.0159 (6)	−0.0019 (4)	−0.0008 (4)	−0.0043 (4)
O2	0.0110 (5)	0.0181 (6)	0.0123 (6)	0.0013 (4)	−0.0024 (4)	0.0014 (4)
O3	0.0088 (5)	0.0123 (5)	0.0169 (6)	−0.0009 (4)	−0.0020 (4)	0.0019 (4)
O4	0.0108 (5)	0.0093 (5)	0.0213 (6)	−0.0010 (4)	−0.0052 (4)	0.0021 (4)
P2	0.00841 (19)	0.0120 (2)	0.0126 (2)	−0.00073 (14)	−0.00240 (14)	−0.00054 (15)
O5	0.0098 (5)	0.0251 (6)	0.0198 (6)	0.0011 (4)	−0.0054 (4)	−0.0024 (5)
O6	0.0110 (5)	0.0196 (6)	0.0164 (6)	−0.0024 (4)	−0.0037 (4)	0.0049 (4)
O7	0.0109 (5)	0.0162 (5)	0.0206 (6)	−0.0022 (4)	−0.0016 (4)	−0.0082 (4)
O8	0.0186 (6)	0.0138 (5)	0.0173 (6)	−0.0051 (4)	0.0047 (4)	−0.0037 (4)

Geometric parameters (Å, º)

N1—C1	1.337 (2)	C8—C9	1.392 (2)
N1—C5	1.3477 (19)	C8—H8A	0.9500
N2—C6	1.144 (2)	C9—C10	1.389 (2)
C4—C5	1.381 (2)	C9—C12	1.453 (2)
C4—C3	1.394 (2)	C10—H10A	0.9500
C4—H4A	0.9500	C7—H7A	0.9500
C5—H5A	0.9500	P1—O3	1.5077 (10)
C6—C3	1.450 (2)	P1—O1	1.5176 (11)
C2—C3	1.387 (2)	P1—O2	1.5635 (11)
C2—C1	1.391 (2)	P1—O4	1.5666 (11)
C2—H2A	0.9500	O2—H2	0.8195
C1—H1A	0.9500	O4—H4	0.8198
N3—C11	1.3370 (19)	P2—O5	1.4811 (11)
N3—C7	1.339 (2)	P2—O6	1.5526 (11)
N3—H3	0.9008	P2—O8	1.5560 (11)
N4—C12	1.142 (2)	P2—O7	1.5601 (11)
C11—C10	1.376 (2)	O6—H6	0.8196
C11—H11A	0.9500	O7—H7	0.8208
C8—C7	1.377 (2)	O8—H8	0.8198
C1—N1—C5	118.22 (13)	C10—C9—C12	119.62 (14)
C5—C4—C3	117.97 (14)	C8—C9—C12	119.72 (14)
C5—C4—H4A	121.0	C11—C10—C9	118.16 (14)
C3—C4—H4A	121.0	C11—C10—H10A	120.9
N1—C5—C4	122.96 (14)	C9—C10—H10A	120.9
N1—C5—H5A	118.5	N3—C7—C8	119.76 (14)
C4—C5—H5A	118.5	N3—C7—H7A	120.1
N2—C6—C3	178.62 (18)	C8—C7—H7A	120.1
C3—C2—C1	117.73 (14)	N4—C12—C9	179.83 (17)
C3—C2—H2A	121.1	O3—P1—O1	115.74 (6)
C1—C2—H2A	121.1	O3—P1—O2	108.01 (6)
C2—C3—C4	119.98 (14)	O1—P1—O2	109.65 (6)
C2—C3—C6	120.42 (14)	O3—P1—O4	110.60 (6)
C4—C3—C6	119.60 (14)	O1—P1—O4	106.72 (6)
N1—C1—C2	123.16 (14)	O2—P1—O4	105.66 (6)
N1—C1—H1A	118.4	P1—O2—H2	107.9
C2—C1—H1A	118.4	P1—O4—H4	115.8
C11—N3—C7	122.80 (13)	O5—P2—O6	109.29 (6)
C11—N3—H3	113.8	O5—P2—O8	115.10 (6)
C7—N3—H3	123.0	O6—P2—O8	105.90 (6)
N3—C11—C10	120.21 (14)	O5—P2—O7	113.15 (6)
N3—C11—H11A	119.9	O6—P2—O7	109.13 (6)
C10—C11—H11A	119.9	O8—P2—O7	103.84 (6)
C7—C8—C9	118.43 (15)	P2—O6—H6	114.0
C7—C8—H8A	120.8	P2—O7—H7	111.9
C9—C8—H8A	120.8	P2—O8—H8	112.0

C10—C9—C8	120.65 (14)		
C1—N1—C5—C4	0.0 (2)	C7—N3—C11—C10	0.3 (2)
C3—C4—C5—N1	0.2 (2)	C7—C8—C9—C10	0.9 (2)
C1—C2—C3—C4	0.0 (2)	C7—C8—C9—C12	−177.97 (14)
C1—C2—C3—C6	−179.54 (14)	N3—C11—C10—C9	−0.2 (2)
C5—C4—C3—C2	−0.2 (2)	C8—C9—C10—C11	−0.5 (2)
C5—C4—C3—C6	179.34 (14)	C12—C9—C10—C11	178.46 (14)
C5—N1—C1—C2	−0.2 (2)	C11—N3—C7—C8	0.2 (2)
C3—C2—C1—N1	0.2 (2)	C9—C8—C7—N3	−0.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O5^{i}	0.82	1.75	2.5576 (14)	169
O4—H4···O3^{ii}	0.82	1.74	2.5611 (14)	176
O6—H6···N1^{iii}	0.82	1.86	2.6749 (17)	178
O7—H7···O1^{iv}	0.82	1.70	2.5150 (15)	173
O8—H8···O3^{ii}	0.82	1.76	2.5795 (15)	177
N3—H3···O1	0.90	1.77	2.6466 (16)	162
C1—H1*A*···O2^{v}	0.95	2.44	3.2549 (19)	144
C8—H8*A*···N2^{vi}	0.95	2.51	3.273 (2)	138
C10—H10*A*···O7^{vii}	0.95	2.31	3.1631 (19)	149
C11—H11*A*···O1^{v}	0.95	2.52	3.3321 (19)	144

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) −*x*, −*y*+1, −*z*+2; (iii) *x*−1, *y*+1, *z*; (iv) *x*, *y*+1, *z*; (v) −*x*+1, −*y*, −*z*+2; (vi) −*x*+1, −*y*+1, −*z*+1; (vii) *x*+1, *y*−1, *z*.