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Propane-1,3-diaminium pyridine-2,3-dicarboxylate monohydrate

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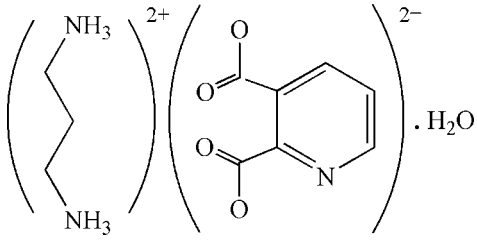
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.097; data-to-parameter ratio = 17.6.

The title compound, $C_3H_{12}N_2^{2+}\cdot C_7H_3NO_4^{2-}\cdot H_2O$, contains one dicationic fragment, one dianionic fragment and one water molecule. The two carboxylate groups of the pyridine-2,3-dicarboxylate ($pydc^{2-}$) fragment are almost perpendicular to each other [dihedral angle 83.10 (8)°]. In the crystal structure, intermolecular N—H···O, N—H···N, O—H···O and C—H···O hydrogen bonds and edge-to-face π–π stacking, together with ion pairing, are responsible for extending the structure in three dimensions, resulting in a supramolecular network.

Related literature

For general backgroud, see: Allen *et al.* (1987); Mendoza-Diaz *et al.* (2005); Chandrasekhar *et al.* (2001).



Experimental

Crystal data

$C_3H_{12}N_2^{2+}\cdot C_7H_3NO_4^{2-}\cdot H_2O$
M_r = 259.27
Triclinic, $P\overline{1}$
a = 7.4274 (5) Å
b = 8.6176 (5) Å
c = 10.7314 (7) Å
α = 109.989 (1)°
β = 102.225 (1)°
γ = 100.883 (1)°
V = 604.81 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 100 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.970, T_{max} = 0.978
6110 measured reflections
2897 independent reflections
2467 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.04
2897 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O4^{i}	0.91	2.24	2.918 (1)	131
N2—H2B···N1^{i}	0.91	2.14	2.967 (2)	150
N2—H2C···O1	0.91	1.92	2.823 (1)	175
N2—H2D···O4^{ii}	0.91	1.89	2.798 (2)	174
N3—H3B···O2^{iii}	0.91	1.86	2.752 (2)	168
N3—H3C···O5	0.91	1.97	2.836 (2)	158
N3—H3D···O3^{iv}	0.91	1.89	2.799 (1)	174
O5—H5A···O1^{v}	0.82	1.91	2.702 (1)	161
O5—H5B···O1^{ii}	0.82	2.52	3.094 (1)	128
O5—H5B···O3^{ii}	0.82	2.15	2.890 (1)	151
C10—H10B···O2^{iv}	0.99	2.38	3.102 (2)	129

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); 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: HK2241).

References

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supplementary materials

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Propane-1,3-diaminium pyridine-2,3-dicarboxylate monohydrate

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Comment

Pyridinedicarboxylic acids are applied as proton donors in ion pairs, as ligands in coordination compounds, and as hydrogen donor or acceptor in hydrogen bondings. However, their metal complexes have interesting properties in biological systems (Mendoza-Diaz *et al.*, 2005).

The molecule of the title compound, (I), contains one dicationic and one dianionic fragments and also one water molecule (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The two carboxylate groups of $(pydc)^{2-}$ fragment are perpendicular to each other.

As can be seen from the packing diagram (Fig. 2), the intramolecular N—H···O and intermolecular N—H···O, N—H···N, O—H···O and C—H···O hydrogen bonds (Table 1) and edge to face π-π stacking together with ion pairing are responsible for expanding the structure in three dimension resulting in a supramolecular network.

The bond distances and angles of C–H···π stacking are 2.81 Å (H···π) and 136° (C–H···π), which are within normal range (Chandrasekhar *et al.*, 2001). Another notable feature of the structure as shown in Fig. 2, is that the hydrogen bonds between water molecules, NH_3^+ tail of diamine and O atom of carboxylate group (*i.e.* two O5, two N3 and two O3 atoms and the related H atoms) form a 12-membered cyclic arrangement with a centre of symmetry in the middle of the ring.

Experimental

The title compound was synthesized by adding pyridine-2,3-dicarboxylic acid (10 mmol) to propane-1,3-diamine (10 mmol) in tetrahydrofuran (40 ml), and refluxing it. After a while, a white precipitate was obtained which was recrystallized to colorless crystals suitable for X-ray analysis.

Refinement

H atoms were positioned geometrically, with O—H = 0.82 Å (for OH_2), N—H = 0.91 Å (for NH_3) and C—H = 0.95 and 0.99 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,O,N)$.

Figures

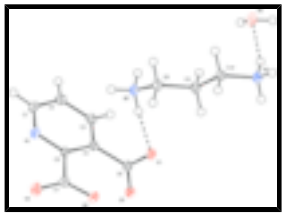

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

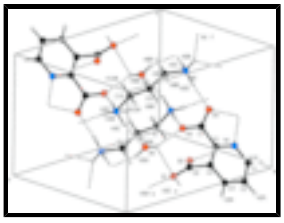

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

Propane-1,3-diaminium pyridine-2,3-dicarboxylate monohydrate

Crystal data

$C_3H_{12}N_2^{2+}\cdot C_7H_3NO_4^{2-}\cdot H_2O$	$Z = 2$
$M_r = 259.27$	$F_{000} = 276$
Triclinic, $P\overline{1}$	$D_x = 1.424$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.4274$ (5) Å	Cell parameters from 265 reflections
$b = 8.6176$ (5) Å	$\theta = 3$–28º
$c = 10.7314$ (7) Å	$\mu = 0.12$ mm^{-1}
$\alpha = 109.989$ (1)º	$T = 100$ (2) K
$\beta = 102.225$ (1)º	Prism, colourless
$\gamma = 100.883$ (1)º	0.24 × 0.22 × 0.18 mm
$V = 604.81$ (7) Å^3	

Data collection

Bruker APEXII CCD area-detector diffractometer	2897 independent reflections
Radiation source: fine-focus sealed tube	2467 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 100$(2) K	$\theta_{max} = 28.0$º
ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -9\rightarrow9$
$T_{min} = 0.970$, $T_{max} = 0.978$	$k = -11\rightarrow11$
6110 measured reflections	$l = -13\rightarrow14$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.1817P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.097$	$\Delta\rho_{max} = 0.38$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.24$ e Å^{-3}
2897 reflections	Extinction correction: none

165 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: constr

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.97996 (13)	0.66664 (11)	0.41529 (9)	0.01712 (19)
O2	1.21462 (12)	0.91436 (10)	0.50032 (8)	0.01534 (19)
O3	1.32210 (12)	0.61030 (11)	0.31424 (8)	0.01571 (19)
O4	1.39865 (14)	0.59160 (13)	0.11966 (10)	0.0227 (2)
N1	1.10608 (14)	0.71092 (12)	0.03480 (10)	0.0140 (2)
C1	0.97376 (17)	0.78571 (15)	−0.00249 (12)	0.0153 (2)
H1A	0.9425	0.7817	−0.0943	0.018*
C2	0.88011 (17)	0.86906 (15)	0.08756 (12)	0.0163 (2)
H2A	0.7908	0.9249	0.0588	0.020*
C3	0.91878 (17)	0.86966 (15)	0.21982 (12)	0.0150 (2)
H3A	0.8557	0.9249	0.2829	0.018*
C4	1.05220 (16)	0.78750 (14)	0.25870 (11)	0.0119 (2)
C5	1.14535 (16)	0.71214 (14)	0.16337 (11)	0.0116 (2)
C6	1.08930 (16)	0.78848 (14)	0.40348 (12)	0.0125 (2)
C7	1.30215 (17)	0.63029 (14)	0.20165 (12)	0.0135 (2)
N2	0.69146 (14)	0.43762 (12)	0.16715 (10)	0.0141 (2)
H2B	0.7342	0.4160	0.0912	0.017*
H2C	0.7898	0.5099	0.2445	0.017*
H2D	0.5948	0.4876	0.1572	0.017*
N3	0.37974 (15)	0.18365 (13)	0.44053 (10)	0.0153 (2)
H3B	0.3267	0.0846	0.4484	0.018*
H3C	0.2909	0.2425	0.4341	0.018*
H3D	0.4825	0.2498	0.5167	0.018*
C8	0.61920 (17)	0.27374 (15)	0.18108 (12)	0.0151 (2)
H8A	0.7223	0.2162	0.1869	0.018*
H8B	0.5102	0.1961	0.0980	0.018*
C9	0.55396 (17)	0.30690 (15)	0.31028 (12)	0.0152 (2)
H9A	0.4709	0.3846	0.3133	0.018*

H9B	0.6677	0.3655	0.3937	0.018*
C10	0.44394 (17)	0.14129 (15)	0.31345 (12)	0.0151 (2)
H10A	0.3310	0.0806	0.2295	0.018*
H10B	0.5275	0.0645	0.3141	0.018*
O5	0.09016 (13)	0.32465 (11)	0.34573 (9)	0.0181 (2)
H5A	0.0514	0.3400	0.4139	0.022*
H5B	0.1245	0.4204	0.3448	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0195 (4)	0.0172 (4)	0.0141 (4)	0.0004 (3)	0.0058 (3)	0.0077 (3)
O2	0.0180 (4)	0.0143 (4)	0.0116 (4)	0.0020 (3)	0.0033 (3)	0.0045 (3)
O3	0.0190 (4)	0.0167 (4)	0.0132 (4)	0.0066 (3)	0.0045 (3)	0.0074 (3)
O4	0.0254 (5)	0.0335 (5)	0.0210 (5)	0.0182 (4)	0.0135 (4)	0.0155 (4)
N1	0.0170 (5)	0.0128 (5)	0.0113 (5)	0.0027 (4)	0.0043 (4)	0.0047 (4)
C1	0.0183 (6)	0.0151 (5)	0.0113 (5)	0.0031 (4)	0.0019 (4)	0.0060 (4)
C2	0.0152 (6)	0.0165 (6)	0.0174 (6)	0.0054 (5)	0.0027 (5)	0.0079 (5)
C3	0.0159 (6)	0.0146 (5)	0.0146 (5)	0.0044 (4)	0.0059 (4)	0.0052 (4)
C4	0.0128 (5)	0.0100 (5)	0.0112 (5)	0.0003 (4)	0.0033 (4)	0.0040 (4)
C5	0.0129 (5)	0.0100 (5)	0.0107 (5)	0.0007 (4)	0.0033 (4)	0.0043 (4)
C6	0.0145 (5)	0.0136 (5)	0.0121 (5)	0.0066 (4)	0.0059 (4)	0.0056 (4)
C7	0.0144 (5)	0.0106 (5)	0.0136 (5)	0.0023 (4)	0.0035 (4)	0.0038 (4)
N2	0.0152 (5)	0.0150 (5)	0.0123 (5)	0.0031 (4)	0.0053 (4)	0.0056 (4)
N3	0.0165 (5)	0.0147 (5)	0.0150 (5)	0.0024 (4)	0.0038 (4)	0.0079 (4)
C8	0.0165 (6)	0.0131 (5)	0.0143 (5)	0.0027 (4)	0.0044 (4)	0.0048 (4)
C9	0.0170 (6)	0.0133 (5)	0.0148 (5)	0.0025 (4)	0.0053 (4)	0.0057 (4)
C10	0.0155 (5)	0.0149 (5)	0.0142 (5)	0.0033 (4)	0.0033 (4)	0.0062 (4)
O5	0.0245 (5)	0.0168 (4)	0.0158 (4)	0.0061 (4)	0.0098 (4)	0.0077 (3)

Geometric parameters (Å, °)

O1—C6	1.2612 (14)	N2—H2C	0.9100
O2—C6	1.2495 (14)	N2—H2D	0.9100
O3—C7	1.2601 (14)	N3—C10	1.4905 (15)
O4—C7	1.2473 (15)	N3—H3B	0.9100
N1—C1	1.3380 (15)	N3—H3C	0.9100
N1—C5	1.3444 (14)	N3—H3D	0.9100
C1—C2	1.3902 (17)	C8—C9	1.5181 (16)
C1—H1A	0.9500	C8—H8A	0.9900
C2—C3	1.3853 (16)	C8—H8B	0.9900
C2—H2A	0.9500	C9—C10	1.5199 (16)
C3—C4	1.3961 (16)	C9—H9A	0.9900
C3—H3A	0.9500	C9—H9B	0.9900
C4—C5	1.4008 (15)	C10—H10A	0.9900
C4—C6	1.5162 (15)	C10—H10B	0.9900
C5—C7	1.5258 (15)	O5—H5A	0.8200
N2—C8	1.4822 (15)	O5—H5B	0.8200
N2—H2B	0.9100		

C1—N1—C5	118.43 (10)	H2C—N2—H2D	109.5
N1—C1—C2	122.72 (11)	C10—N3—H3B	109.5
N1—C1—H1A	118.6	C10—N3—H3C	109.5
C2—C1—H1A	118.6	H3B—N3—H3C	109.5
C3—C2—C1	119.13 (11)	C10—N3—H3D	109.5
C3—C2—H2A	120.4	H3B—N3—H3D	109.5
C1—C2—H2A	120.4	H3C—N3—H3D	109.5
C2—C3—C4	118.75 (11)	N2—C8—C9	110.58 (9)
C2—C3—H3A	120.6	N2—C8—H8A	109.5
C4—C3—H3A	120.6	C9—C8—H8A	109.5
C3—C4—C5	118.43 (10)	N2—C8—H8B	109.5
C3—C4—C6	117.43 (10)	C9—C8—H8B	109.5
C5—C4—C6	124.13 (10)	H8A—C8—H8B	108.1
N1—C5—C4	122.47 (10)	C8—C9—C10	112.02 (9)
N1—C5—C7	116.06 (10)	C8—C9—H9A	109.2
C4—C5—C7	121.45 (10)	C10—C9—H9A	109.2
O2—C6—O1	126.29 (11)	C8—C9—H9B	109.2
O2—C6—C4	117.55 (10)	C10—C9—H9B	109.2
O1—C6—C4	115.97 (10)	H9A—C9—H9B	107.9
O4—C7—O3	126.58 (11)	N3—C10—C9	109.16 (9)
O4—C7—C5	117.09 (10)	N3—C10—H10A	109.8
O3—C7—C5	116.33 (10)	C9—C10—H10A	109.8
C8—N2—H2B	109.5	N3—C10—H10B	109.8
C8—N2—H2C	109.5	C9—C10—H10B	109.8
H2B—N2—H2C	109.5	H10A—C10—H10B	108.3
C8—N2—H2D	109.5	H5A—O5—H5B	105.4
H2B—N2—H2D	109.5		
C5—N1—C1—C2	2.11 (17)	C3—C4—C6—O2	−87.25 (13)
N1—C1—C2—C3	−2.61 (18)	C5—C4—C6—O2	91.67 (14)
C1—C2—C3—C4	0.55 (17)	C3—C4—C6—O1	87.95 (13)
C2—C3—C4—C5	1.79 (17)	C5—C4—C6—O1	−93.12 (13)
C2—C3—C4—C6	−179.22 (10)	N1—C5—C7—O4	9.82 (15)
C1—N1—C5—C4	0.43 (17)	C4—C5—C7—O4	−168.65 (11)
C1—N1—C5—C7	−178.02 (10)	N1—C5—C7—O3	−170.88 (10)
C3—C4—C5—N1	−2.37 (17)	C4—C5—C7—O3	10.65 (16)
C6—C4—C5—N1	178.71 (10)	N2—C8—C9—C10	168.92 (9)
C3—C4—C5—C7	176.00 (10)	C8—C9—C10—N3	−178.42 (9)
C6—C4—C5—C7	−2.92 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···O4^{i}	0.91	2.24	2.918 (1)	131
N2—H2B···N1^{i}	0.91	2.14	2.967 (2)	150
N2—H2C···O1	0.91	1.92	2.823 (1)	175
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N3—H3D···O3^{iv}	0.91	1.89	2.799 (1)	174
O5—H5A···O1^{v}	0.82	1.91	2.702 (1)	161
O5—H5B···O1^{ii}	0.82	2.52	3.094 (1)	128
O5—H5B···O3^{ii}	0.82	2.15	2.890 (1)	151
C10—H10B···O2^{iv}	0.99	2.38	3.102 (2)	129

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

Fig. 1

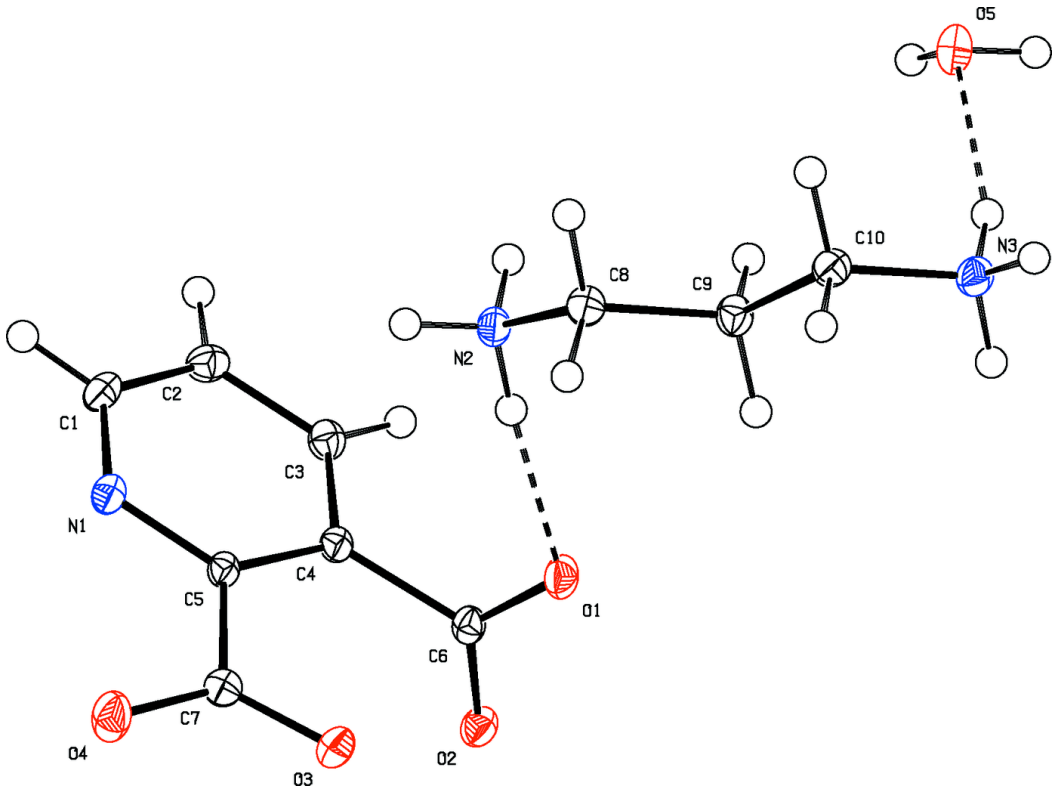
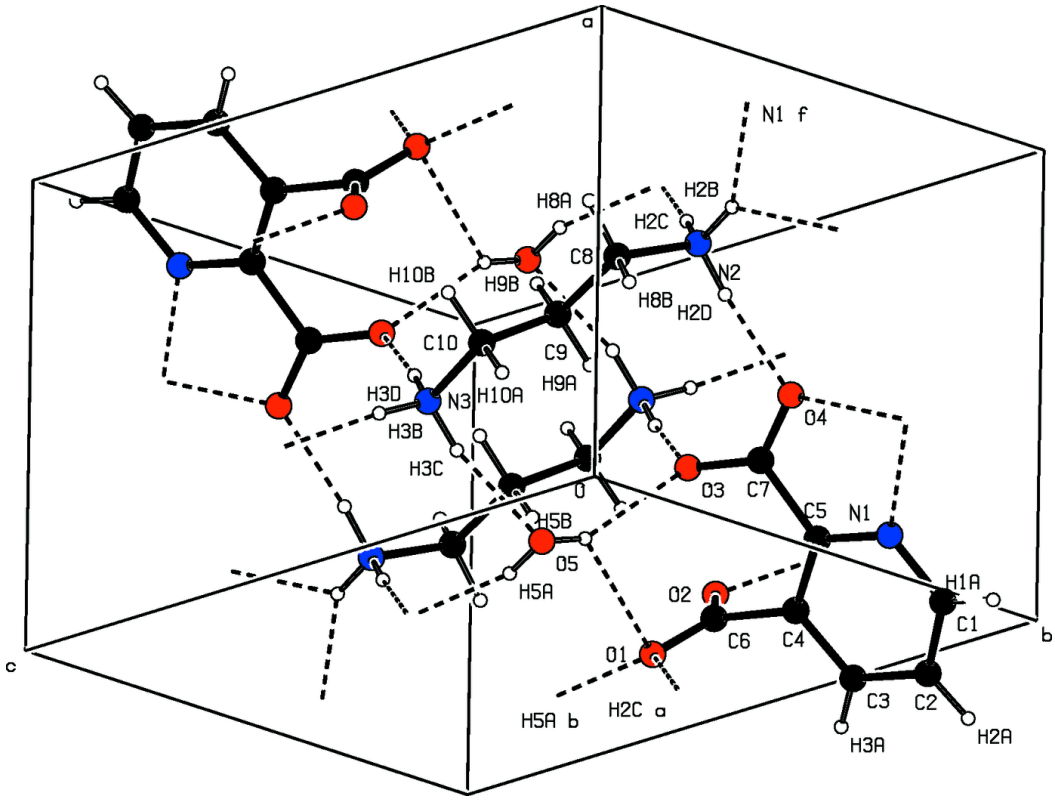


Fig. 2


a
N1 f
H8A
H2B
H2C
C8
N2
H10B
H9B
H8B
H2D
C10
C9
H9A
H3D
N3
H10A
H3B
O4
H3C
O3
C7
C5
N1
O
H5B
O5
H5A
H1A
O2
C1
C6
C4
O1
C3
C2
H5A b
H2C a
H3A
H2A
b
c