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Quinoxaline–dihydroxyacetic acid (1/1)

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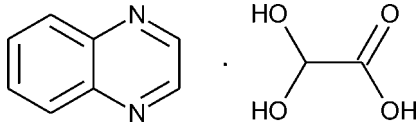
Key indicators: single-crystal X-ray study; T = 130 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.050; wR factor = 0.109; data-to-parameter ratio = 11.3.

In the title 1:1 cocrystal of quinoxaline with dihydroxyacetic acid, $C_8H_6N_2{\cdot}C_2H_4O_4$, both N atoms of the heterocycle are involved in O—H···N hydrogen bonds. The carboxyl group is linked to the quinoxaline molecule by O—H···N and C—H···O interactions, generating a cyclic $R_2^2(7)$ motif. One of the acid hydroxy groups interacts with the quinoxaline N atom, whereas the other forms an infinite chain of O—H···O hydrogen bonds with the hydroxyl H atom disordered equally over two positions. The quinoxaline molecules are arranged into infinite columns by π–π stacking interactions [interplanar distance = 3.427 (7) Å].

Related literature

For the crystal structure of dihydroxyacetic acid, see: Lis (1982). For crystal structures of quinoxaline complexes with carboxylic acids, see: Czapik & Gdaniec (2007); Jankowski *et al.* (2007); Olenik *et al.* (2003).



Experimental

Crystal data

$C_8H_6N_2{\cdot}C_2H_4O_4$
M_r = 222.20
Triclinic, $P\overline{1}$
a = 4.0384 (9) Å
b = 10.406 (4) Å
c = 12.052 (3) Å
α = 88.83 (2)°
β = 83.981 (18)°
γ = 82.50 (2)°
V = 499.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 130.0 (2) K
0.50 × 0.15 × 0.07 mm

Data collection

Kuma KM-4-CCD κ-geometry diffractometer
Absorption correction: none
4271 measured reflections
1760 independent reflections
1482 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.109
S = 1.23
1760 reflections
156 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···N1A	1.01 (4)	1.71 (4)	2.722 (3)	179 (3)
O3—H3O···O3^{i}	0.85	1.87	2.678 (4)	157
O3—H3O′···O3^{ii}	0.85	1.97	2.763 (5)	156
O4—H4O···N4A^{iii}	0.93 (4)	1.86 (4)	2.780 (3)	173 (3)
C2A—H2A···O2	0.93	2.63	3.287 (3)	128
C2—H2···O4^{iv}	0.98	2.43	3.390 (3)	166
C3A—H3A···O2^{v}	0.93	2.36	3.138 (3)	141

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989) and *Mercury* (Version 1.4; Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Polish Ministry of Education and Science (grant No. 3 T09A 11327).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2434).

References

Czapik, A. & Gdaniec, M. (2007). *Acta Cryst.* E**63**, o1480–o1482.
Jankowski, W., Kadzewski, A. & Gdaniec, M. (2007). *Pol. J. Chem.* **81**, 1095–1108.
Lis, T. (1983). *Acta Cryst.* C**39**, 1082–1084.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Olenik, B., Smolka, T., Boese, R. & Sustmann, R. (2003). *Cryst. Growth Des.* **3**, 183–188.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Version 1.171.31 Oxford Diffraction, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Siemens (1989). *Stereochemical Workstation Operation Manual.* Release 3.4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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supplementary materials

Acta Cryst. (2007). E**63**, o3081 [**doi:10.1107/S1600536807025792**]

Quinoxaline-dihydroxyacetic acid (1/1)

A. Czapik and M. Gdaniec

Comment

In the chemical literature dihydroxyacetic acid is often referred to as glyoxalic acid monohydrate. The latter name is also used by the chemical companies selling this compound. Already some 25 years ago Lis (1983) published the crystal structure of 'glyoxalic acid monohydrate' and clearly showed that the compound is not a monohydrate but a product of the reaction of glyoxalic acid with water, namely dihydroxyacetic acid. Interestingly, this simple and small molecule, which can act as a triple donor in hydrogen bonding, has not been applied as reagent in supramolecular chemistry up till now.

In course of our studies on molecular complexes of azaaromatic heterocycles we cocrystallized quinoxaline with dihydroxyacetic acid obtaining the title molecular complex, (I), of 1:1 stoichiometry. It is evident from the crystal structure of the complex that the acid cocrystallizes with the aromatic base in the form of dihydroxyacetic acid (Fig. 1).

Crystal packing of (I) is shown in Fig. 2. The dihydroxyacetic acid molecules interact with the two N atoms of the base *via* the carboxylic group and one hydroxy group forming a chain parallel to [110]. The carboxylic group is approximately coplanar with the aromatic base and is linked to the quinoxaline molecule by N—H···O and C—H···O interactions generating the cyclic $R_2^2(7)$ motif (Fig. 2, Table 1). The supramolecular chains are further assembled into a three-dimensional network *via* ···O—H···O—H··· hydrogen bonds joining the hydroxy groups of the acid. The hydrogen atom involved in this interaction is disordered over two positions, corresponding to two different directions of the ···O—H···O—H··· hydrogen-bonding chain (Fig. 3). The three-dimensional network of molecules is additionaly stabilized by weaker C—H···O interactions (Table 1). The quinoxaline molecules are arranged into infinite columns by π–π stacking interactions, with the interplanar distance of 3.427 (7) Å between the best planes of the neighbouring molecules.

Experimental

The title compound was obtained by dissolving equimolar amounts of quinoxaline (Aldrich) and dihydroxyacetic acid (glyoxalic acid monohydrate, Aldrich) in acetone and slow evaporation of the solution to yield colourles needles of (I).

Refinement

All the H atoms were located in difference maps. The C-bonded H atoms were placed at calculated positions, with C—H = 0.93 Å, and were refined as riding with U_{iso}(H) = 1.2Ueq(C). The atoms H1O and H4O from the carboxy group and one of the hydroxy groups were freely refined. The H atom bonded to O3 was disordered over two positions. The half-occupancy was assumed for each position and the H3O and H3O' were refined as riding on O3 (O—H = 0.85 Å) with the isotropic displacement parameters refined.

Figures

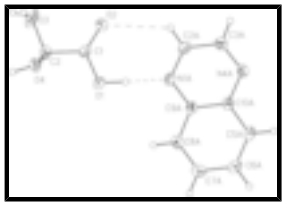

Fig. 1. : The molecular structure of (I) with displacement ellipsoids shown at the 50% probability level (arbitrary spheres for the H atoms). Hydrogen bonds are shown as dashed lines.

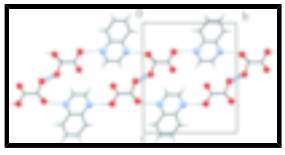

Fig. 2. : Crystal packing viewed for (I) viewed down the *a* axis. Hydrogen bonds are shown with dashed lines.

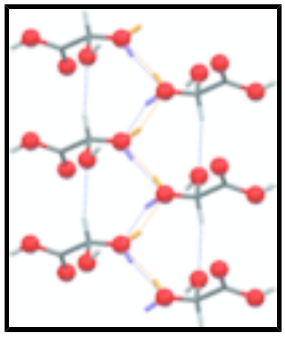

Fig. 3. Hydrogen-bonding interactions between the dihydroxyacetic acid molecules: the infinite O—H···O—H··· chain together with the supporting C—H···O interaction. The two positions of the disordered H atom are shown in different colours. Hydrogen bonds are shown with dashed lines.

Quinoxaline–dihydroxyacetic acid (1/1)

Crystal data

$C_8H_6N_2·C_2H_4O_4$	$Z = 2$
$M_r = 222.20$	$F_{000} = 232$
Triclinic, $P\overline{1}$	$D_x = 1.478$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 4.0384$ (9) Å	Cell parameters from 1787 reflections
$b = 10.406$ (4) Å	$\theta = 4$–25º
$c = 12.052$ (3) Å	$\mu = 0.12$ mm^{-1}
$\alpha = 88.83$ (2)º	$T = 130.0$ (2) K
$\beta = 83.981$ (18)º	Needle, colourless
$\gamma = 82.50$ (2)º	0.50 × 0.15 × 0.07 mm
$V = 499.4$ (3) Å^3	

Data collection

Kuma KM-4-CCD κ-geometry diffractometer	1482 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.014$
Monochromator: graphite	$\theta_{max} = 25.0$º
$T = 130.0(2)$ K	$\theta_{min} = 4.3$º

ω scans
Absorption correction: none
4271 measured reflections
1760 independent reflections

$h = -4 \rightarrow 4$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.109$
$S = 1.23$
1760 reflections
156 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0135P)^2 + 0.6727P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.022 (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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
N1A	0.6041 (6)	0.4249 (2)	0.74215 (17)	0.0230 (5)	
C2A	0.8100 (7)	0.3827 (3)	0.6554 (2)	0.0259 (6)	
H2A	0.8333	0.4366	0.5934	0.031*	
C3A	0.9982 (7)	0.2579 (2)	0.6529 (2)	0.0253 (6)	
H3A	1.1405	0.2322	0.5893	0.030*	
N4A	0.9772 (5)	0.17718 (19)	0.73826 (17)	0.0219 (5)	
C5A	0.7358 (7)	0.1375 (2)	0.9245 (2)	0.0239 (6)	
H5A	0.8556	0.0548	0.9233	0.029*	
C6A	0.5324 (7)	0.1800 (3)	1.0178 (2)	0.0268 (6)	
H6A	0.5193	0.1266	1.0806	0.032*	
C7A	0.3426 (7)	0.3036 (3)	1.0201 (2)	0.0267 (6)	
H7A	0.2033	0.3310	1.0839	0.032*	

C8A	0.3625 (6)	0.3834 (2)	0.9289 (2)	0.0231 (6)	
H8A	0.2343	0.4645	0.9304	0.028*	
C9A	0.5765 (6)	0.3433 (2)	0.83244 (19)	0.0191 (5)	
C10A	0.7644 (6)	0.2187 (2)	0.8300 (2)	0.0194 (5)	
C1	0.3848 (6)	0.7312 (2)	0.6450 (2)	0.0211 (6)	
C2	0.2222 (7)	0.8718 (2)	0.6412 (2)	0.0243 (6)	
H2	−0.0171	0.8771	0.6677	0.029*	
O1	0.2775 (5)	0.67053 (18)	0.73625 (15)	0.0294 (5)	
H1O	0.396 (9)	0.579 (4)	0.739 (3)	0.060 (11)*	
O2	0.5905 (5)	0.68219 (19)	0.57360 (16)	0.0386 (6)	
O3	0.2604 (6)	0.91914 (19)	0.53107 (16)	0.0446 (6)	
H3O	0.1363	0.9770	0.4983	0.06 (2)*	0.50
H3O'	0.4008	0.9730	0.5326	0.028 (17)*	0.50
O4	0.3842 (5)	0.94045 (17)	0.71321 (15)	0.0249 (4)	
H4O	0.242 (9)	1.017 (4)	0.727 (3)	0.057 (11)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0281 (12)	0.0176 (11)	0.0232 (11)	−0.0019 (9)	−0.0045 (9)	0.0032 (9)
C2A	0.0336 (16)	0.0229 (13)	0.0213 (13)	−0.0051 (11)	−0.0027 (12)	0.0049 (11)
C3A	0.0311 (15)	0.0231 (13)	0.0212 (13)	−0.0045 (11)	0.0005 (11)	−0.0011 (11)
N4A	0.0235 (12)	0.0179 (11)	0.0235 (11)	0.0001 (9)	−0.0019 (9)	−0.0020 (9)
C5A	0.0255 (15)	0.0193 (13)	0.0273 (14)	−0.0015 (11)	−0.0075 (11)	0.0051 (11)
C6A	0.0311 (15)	0.0282 (14)	0.0220 (13)	−0.0068 (12)	−0.0043 (11)	0.0062 (11)
C7A	0.0270 (15)	0.0322 (15)	0.0213 (13)	−0.0063 (12)	0.0000 (11)	−0.0019 (11)
C8A	0.0225 (14)	0.0210 (13)	0.0255 (13)	−0.0017 (11)	−0.0020 (11)	−0.0019 (11)
C9A	0.0210 (13)	0.0175 (12)	0.0194 (12)	−0.0027 (10)	−0.0051 (10)	0.0004 (10)
C10A	0.0210 (13)	0.0168 (12)	0.0213 (13)	−0.0033 (10)	−0.0049 (10)	−0.0006 (10)
C1	0.0241 (14)	0.0199 (13)	0.0191 (13)	−0.0033 (11)	−0.0021 (11)	0.0036 (10)
C2	0.0255 (14)	0.0206 (13)	0.0255 (13)	0.0029 (11)	−0.0052 (11)	0.0034 (11)
O1	0.0352 (12)	0.0237 (10)	0.0248 (10)	0.0040 (9)	0.0064 (8)	0.0087 (8)
O2	0.0498 (14)	0.0280 (11)	0.0293 (11)	0.0101 (10)	0.0160 (10)	0.0052 (9)
O3	0.0832 (18)	0.0238 (11)	0.0291 (11)	−0.0051 (12)	−0.0214 (11)	0.0104 (9)
O4	0.0264 (10)	0.0194 (9)	0.0272 (10)	0.0053 (8)	−0.0049 (8)	−0.0019 (8)

Geometric parameters (Å, °)

N1A—C2A	1.311 (3)	C8A—C9A	1.409 (4)
N1A—C9A	1.371 (3)	C8A—H8A	0.9300
C2A—C3A	1.416 (4)	C9A—C10A	1.413 (3)
C2A—H2A	0.9300	C1—O2	1.203 (3)
C3A—N4A	1.318 (3)	C1—O1	1.319 (3)
C3A—H3A	0.9300	C1—C2	1.526 (3)
N4A—C10A	1.368 (3)	C2—O4	1.399 (3)
C5A—C6A	1.367 (4)	C2—O3	1.407 (3)
C5A—C10A	1.409 (3)	C2—H2	0.9800
C5A—H5A	0.9300	O1—H1O	1.01 (4)
C6A—C7A	1.408 (4)	O3—H3O	0.8500

C6A—H6A	0.9300	O3—H3O'	0.8500
C7A—C8A	1.367 (4)	O4—H4O	0.93 (4)
C7A—H7A	0.9300		
C2A—N1A—C9A	117.4 (2)	N1A—C9A—C8A	120.2 (2)
N1A—C2A—C3A	122.3 (2)	N1A—C9A—C10A	120.3 (2)
N1A—C2A—H2A	118.8	C8A—C9A—C10A	119.5 (2)
C3A—C2A—H2A	118.8	N4A—C10A—C5A	119.7 (2)
N4A—C3A—C2A	121.8 (2)	N4A—C10A—C9A	121.0 (2)
N4A—C3A—H3A	119.1	C5A—C10A—C9A	119.2 (2)
C2A—C3A—H3A	119.1	O2—C1—O1	124.1 (2)
C3A—N4A—C10A	117.2 (2)	O2—C1—C2	123.8 (2)
C6A—C5A—C10A	120.1 (2)	O1—C1—C2	112.1 (2)
C6A—C5A—H5A	119.9	O4—C2—O3	111.8 (2)
C10A—C5A—H5A	119.9	O4—C2—C1	106.4 (2)
C5A—C6A—C7A	120.8 (2)	O3—C2—C1	109.5 (2)
C5A—C6A—H6A	119.6	O4—C2—H2	109.7
C7A—C6A—H6A	119.6	O3—C2—H2	109.7
C8A—C7A—C6A	120.2 (2)	C1—C2—H2	109.7
C8A—C7A—H7A	119.9	C1—O1—H1O	111 (2)
C6A—C7A—H7A	119.9	C2—O3—H3O	129.8
C7A—C8A—C9A	120.3 (2)	C2—O3—H3O'	104.2
C7A—C8A—H8A	119.9	H3O—O3—H3O'	88.0
C9A—C8A—H8A	119.9	C2—O4—H4O	105 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···N1A	1.01 (4)	1.71 (4)	2.722 (3)	179 (3)
O3—H3O···O3^{i}	0.85	1.87	2.678 (4)	157
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C2—H2···O4^{iv}	0.98	2.43	3.390 (3)	166
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Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+2, −*z*+1; (iii) *x*−1, *y*+1, *z*; (iv) *x*−1, *y*, *z*; (v) −*x*+2, −*y*+1, −*z*+1.

Fig. 1

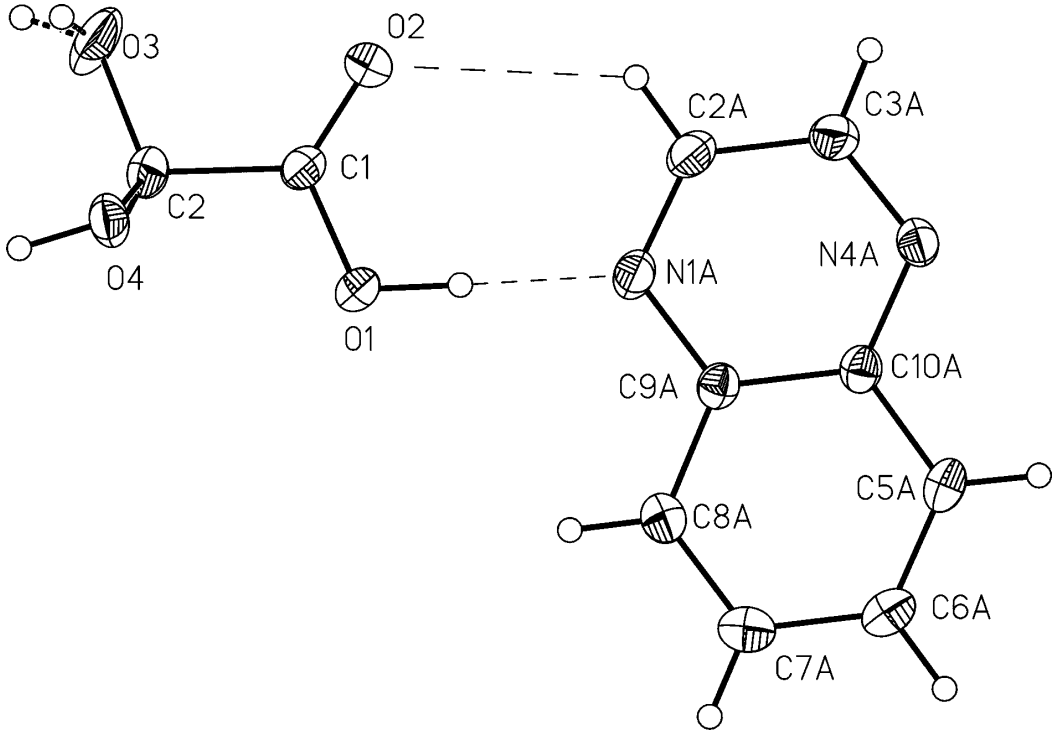

Fig. 2

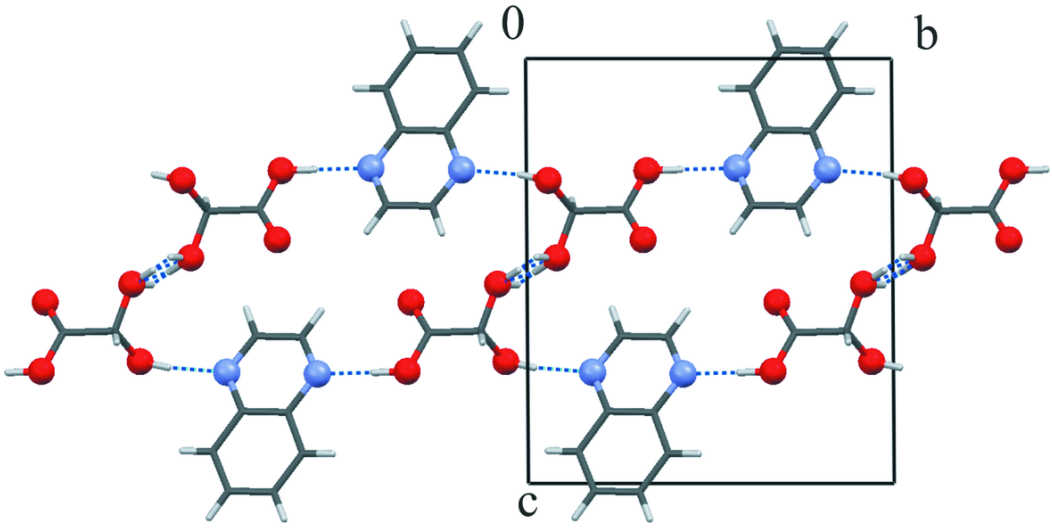

Fig. 3