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2-Amino-3-carboxypyrazin-1-ium dihydrogen phosphate

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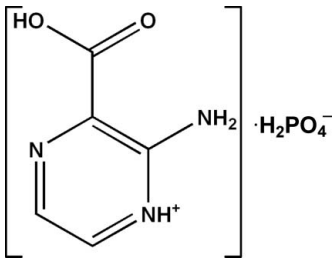
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.079; data-to-parameter ratio = 14.4.

In the crystal structure of the title compound, $C_5H_6N_3O_2^+\cdot H_2PO_4^-$, the dihydrogen phosphate anions are linked through short O—H···O hydrogen bonds, forming infinite double chains running parallel to the b axis. Centrosymetric N—H···O hydrogen-bonded cationic dimers form bridges between these chains by means of intermolecular N—H···O and O—H···O hydrogen bonds, leading to a two-dimensional network parallel to (100) in which $R_3^3(12)$, $R_4^3(10)$ $R_2^2(8)$ and $C(4)$ graph-set motifs are generated. Weak intermolecular C—H···O hydrogen bonds connect these layers, forming a three-dimensional network.

Related literature

For hybrid compounds based on *N*-heterocycles, see: Akriche & Rzaigui (2007); Berrah *et al.* (2011*a*,*b*,*c*); Ouakkaf *et al.* (2011). For related dihydrogenphosphte compounds, see: Lin *et al.* (2009); Shao *et al.* (2010). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$C_5H_6N_3O_2^+\cdot H_2PO_4^-$
M_r = 237.11
Monoclinic, $P2_1/c$
a = 8.6076 (5) Å
b = 4.6703 (3) Å
c = 21.9431 (13) Å
β = 95.573 (2)°
V = 877.94 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 150 K
0.45 × 0.06 × 0.04 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.898, T_{max} = 0.987
7993 measured reflections
2004 independent reflections
1781 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.079
S = 1.04
2004 reflections
139 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O14	0.88	1.94	2.8171 (17)	171
N1—H1B···O9	0.88	2.09	2.7275 (17)	128
N1—H1B···O9^{i}	0.88	2.37	3.0640 (19)	136
N3—H3···O11	0.88	1.79	2.6690 (16)	173
O10—H10···O13^{ii}	0.84	1.83	2.6591 (16)	169
O12—H12···O11^{iii}	0.84	1.72	2.5386 (14)	166
O13—H13···O14^{iv}	0.84	1.64	2.4634 (16)	164
C4—H4···O11^{v}	0.95	2.43	3.3377 (19)	160

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

Data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5248).

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supplementary materials

Acta Cryst. (2011). E**67**, o1409-o1410 [**doi:10.1107/S1600536811017521**]

2-Amino-3-carboxypyrazin-1-ium dihydrogen phosphate

F. Berrah, S. Bouacida and T. Roisnel

Comment

In continuation of our search for new hybrids based on protonated N-heterocyclic compounds and inorganic acids we have prepared the title compound. Our recent investigation in this field has revealed the ability of N-heterocyclic derivatives to generate original networks stabilized by hydrogen bonds and has shown how anion substitution may influence the hydrogen-bonding patterns (Berrah *et al.*, 2011*a*,*b*,*c*; Ouakkaf *et al.*, 2011).

The asymmetric unit of the title conpound compound contains one 2-amino-3-carboxypyrazin-1-ium cation and one dihydrogen phosphate anion (Fig. 1). Both entities display geometry similar to that reported in related compounds (Akriche & Rzaigui 2007; Berrah *et al.*, 2011*b*; Shao *et al.*, 2010). dihydrogen phosphate anions linked through strong O—H···O hydrogen bonds (Table 1), form double infinite chains running parallel to the *b* axis (Fig. 2). Similar chains were previously observed in related compounds (Akriche & Rzaigui 2007; Lin *et al.*, 2009). 2-Amino-3-carboxypyrazin-1-ium centrosymetric dimers form bridges between these chains by means of N—H···O and O—H···O hydrogen bonds (Fig. 3) leading to a two-dimensional network (Fig. 4) where $R^3_3(12)$, $R^3_4(10)$, $R^2_2(8)$ and C(4) graph-set motifs are generated (Fig. 2 and Fig. 3)(Etter *et al.*, 1990; Bernstein *et al.*, 1995). Further stabilization is provided by intermolecular C—H···O contacts.

Experimental

The title compound was synthesized by reacting 3-amino-pyrazine-2-carboxylic acid with phosphoricic acid in a solution of equal volume of H_2O and CH_3OH. Slow evaporation leads to well crystallized colourless needles.

Refinement

H atoms were located in Fourier maps but introduced in calculated positions and treated as riding on their parent atoms (C, N or O) with C—H = 0.95 Å, O—H = 0.84 Å and N—H = 0.88 Å with U_{iso}(H) = 1.2 U_{eq}(C or N) and U_{iso}(H = 1.5 U_{eq}(O).

Figures

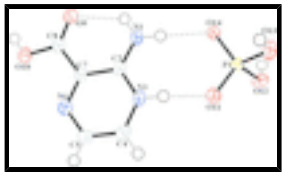

Fig. 1. The asymmetric unit of the title compound. Displacement are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

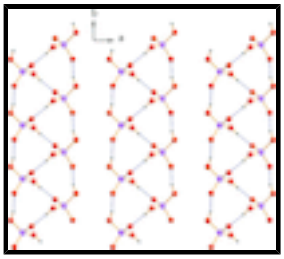

Fig. 2. Part of the crystal structure viewed along [001] showing infinite double chains. Hydrogen bonds are shown as dashed lines.

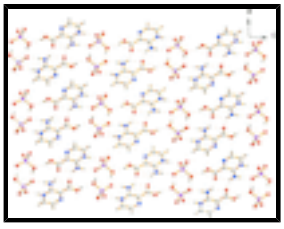

Fig. 3. A view parallel to (010) showing cationic dimers and how they link double infinite anionic chains. C—H···O contacts have been omitted for clarity.

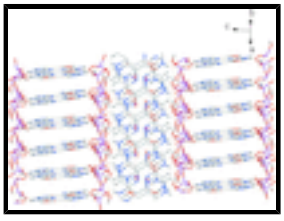

Fig. 4. The two-dimensional packing. Hydrogen bonds are shown as dashed lines.

2-Amino-3-carboxypyrazin-1-ium dihydrogen phosphate

Crystal data

$C_5H_6N_3O_2^+·H_2PO_4^-$	$F(000) = 488$
$M_r = 237.11$	$D_x = 1.794$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 4062 reflections
$a = 8.6076$ (5) Å	$\theta = 3.2$–$27.5°$
$b = 4.6703$ (3) Å	$\mu = 0.33$ mm^{-1}
$c = 21.9431$ (13) Å	$T = 150$ K
$\beta = 95.573$ (2)°	Needle, colourless
$V = 877.94$ (9) Å^3	0.45 × 0.06 × 0.04 mm
$Z = 4$	

Data collection

Bruker APEXII diffractometer	1781 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.025$
CCD rotation images, thin slices scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)	$h = -11 \rightarrow 7$
$T_{min} = 0.898$, $T_{max} = 0.987$	$k = -6 \rightarrow 6$
7993 measured reflections	$l = -28 \rightarrow 28$
2004 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.028$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.079$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.6558P]$ where $P = (F_o^2 + 2F_c^2)/3$
2004 reflections	$(\Delta/\sigma)_{max} = 0.001$
139 parameters	$\Delta\rho_{max} = 0.39$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.39$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.86142 (15)	0.8016 (3)	0.90992 (6)	0.0173 (3)
H1A	0.8733	0.8741	0.8736	0.021*
H1B	0.9191	0.8645	0.9425	0.021*
C2	0.75718 (17)	0.5988 (3)	0.91547 (6)	0.0137 (3)
N3	0.66953 (15)	0.5047 (3)	0.86483 (5)	0.0149 (3)
H3	0.6834	0.583	0.8293	0.018*
C4	0.56229 (17)	0.2967 (3)	0.86666 (7)	0.0165 (3)
H4	0.5045	0.2339	0.83	0.02*
C5	0.53675 (17)	0.1756 (3)	0.92166 (7)	0.0171 (3)
H5	0.4608	0.0288	0.923	0.02*
N6	0.61838 (15)	0.2626 (3)	0.97404 (6)	0.0166 (3)
C7	0.72487 (17)	0.4646 (3)	0.97204 (6)	0.0142 (3)
C8	0.81279 (17)	0.5559 (3)	1.03115 (7)	0.0155 (3)
O9	0.91059 (13)	0.7446 (2)	1.03400 (5)	0.0214 (3)
O10	0.77252 (13)	0.4096 (3)	1.07821 (5)	0.0212 (3)
H10	0.8219	0.4712	1.1104	0.032*
P1	0.79097 (4)	0.97152 (8)	0.740127 (16)	0.01125 (11)
O11	0.70388 (12)	0.7004 (2)	0.75265 (5)	0.0161 (2)

O12	0.66950 (12)	1.1937 (2)	0.71167 (5)	0.0162 (2)
H12	0.696	1.3588	0.7238	0.024*
O13	0.89962 (12)	0.9251 (2)	0.68787 (5)	0.0183 (2)
H13	0.9697	0.8065	0.6994	0.027*
O14	0.88101 (12)	1.0854 (2)	0.79764 (5)	0.0158 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0199 (6)	0.0194 (7)	0.0125 (6)	−0.0029 (5)	0.0013 (5)	0.0017 (5)
C2	0.0139 (7)	0.0139 (7)	0.0134 (6)	0.0041 (5)	0.0019 (5)	−0.0008 (5)
N3	0.0177 (6)	0.0157 (6)	0.0113 (6)	0.0025 (5)	0.0013 (5)	0.0010 (5)
C4	0.0150 (7)	0.0166 (7)	0.0173 (7)	0.0029 (6)	−0.0014 (6)	−0.0021 (6)
C5	0.0141 (7)	0.0177 (7)	0.0193 (7)	−0.0006 (6)	0.0015 (6)	−0.0016 (6)
N6	0.0160 (6)	0.0182 (6)	0.0158 (6)	0.0017 (5)	0.0027 (5)	0.0000 (5)
C7	0.0144 (7)	0.0160 (7)	0.0123 (6)	0.0027 (5)	0.0020 (5)	−0.0003 (5)
C8	0.0160 (7)	0.0172 (7)	0.0135 (7)	0.0028 (6)	0.0023 (5)	−0.0006 (5)
O9	0.0245 (6)	0.0223 (6)	0.0168 (5)	−0.0050 (5)	−0.0003 (4)	−0.0013 (4)
O10	0.0233 (6)	0.0296 (6)	0.0106 (5)	−0.0056 (5)	0.0011 (4)	0.0010 (4)
P1	0.01218 (19)	0.01066 (18)	0.01087 (18)	0.00067 (13)	0.00093 (13)	−0.00036 (13)
O11	0.0213 (5)	0.0112 (5)	0.0156 (5)	−0.0021 (4)	0.0010 (4)	−0.0006 (4)
O12	0.0158 (5)	0.0107 (5)	0.0212 (5)	0.0021 (4)	−0.0025 (4)	−0.0026 (4)
O13	0.0182 (5)	0.0239 (6)	0.0132 (5)	0.0094 (4)	0.0034 (4)	0.0031 (4)
O14	0.0168 (5)	0.0181 (5)	0.0121 (5)	−0.0043 (4)	0.0004 (4)	0.0003 (4)

Geometric parameters (Å, °)

N1—C2	1.319 (2)	N6—C7	1.319 (2)
N1—H1A	0.88	C7—C8	1.4987 (19)
N1—H1B	0.88	C8—O9	1.2161 (19)
C2—N3	1.3543 (18)	C8—O10	1.3127 (18)
C2—C7	1.442 (2)	O10—H10	0.84
N3—C4	1.343 (2)	P1—O11	1.5101 (11)
N3—H3	0.88	P1—O14	1.5120 (10)
C4—C5	1.370 (2)	P1—O12	1.5597 (11)
C4—H4	0.95	P1—O13	1.5636 (11)
C5—N6	1.3503 (19)	O12—H12	0.84
C5—H5	0.95	O13—H13	0.84
C2—N1—H1A	120	N6—C7—C2	122.16 (13)
C2—N1—H1B	120	N6—C7—C8	117.96 (13)
H1A—N1—H1B	120	C2—C7—C8	119.88 (13)
N1—C2—N3	119.16 (13)	O9—C8—O10	124.84 (14)
N1—C2—C7	125.57 (13)	O9—C8—C7	122.65 (14)
N3—C2—C7	115.26 (13)	O10—C8—C7	112.51 (13)
C4—N3—C2	122.68 (13)	C8—O10—H10	109.5
C4—N3—H3	118.7	O11—P1—O14	111.49 (6)
C2—N3—H3	118.7	O11—P1—O12	107.77 (6)
N3—C4—C5	119.62 (14)	O14—P1—O12	111.69 (6)

N3—C4—H4	120.2	O11—P1—O13	111.11 (6)
C5—C4—H4	120.2	O14—P1—O13	111.48 (6)
N6—C5—C4	120.73 (14)	O12—P1—O13	102.94 (6)
N6—C5—H5	119.6	P1—O12—H12	109.5
C4—C5—H5	119.6	P1—O13—H13	109.5
C7—N6—C5	119.53 (13)		
N1—C2—N3—C4	179.23 (13)	N3—C2—C7—N6	0.6 (2)
C7—C2—N3—C4	−1.4 (2)	N1—C2—C7—C8	0.5 (2)
C2—N3—C4—C5	1.2 (2)	N3—C2—C7—C8	−178.86 (12)
N3—C4—C5—N6	−0.1 (2)	N6—C7—C8—O9	−178.37 (14)
C4—C5—N6—C7	−0.6 (2)	C2—C7—C8—O9	1.1 (2)
C5—N6—C7—C2	0.4 (2)	N6—C7—C8—O10	1.9 (2)
C5—N6—C7—C8	179.86 (13)	C2—C7—C8—O10	−178.58 (13)
N1—C2—C7—N6	179.93 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O14	0.88	1.94	2.8171 (17)	171
N1—H1B···O9	0.88	2.09	2.7275 (17)	128
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C4—H4···O11^{v}	0.95	2.43	3.3377 (19)	160

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+2; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) *x*, *y*+1, *z*; (iv) −*x*+2, *y*−1/2, −*z*+3/2; (v) −*x*+1, *y*−1/2, −*z*+3/2.

Fig. 1

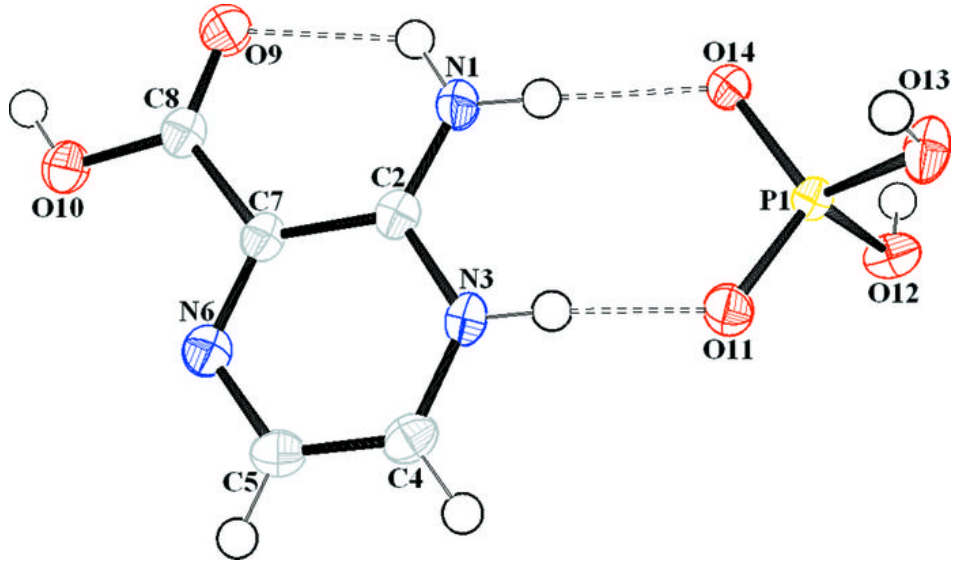

Fig. 2

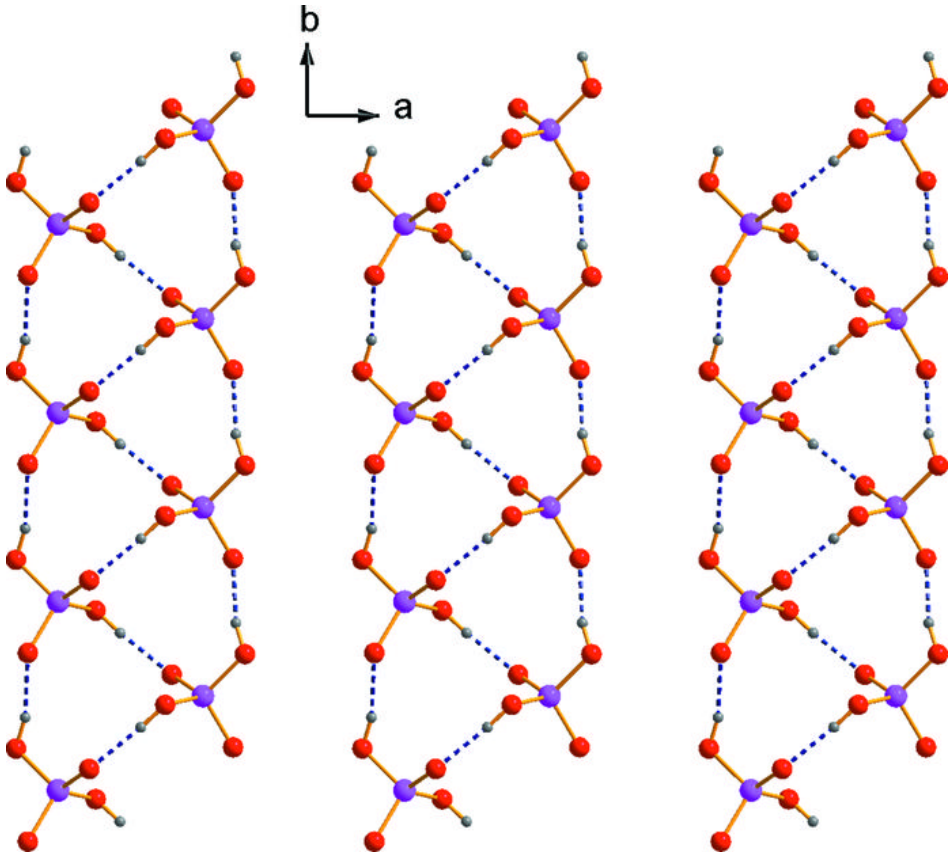

Fig. 3

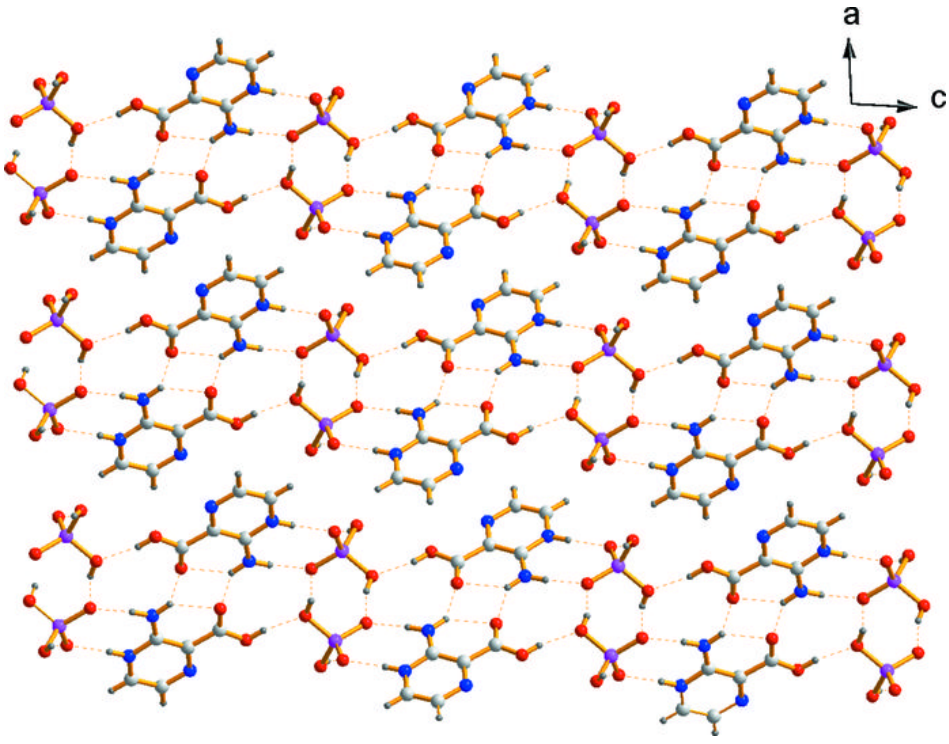

Fig. 4