Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,4′-Iminodipyridinium 5-hydroxyisophthalate trihydrate

Maxwell A. Braverman and Robert L. LaDuca*

Lyman Briggs School of Science, Department of Chemistry, Michigan State University, East Lansing, MI 48825, USA
Correspondence e-mail: laduca@msu.edu

Received 31 May 2007; accepted 5 June 2007

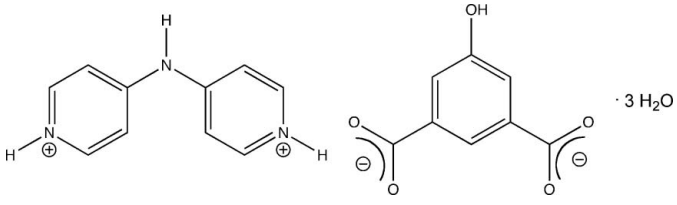
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.119; data-to-parameter ratio = 14.7.

In the title hydrated salt, $C_{10}H_{11}N_2^+\cdot C_8H_4O_5^-\cdot 3H_2O$, doubly protonated 4,4′-dipyridylamine molecules engage in N—H···O hydrogen bonding with 5-hydroxyisophthalate dianions to form undulating one-dimensional chains, which aggregate into three dimensions by O—H···O and N—H···O hydrogen-bonding patterns mediated by water molecules of crystallization.

Related literature

A salt containing monoprotonated 4,4′-dipyridylamine and singly deprotonated isophthalic acid has been reported previously (Amoore & Kepert, 2005). In contrast to the title compound, no water molecules of crystallization were observed.

For related literature, see: Bhogala & Nangia (2003); Horiuchi *et al.* (2005); Trask *et al.* (2005).



Experimental

Crystal data

$C_{10}H_{11}N_2^+\cdot C_8H_4O_5^-\cdot 3H_2O$
M_r = 407.38
Monoclinic, $P2_1/c$
a = 7.266 (3) Å
b = 32.701 (12) Å
c = 8.373 (3) Å
β = 109.971 (6)°
V = 1869.7 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
0.70 × 0.25 × 0.25 mm

Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.844, T_{max} = 0.970
18586 measured reflections
4299 independent reflections
3383 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.119
S = 1.05
4299 reflections
292 parameters
13 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WA···O3^{i}	0.878 (15)	1.917 (16)	2.7732 (19)	164.8 (19)
O1W—H1WB···O4	0.872 (15)	1.794 (15)	2.6661 (19)	178 (2)
O2W—H2WA···O1	0.861 (16)	1.883 (16)	2.7351 (18)	170 (2)
O2W—H2WB···O1^{ii}	0.871 (16)	1.985 (16)	2.846 (2)	170 (2)
O3W—H3WA···O2W	0.855 (16)	1.960 (17)	2.809 (2)	172 (3)
O3W—H3WB···O2^{iii}	0.841 (17)	1.984 (18)	2.813 (2)	169 (3)
O5—H5O···O3W^{ii}	0.882 (15)	1.834 (16)	2.700 (2)	167 (2)
N1—H1N···O3^{iv}	0.934 (14)	1.674 (15)	2.5876 (17)	164.9 (17)
N2—H2N···O1W^{v}	0.912 (14)	1.860 (14)	2.7712 (19)	176.5 (16)
N3—H3N···O2^{vi}	0.922 (14)	1.783 (15)	2.7034 (19)	175.6 (18)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalMaker* (CrystalMaker, 2005); software used to prepare material for publication: *SHELXL97*.

The authors gratefully acknowledge Michigan State University for funding this work and Ms Malexa Krost for experimental assistance. MAB acknowledges the MSU Quality Fund Undergraduate Research Program for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2416).

References

Amoore, J. J. M. & Kepert, C. J. (2005). *Acta Cryst.* E**61**, o3937–o3938.
Bhogala, B. R. & Nangia, A. (2003). *Cryst. Growth Des.* **3**, 547–554.
Bruker (2001). *SMART*. Version 5.624. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SAINT-Plus*. Version 6.25. Bruker AXS Inc., Madison, Wisconsin, USA.
Horiuchi, S., Ishii, F., Kumai, R., Okimoto, Y., Tachibana, H., Nagaora, N. & Tokura, Y. (2005). *Nat. Mater.* **4**, 163–166.
CrystalMaker (2005). *CrystalMaker*. Version 7.1. CrystalMaker Software, Bicester, Oxfordshire, England.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Trask, A. V., Motherwell, W. D. S. & Jones, W. (2005). *Cryst. Growth Des.* **5**, 1013–1021.

 doi:10.1107/S1600536807027651

supplementary materials

Acta Cryst. (2007). E**63**, o3167 [**doi:10.1107/S1600536807027651**]

4,4'-Iminodipyridinium 5-hydroxyisophthalate trihydrate

M. A. Braverman and R. L. LaDuca

Comment

Co-crystals of carboxylic acids and amines or imines have been the focus of recent studies in part because of potentially useful physical properties such as ferroelectricity (Horiuchi *et al.*, 2005). Also, the stability of pharmaceutically important formulations can be increased by co-crystal formation (Trask *et al.*, 2005). Proton transfer between carboxylic acid and pyridine components has been observed in several co-crystals, with the resulting charge-separated hydrogen bonding interactions exerting strong structure directing effects (Bhogala & Nangia, 2003).

The hydrated title salt was obtained during an attempt to synthesize a zinc-based coordination polymer containing 4,4'-dipyridylamine (dpa) and 5-hydroxyisophthalate (hip). The asymmetric unit consists of a doubly protonated $[H_2dpa]^{2+}$ dication, a doubly deprotonated $[hip]^{2-}$ anion, and three water molecules of crystallization (Fig. 1). The similar C—O bond lengths at the carboxylate termini reflect the presence of delocalized π bonds (Table 1).

Adjacent $[H_2dpa]^{2+}$ and $[hip]^{2-}$ moieties interact *via* N—H···O hydrogen bonding to construct undulating one-dimensional chains that course parallel to the *b* crystal direction. These in turn interact through O—H···O and N—H···O supramolecular interactions mediated by the amine functional group of $[H_2dpa]^{2+}$, the hydroxy group within $[hip]^{2-}$ and water molecules of crystallization to form two-dimensional layer motifs coincident with the [101] crystal planes (Fig. 2). The $[H_2dpa]^{2+}$ cations exhibit a slight inter-ring torsion of ~0.8° in order to maximize the supramolecular contacts. The $\{[H_2dpa][hip]\cdot 3H_2O\}$ layers subsequently stack along the (101) crystal direction through O—H···O hydrogen bonding patterns between water molecules of type O2W and O3W to create the three-dimensional structure of (I) (Fig. 3). Interlayer connectivity is enhanced by π-π stacking mechanisms and non-classical C—H···O interactions. Metrical parameters for the classical hydrogen bonding interactions are given in the table.

Experimental

Zinc nitrate hexahydrate and 5-hydroxyisophthalic acid were obtained commercially. 4,4'-dipyridylamine was prepared *via* a published procedure (GET, *et al.*, GET). A mixture of zinc nitrate hexahydrate (110 mg, 0.37 mmol), 5-hydroxyisophthalic acid (67 mg, 0.37 mmol), 4,4'-dipyridylamine (127 mg, 0.74 mmol) and 10.0 g water (550 mmol) was placed into a 23 ml Teflon-lined Parr Acid Digestion bomb, which was then heated under autogenous pressure at 393 K for 72 h. Large white rhombs of the title compound were isolated after cooling, along with an amorphous white solid.

Refinement

All H atoms bound to C atoms were placed in calculated positions, with C—H = 0.95 Å and refined in riding mode with U_{iso} = 1.2U_{eq}(C). All H atoms bound to O atoms were placed in calculated positions. The H atoms bound to N and O were found *via* Fourier difference map, restrained with N—H = 0.85 (2) Å or O—H = 0.89 (2) Å, and refined with U_{iso} = 1.2U_{eq}(O,*N*).

Figures

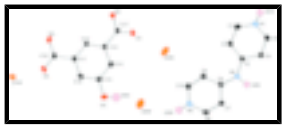


Fig. 1. Asymmetric unit of the title compound, showing 50% probability ellipsoids and atom numbering scheme. Most hydrogen atoms have been omitted for clarity.

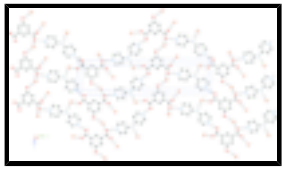


Fig. 2. A layer in the title compund. Color codes: light-blue N, red O within organic moieties, orange O within water molecules, black C, pink H. Hydrogen bonding is shown as dashed lines.

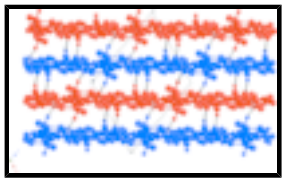


Fig. 3. Packing diagram illustrating the stacking of layers to form the 3-D crystal structure of the title compound. Hydrogen bonding is shown as dashed lines.

4,4'-Iminodipyridinium 5-hydroxyisophthalate trihydrate

Crystal data

$C_{10}H_{11}N_2^+ \cdot C_8H_4O_5^- \cdot 3H_2O$	$F_{000} = 856$
$M_r = 407.38$	$D_x = 1.447$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 18586 reflections
$a = 7.266$ (3) Å	$\theta = 2.5$–28.2º
$b = 32.701$ (12) Å	$\mu = 0.12$ mm^{-1}
$c = 8.373$ (3) Å	$T = 293$ (2) K
$\beta = 109.971$ (6)º	Rhomb, colourless
$V = 1869.7$ (12) Å^3	0.70 × 0.25 × 0.25 mm
$Z = 4$	

Data collection

Bruker SMART 1K diffractometer	4299 independent reflections
Radiation source: fine-focus sealed tube	3383 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.031$
$T = 293$(2) K	$\theta_{max} = 28.2$º
ω scans	$\theta_{min} = 2.5$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.844$, $T_{max} = 0.970$	$k = -43 \rightarrow 42$
18586 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.119$	$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.5827P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
4299 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
292 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
13 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
O1	0.17693 (18)	0.21437 (3)	0.25784 (15)	0.0438 (3)
O1W	0.1022 (2)	−0.07026 (4)	0.21597 (15)	0.0478 (3)
H1WA	0.141 (3)	−0.0644 (6)	0.130 (2)	0.057*
H1WB	0.056 (3)	−0.0470 (5)	0.238 (3)	0.057*
O2	−0.08396 (17)	0.18913 (3)	0.05970 (14)	0.0421 (3)
O2W	0.3655 (2)	0.26570 (5)	0.52078 (19)	0.0629 (4)
H2WA	0.303 (3)	0.2478 (7)	0.447 (3)	0.075*
H2WB	0.299 (3)	0.2692 (7)	0.588 (3)	0.075*
O3	−0.19225 (18)	0.03830 (3)	0.05520 (14)	0.0434 (3)
O3W	0.5209 (2)	0.33012 (5)	0.3846 (2)	0.0749 (5)
H3WA	0.463 (4)	0.3108 (7)	0.418 (3)	0.090*
H3WB	0.643 (2)	0.3273 (8)	0.431 (3)	0.090*
O4	−0.0280 (2)	0.00170 (3)	0.28294 (16)	0.0501 (3)
O5	0.3678 (2)	0.10164 (4)	0.70746 (15)	0.0517 (4)
H5O	0.410 (3)	0.1259 (5)	0.750 (3)	0.062*
N1	0.3734 (2)	0.47531 (4)	0.61321 (16)	0.0352 (3)

H1N	0.301 (2)	0.4950 (5)	0.537 (2)	0.042*
N2	0.69030 (18)	0.39251 (4)	0.97808 (15)	0.0296 (3)
H2N	0.754 (2)	0.4053 (5)	1.0786 (18)	0.035*
N3	0.8395 (2)	0.27018 (4)	1.02502 (17)	0.0379 (3)
H3N	0.872 (3)	0.2428 (5)	1.039 (2)	0.045*
C1	0.3611 (2)	0.43590 (5)	0.5722 (2)	0.0403 (4)
H1	0.2815	0.4280	0.4639	0.048*
C2	0.4631 (2)	0.40653 (5)	0.6854 (2)	0.0376 (4)
H2	0.4528	0.3791	0.6535	0.045*
C3	0.5821 (2)	0.41810 (4)	0.84837 (18)	0.0274 (3)
C4	0.5959 (2)	0.45988 (5)	0.88553 (19)	0.0368 (4)
H4	0.6770	0.4690	0.9914	0.044*
C5	0.4905 (3)	0.48740 (5)	0.7667 (2)	0.0398 (4)
H5	0.5005	0.5151	0.7932	0.048*
C6	0.9166 (3)	0.29658 (5)	1.1534 (2)	0.0434 (4)
H6	1.0078	0.2873	1.2546	0.052*
C7	0.8632 (3)	0.33660 (5)	1.1376 (2)	0.0387 (4)
H7	0.9159	0.3543	1.2287	0.046*
C8	0.7285 (2)	0.35143 (4)	0.98427 (18)	0.0272 (3)
C9	0.6495 (3)	0.32274 (5)	0.8556 (2)	0.0418 (4)
H9	0.5574	0.3308	0.7528	0.050*
C10	0.7077 (3)	0.28279 (5)	0.8809 (2)	0.0429 (4)
H10	0.6533	0.2639	0.7944	0.052*
C11	0.0241 (2)	0.07331 (4)	0.29357 (18)	0.0270 (3)
C12	−0.0087 (2)	0.11074 (4)	0.20894 (17)	0.0268 (3)
H12	−0.0965	0.1125	0.0983	0.032*
C13	0.0899 (2)	0.14548 (4)	0.28976 (17)	0.0260 (3)
C14	0.2193 (2)	0.14313 (5)	0.45664 (18)	0.0298 (3)
H14	0.2876	0.1662	0.5098	0.036*
C15	0.2460 (2)	0.10607 (5)	0.54350 (18)	0.0324 (3)
C16	0.1486 (2)	0.07122 (4)	0.46149 (18)	0.0312 (3)
H16	0.1670	0.0464	0.5193	0.037*
C17	0.0591 (2)	0.18573 (4)	0.19650 (18)	0.0295 (3)
C18	−0.0714 (2)	0.03461 (4)	0.20551 (19)	0.0316 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0534 (7)	0.0261 (6)	0.0406 (6)	−0.0103 (5)	0.0013 (5)	0.0009 (5)
O1W	0.0707 (9)	0.0344 (6)	0.0330 (6)	0.0092 (6)	0.0108 (6)	0.0052 (5)
O2	0.0473 (7)	0.0253 (6)	0.0385 (6)	0.0005 (5)	−0.0051 (5)	0.0047 (5)
O2W	0.0617 (9)	0.0714 (10)	0.0543 (9)	−0.0202 (8)	0.0182 (7)	−0.0257 (7)
O3	0.0573 (7)	0.0304 (6)	0.0346 (6)	−0.0140 (5)	0.0055 (5)	−0.0067 (4)
O3W	0.0541 (9)	0.0675 (11)	0.0819 (12)	0.0072 (8)	−0.0040 (8)	0.0278 (9)
O4	0.0779 (9)	0.0212 (6)	0.0485 (7)	−0.0009 (6)	0.0183 (6)	0.0022 (5)
O5	0.0616 (8)	0.0461 (8)	0.0292 (6)	−0.0090 (6)	−0.0081 (5)	0.0076 (5)
N1	0.0388 (7)	0.0313 (7)	0.0320 (7)	0.0102 (6)	0.0075 (5)	0.0083 (5)
N2	0.0344 (7)	0.0240 (6)	0.0253 (6)	0.0024 (5)	0.0038 (5)	0.0012 (5)

N3	0.0455 (8)	0.0231 (6)	0.0403 (7)	0.0057 (6)	0.0086 (6)	0.0056 (5)
C1	0.0429 (9)	0.0384 (9)	0.0299 (8)	0.0056 (7)	−0.0003 (7)	0.0015 (6)
C2	0.0444 (9)	0.0250 (8)	0.0339 (8)	0.0045 (6)	0.0012 (7)	−0.0003 (6)
C3	0.0278 (7)	0.0260 (7)	0.0282 (7)	0.0037 (5)	0.0090 (6)	0.0036 (5)
C4	0.0458 (9)	0.0286 (8)	0.0281 (7)	0.0037 (7)	0.0024 (6)	−0.0005 (6)
C5	0.0495 (10)	0.0264 (8)	0.0377 (9)	0.0071 (7)	0.0075 (7)	0.0015 (6)
C6	0.0527 (10)	0.0334 (9)	0.0333 (8)	0.0094 (7)	0.0007 (7)	0.0079 (6)
C7	0.0495 (10)	0.0298 (8)	0.0282 (8)	0.0035 (7)	0.0020 (7)	0.0009 (6)
C8	0.0274 (7)	0.0248 (7)	0.0290 (7)	0.0018 (5)	0.0092 (6)	0.0040 (5)
C9	0.0475 (10)	0.0300 (8)	0.0334 (8)	0.0060 (7)	−0.0049 (7)	0.0007 (6)
C10	0.0495 (10)	0.0282 (8)	0.0400 (9)	0.0024 (7)	0.0009 (7)	−0.0035 (7)
C11	0.0299 (7)	0.0219 (7)	0.0288 (7)	0.0007 (5)	0.0098 (6)	−0.0007 (5)
C12	0.0285 (7)	0.0235 (7)	0.0243 (7)	0.0012 (5)	0.0037 (5)	−0.0009 (5)
C13	0.0278 (7)	0.0223 (7)	0.0272 (7)	0.0018 (5)	0.0084 (6)	−0.0008 (5)
C14	0.0303 (7)	0.0269 (7)	0.0291 (7)	−0.0034 (6)	0.0062 (6)	−0.0046 (5)
C15	0.0324 (8)	0.0349 (8)	0.0249 (7)	−0.0001 (6)	0.0034 (6)	0.0025 (6)
C16	0.0357 (8)	0.0246 (7)	0.0312 (8)	0.0027 (6)	0.0086 (6)	0.0059 (6)
C17	0.0357 (8)	0.0211 (7)	0.0286 (7)	0.0009 (6)	0.0070 (6)	−0.0016 (5)
C18	0.0398 (8)	0.0243 (7)	0.0329 (8)	−0.0027 (6)	0.0150 (6)	−0.0032 (6)

Geometric parameters (Å, °)

O1—C17	1.2546 (18)	C2—H2	0.9300
O1W—H1WA	0.878 (15)	C3—C4	1.397 (2)
O1W—H1WB	0.872 (15)	C4—C5	1.366 (2)
O2—C17	1.2614 (18)	C4—H4	0.9300
O2W—H2WA	0.861 (16)	C5—H5	0.9300
O2W—H2WB	0.871 (16)	C6—C7	1.358 (2)
O3—C18	1.2706 (19)	C6—H6	0.9300
O3W—H3WA	0.855 (16)	C7—C8	1.408 (2)
O3W—H3WB	0.841 (17)	C7—H7	0.9300
O4—C18	1.2402 (19)	C8—C9	1.396 (2)
O5—C15	1.3632 (18)	C9—C10	1.367 (2)
O5—H5O	0.882 (15)	C9—H9	0.9300
N1—C1	1.329 (2)	C10—H10	0.9300
N1—C5	1.336 (2)	C11—C16	1.390 (2)
N1—H1N	0.934 (14)	C11—C12	1.393 (2)
N2—C8	1.3691 (19)	C11—C18	1.509 (2)
N2—C3	1.3836 (18)	C12—C13	1.3895 (19)
N2—H2N	0.912 (14)	C12—H12	0.9300
N3—C10	1.325 (2)	C13—C14	1.395 (2)
N3—C6	1.343 (2)	C13—C17	1.507 (2)
N3—H3N	0.922 (14)	C14—C15	1.392 (2)
C1—C2	1.375 (2)	C14—H14	0.9300
C1—H1	0.9300	C15—C16	1.393 (2)
C2—C3	1.396 (2)	C16—H16	0.9300
H1WA—O1W—H1WB	103.5 (18)	N2—C8—C9	127.28 (13)
H2WA—O2W—H2WB	107 (2)	N2—C8—C7	116.18 (13)
H3WA—O3W—H3WB	109 (2)	C9—C8—C7	116.53 (14)

C15—O5—H5O	109.3 (14)	C10—C9—C8	119.94 (14)
C1—N1—C5	120.00 (14)	C10—C9—H9	120.0
C1—N1—H1N	121.3 (11)	C8—C9—H9	120.0
C5—N1—H1N	118.7 (11)	N3—C10—C9	121.84 (15)
C8—N2—C3	132.57 (13)	N3—C10—H10	119.1
C8—N2—H2N	112.5 (11)	C9—C10—H10	119.1
C3—N2—H2N	114.8 (11)	C16—C11—C12	119.78 (13)
C10—N3—C6	120.21 (14)	C16—C11—C18	119.01 (13)
C10—N3—H3N	119.6 (12)	C12—C11—C18	121.20 (13)
C6—N3—H3N	120.1 (12)	C13—C12—C11	120.04 (13)
N1—C1—C2	121.76 (15)	C13—C12—H12	120.0
N1—C1—H1	119.1	C11—C12—H12	120.0
C2—C1—H1	119.1	C12—C13—C14	120.11 (13)
C1—C2—C3	119.54 (15)	C12—C13—C17	120.06 (13)
C1—C2—H2	120.2	C14—C13—C17	119.82 (13)
C3—C2—H2	120.2	C15—C14—C13	119.83 (13)
N2—C3—C2	126.87 (14)	C15—C14—H14	120.1
N2—C3—C4	115.99 (13)	C13—C14—H14	120.1
C2—C3—C4	117.14 (13)	O5—C15—C14	123.04 (14)
C5—C4—C3	120.15 (14)	O5—C15—C16	117.08 (14)
C5—C4—H4	119.9	C14—C15—C16	119.87 (13)
C3—C4—H4	119.9	C11—C16—C15	120.28 (13)
N1—C5—C4	121.35 (15)	C11—C16—H16	119.9
N1—C5—H5	119.3	C15—C16—H16	119.9
C4—C5—H5	119.3	O1—C17—O2	122.78 (13)
N3—C6—C7	120.96 (15)	O1—C17—C13	118.67 (13)
N3—C6—H6	119.5	O2—C17—C13	118.55 (13)
C7—C6—H6	119.5	O4—C18—O3	124.50 (14)
C6—C7—C8	120.46 (15)	O4—C18—C11	118.68 (14)
C6—C7—H7	119.8	O3—C18—C11	116.82 (13)
C8—C7—H7	119.8		
C5—N1—C1—C2	1.5 (3)	C16—C11—C12—C13	2.9 (2)
N1—C1—C2—C3	0.4 (3)	C18—C11—C12—C13	−176.23 (13)
C8—N2—C3—C2	6.9 (3)	C11—C12—C13—C14	−1.1 (2)
C8—N2—C3—C4	−172.55 (15)	C11—C12—C13—C17	178.20 (13)
C1—C2—C3—N2	178.42 (15)	C12—C13—C14—C15	−1.5 (2)
C1—C2—C3—C4	−2.2 (2)	C17—C13—C14—C15	179.22 (13)
N2—C3—C4—C5	−178.40 (15)	C13—C14—C15—O5	−179.00 (14)
C2—C3—C4—C5	2.1 (2)	C13—C14—C15—C16	2.2 (2)
C1—N1—C5—C4	−1.6 (3)	C12—C11—C16—C15	−2.3 (2)
C3—C4—C5—N1	−0.3 (3)	C18—C11—C16—C15	176.95 (14)
C10—N3—C6—C7	−0.8 (3)	O5—C15—C16—C11	−179.19 (14)
N3—C6—C7—C8	−1.4 (3)	C14—C15—C16—C11	−0.3 (2)
C3—N2—C8—C9	−5.9 (3)	C12—C13—C17—O1	−165.67 (14)
C3—N2—C8—C7	173.86 (15)	C14—C13—C17—O1	13.6 (2)
C6—C7—C8—N2	−177.25 (16)	C12—C13—C17—O2	14.1 (2)
C6—C7—C8—C9	2.5 (2)	C14—C13—C17—O2	−166.62 (14)
N2—C8—C9—C10	178.13 (16)	C16—C11—C18—O4	−3.7 (2)
C7—C8—C9—C10	−1.6 (2)	C12—C11—C18—O4	175.53 (14)

C6—N3—C10—C9	1.7 (3)	C16—C11—C18—O3	176.31 (14)
C8—C9—C10—N3	−0.5 (3)	C12—C11—C18—O3	−4.5 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1WA···O3^{i}	0.878 (15)	1.917 (16)	2.7732 (19)	164.8 (19)
O1W—H1WB···O4	0.872 (15)	1.794 (15)	2.6661 (19)	178 (2)
O2W—H2WA···O1	0.861 (16)	1.883 (16)	2.7351 (18)	170 (2)
O2W—H2WB···O1^{ii}	0.871 (16)	1.985 (16)	2.846 (2)	170 (2)
O3W—H3WA···O2W	0.855 (16)	1.960 (17)	2.809 (2)	172 (3)
O3W—H3WB···O2^{iii}	0.841 (17)	1.984 (18)	2.813 (2)	169 (3)
O5—H5O···O3W^{ii}	0.882 (15)	1.834 (16)	2.700 (2)	167 (2)
N1—H1N···O3^{iv}	0.934 (14)	1.674 (15)	2.5876 (17)	164.9 (17)
N2—H2N···O1W^{v}	0.912 (14)	1.860 (14)	2.7712 (19)	176.5 (16)
N3—H3N···O2^{vi}	0.922 (14)	1.783 (15)	2.7034 (19)	175.6 (18)

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

Fig. 1

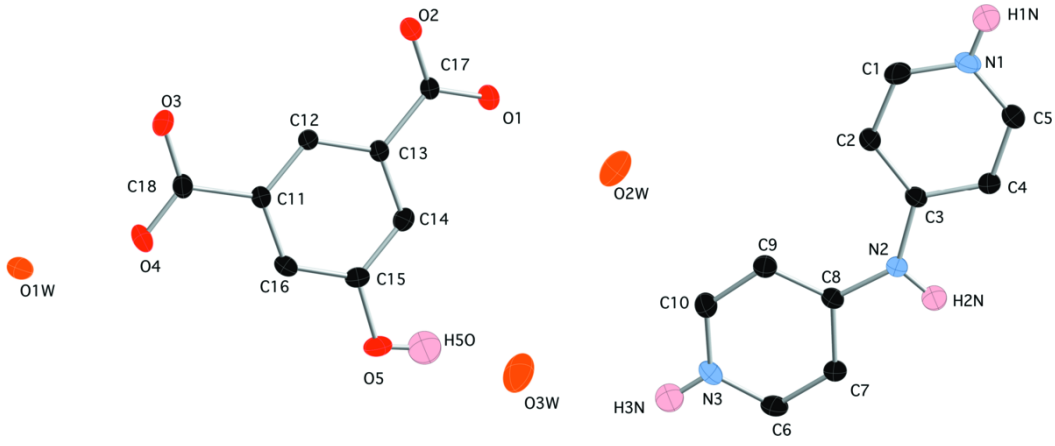

O2
C17
O1
O3
C12
C13
C18
C11
C14
O4
C16
C15
O1W
H5O
O5
O3W
O2W
H1N
N1
C1
C5
C2
C4
C3
C9
N2
C8
C10
H2N
C7
H3N
N3
C6

Fig. 2

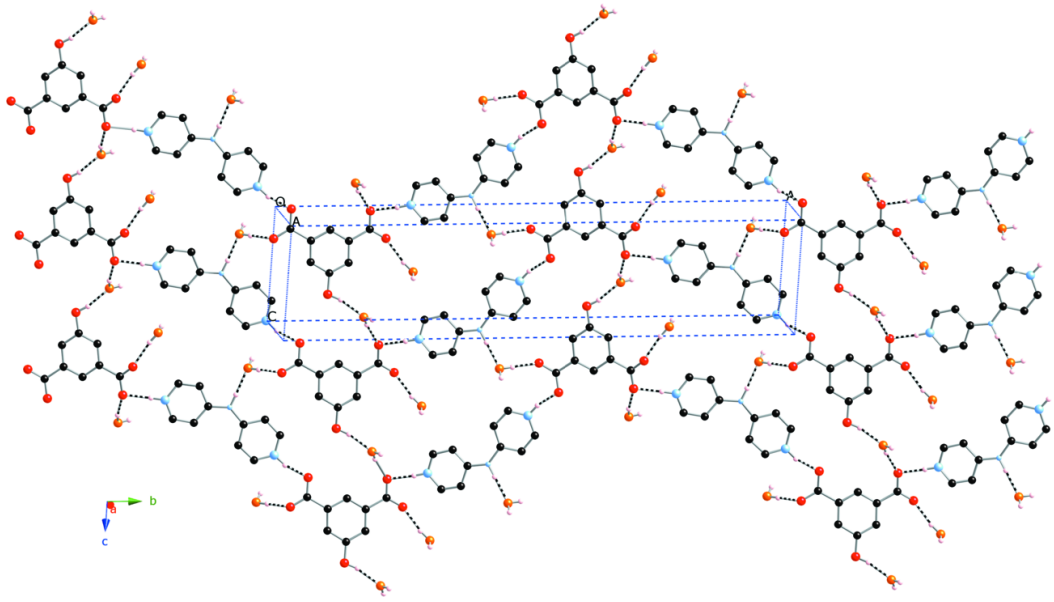


Fig. 3