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5-Amino-2,4,6-triiodoisophthalic acid monohydrate

Tobias Beck* and George M. Sheldrick

Department of Structural Chemistry, Georg-August Universität, Tammannstrasse 4, 37077 Göttingen, Germany
Correspondence e-mail: tbeck@shelx.uni-ac.gwdg.de

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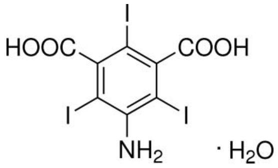
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.026; wR factor = 0.062; data-to-parameter ratio = 15.7.

The title compound, $C_8H_4I_3NO_4\cdot H_2O$, shows an extensive hydrogen-bond network; in the crystal structure, molecules are linked by O—H···O, N—H···O and O—H···N hydrogen bonds involving all possible donors and also the water molecule.

Related literature

For the synthetic procedure, see Larsen *et al.* (1956). For related crystal structure determinations: 1,3,5-triiodobenzene, see: Margraf & Bats (2006); sodium diatrizoate, see: Tonnessen *et al.* (1996). For the 1,3,5-triiodobenzene core as the basis of contrast agents, see: Yu & Watson (1999).



Experimental

Crystal data

$C_8H_4I_3NO_4\cdot H_2O$
M_r = 576.84
Orthorhombic, *Pbca*
a = 9.214 (1) Å
b = 15.735 (2) Å
c = 18.816 (2) Å
V = 2728.0 (5) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 54.11 mm^{-1}
T = 100 (2) K
0.08 × 0.05 × 0.03 mm

Data collection

Bruker SMART 6000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.106, $T_{\max}$ = 0.345 (expected range = 0.061–0.197)
49139 measured reflections
2716 independent reflections
2545 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.061
S = 1.03
2716 reflections
173 parameters
14 restraints
Only H-atom coordinates refined
$\Delta\rho_{\max}$ = 0.71 e Å^{-3}
$\Delta\rho_{\min}$ = −1.71 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H11···O14	0.79 (5)	1.75 (5)	2.540 (5)	173 (7)
O8—H8···O12$^{\rm i}$	0.80 (5)	1.90 (5)	2.662 (5)	161 (7)
O14—H14*A*···O9$^{\rm ii}$	0.81 (4)	1.95 (4)	2.751 (5)	170 (6)
O14—H14*B*···N13$^{\rm iii}$	0.81 (4)	2.05 (4)	2.841 (5)	166 (6)
N13—H13*A*···O14$^{\rm iv}$	0.88 (4)	2.30 (5)	3.067 (6)	147 (5)
N13—H13*B*···O12$^{\rm iv}$	0.88 (4)	2.68 (5)	3.478 (5)	152 (5)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z$; (ii) $-x+\frac{5}{2}, y-\frac{1}{2}, z$; (iii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, z$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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: *SHELXL97* and *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2102).

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supplementary materials

Acta Cryst. (2008). E**64**, o1286 [**doi:10.1107/S1600536808017741**]

5-Amino-2,4,6-triiodoisophthalic acid monohydrate

T. Beck and **G. M. Sheldrick**

Comment

Iodine-based compounds have always been in the focus of contrast agents for X-ray imaging. The 1,3,5-triiodobenzene core has been the basis of many contrast agents (Yu & Watson 1999). The ionic monomer diatrizoate was one of the first compounds used (Tonnessen *et al.* 1996).

The title compound, 5-Amino-2,4,6-triiodoisophthalic acid (hereafter I3C), crystallizes as a monohydrate, due to water impurities in the crystallization solution. It forms hydrogen bonds with all potential donors as well as the lattice water being involved (Fig. 2, Table 1). However, the interaction between N13 and O12 is slightly weaker. In the crystal, the molecules are positioned perpendicular to each other, showing no π-π interactions of the phenyl rings (Fig. 3).

The three functional groups for hydrogen bonding, along with the three iodine atoms, render I3C a suitable agent for experimental phasing of macromolecules (Beck *et al.*, unpublished results). The iodine atoms give rise to a large anomalous signal, even at in-house sources. Additionally, they form an equilateral triangle (I—I 6.0 Å) which is easy to recognize in the heavy atom substructure when this compound is used as a heavy atom derivative for macromolecular phasing.

Experimental

The title compound was prepared according to the reported procedure (Larsen *et al.* 1956). It was recrystallized from a methanol-acetonitrile solution by slowly evaporating the solvents to obtain crystals suitable for X-ray single-crystal diffraction.

Refinement

Hydrogen atoms were located *via* the difference Fourier map and their geometrical positions were refined with restraints. The *U* values were set to 1.5 U_{eq} of their parent atom. Bond lengths for hydrogen atoms were restrained to be equal (SADI in SHELXL-97). Phenyl ring and carboxylate groups were restrained to planarity.

Figures

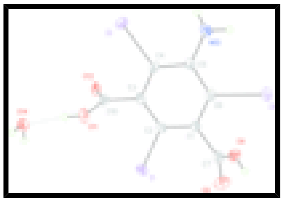

Fig. 1. A view of I3C. Displacement ellipsoids are drawn at the 50% probability level. The hydrogen bond within the asymmetric unit is shown as a dashed line.

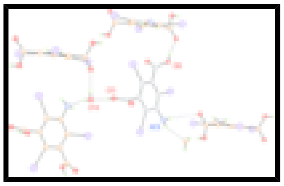
Fig. 2. Hydrogen bonding of I3C. Symmetry equivalents are depicted in orange.

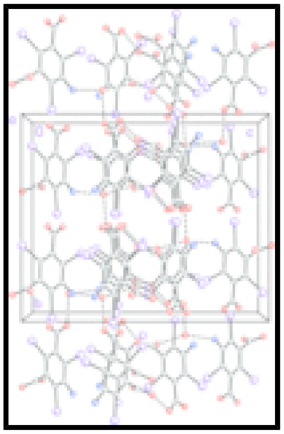
Fig. 3. Packing of I3C, viewed along b. Hydrogen atoms are omitted for clarity. In alternating layers molecules are positioned perpendicular to each other. Hydrogen bonds are shown as dashed lines.

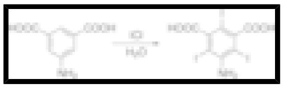
Fig. 4. Synthetic scheme of I3C.

5-Amino-2,4,6-triiodoisophthalic acid monohydrate

Crystal data

$C_8H_4I_3NO_4{\cdot}H_2O$

$M_r = 576.84$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 9.214$ (1) Å

$b = 15.735$ (2) Å

$c = 18.816$ (2) Å

$V = 2728.0$ (5) $Å^3$

$Z = 8$

$F_{000} = 2080$

$D_x = 2.809$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å

Cell parameters from 9945 reflections

$\theta = 4.7–60.8°$

$\mu = 54.11$ mm^{-1}

$T = 100$ (2) K

Block, yellow

0.08 × 0.05 × 0.03 mm

Data collection

Bruker SMART 6000 diffractometer

Radiation source: rotating anode

Monochromator: INCOATEC multilayer optics

Detector resolution: 5.602 pixels mm^{-1}

$T = 100(2)$ K

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

2716 independent reflections

2545 reflections with $I > 2\sigma(I)$

$R_{int} = 0.043$

$\theta_{max} = 74.3°$

$\theta_{min} = 4.7°$

$h = -10 \rightarrow 10$

$k = -17 \rightarrow 19$

$T_{\min} = 0.106$, $T_{\max} = 0.345$	$l = -23 \rightarrow 23$
49139 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.026$	Only H-atom coordinates refined
$wR(F^2) = 0.061$	$w = 1/[\sigma^2(F_o^2) + (0.0277P)^2 + 17.3624P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\max} = 0.002$
2716 reflections	$\Delta\rho_{\max} = 0.72$ e Å^{-3}
173 parameters	$\Delta\rho_{\min} = -1.71$ e Å^{-3}
14 restraints	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.000058 (8)

Special details

Experimental. Intensities were measured with a Bruker *SMART* 6000 area-detector

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 $F^2 > 2\sigma(F^2)$ 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. Hydrogen atoms were located *via* the difference Fourier map and their geometrical positions were refined with restraints. The U values were set to 1.5 U_{eq} of their parent atom.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
I1	1.18424 (3)	0.630616 (18)	0.012748 (16)	0.02419 (10)
I2	0.71499 (3)	0.674286 (18)	0.234093 (15)	0.02375 (10)
I3	1.04238 (3)	0.979824 (17)	0.124476 (17)	0.02693 (10)
C1	1.0827 (5)	0.7970 (3)	0.0781 (2)	0.0161 (8)
C2	1.0606 (5)	0.7094 (3)	0.0789 (2)	0.0161 (9)
C3	0.9544 (5)	0.6745 (3)	0.1231 (2)	0.0153 (9)
C4	0.8655 (5)	0.7276 (3)	0.1631 (2)	0.0163 (9)
C5	0.8803 (5)	0.8172 (3)	0.1604 (2)	0.0165 (9)
C6	0.9945 (5)	0.8492 (3)	0.1199 (2)	0.0180 (9)
C7	1.1981 (5)	0.8362 (3)	0.0320 (3)	0.0202 (10)
O8	1.1458 (4)	0.8648 (2)	−0.02803 (18)	0.0256 (7)
H8	1.205 (6)	0.883 (4)	−0.055 (3)	0.038*

O9	1.3242 (4)	0.8414 (2)	0.05017 (19)	0.0290 (8)
C10	0.9364 (5)	0.5794 (3)	0.1285 (2)	0.0173 (9)
O11	1.0376 (4)	0.5445 (2)	0.16663 (17)	0.0224 (7)
H11	1.031 (7)	0.494 (3)	0.170 (3)	0.034*
O12	0.8358 (4)	0.54161 (19)	0.10032 (17)	0.0213 (7)
N13	0.7889 (5)	0.8702 (2)	0.1997 (2)	0.0210 (8)
H13A	0.698 (5)	0.855 (4)	0.204 (3)	0.031*
H13B	0.785 (6)	0.922 (3)	0.183 (3)	0.031*
O14	1.0395 (4)	0.3834 (2)	0.17540 (18)	0.0235 (7)
H14A	1.074 (7)	0.366 (4)	0.139 (3)	0.035*
H14B	1.099 (6)	0.376 (4)	0.206 (3)	0.035*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02850 (18)	0.02100 (16)	0.02307 (16)	0.00785 (12)	0.00724 (12)	0.00027 (11)
I2	0.02603 (18)	0.02156 (16)	0.02364 (16)	−0.00544 (11)	0.00910 (12)	−0.00016 (11)
I3	0.02852 (19)	0.01260 (15)	0.03966 (19)	−0.00146 (11)	0.00662 (13)	0.00096 (11)
C1	0.018 (2)	0.0143 (19)	0.0162 (19)	0.0012 (17)	0.0007 (17)	0.0000 (16)
C2	0.019 (2)	0.014 (2)	0.0150 (19)	0.0033 (17)	−0.0002 (17)	−0.0002 (16)
C3	0.020 (2)	0.013 (2)	0.0135 (19)	0.0006 (17)	−0.0052 (17)	0.0019 (15)
C4	0.018 (2)	0.016 (2)	0.0150 (19)	−0.0072 (16)	0.0005 (17)	0.0017 (16)
C5	0.018 (2)	0.015 (2)	0.016 (2)	0.0003 (17)	−0.0023 (17)	−0.0006 (16)
C6	0.023 (2)	0.013 (2)	0.017 (2)	−0.0022 (18)	−0.0013 (18)	0.0009 (16)
C7	0.023 (3)	0.014 (2)	0.023 (2)	−0.0007 (17)	0.0010 (19)	−0.0016 (17)
O8	0.0251 (19)	0.0276 (18)	0.0241 (17)	−0.0019 (14)	0.0036 (15)	0.0093 (14)
O9	0.0216 (19)	0.038 (2)	0.0276 (18)	−0.0045 (15)	0.0035 (15)	0.0013 (15)
C10	0.023 (2)	0.014 (2)	0.015 (2)	−0.0007 (18)	0.0048 (18)	0.0001 (16)
O11	0.0274 (18)	0.0132 (15)	0.0265 (17)	−0.0007 (13)	−0.0074 (14)	0.0043 (13)
O12	0.0279 (18)	0.0121 (14)	0.0239 (16)	−0.0039 (13)	−0.0064 (14)	0.0014 (12)
N13	0.021 (2)	0.0148 (18)	0.027 (2)	0.0019 (15)	0.0035 (17)	−0.0026 (15)
O14	0.0255 (19)	0.0217 (16)	0.0233 (17)	0.0028 (14)	−0.0003 (14)	0.0027 (14)

Geometric parameters (Å, °)

I1—C2	2.094 (4)	C5—N13	1.396 (6)
I2—C4	2.100 (4)	C7—O9	1.214 (6)
I3—C6	2.103 (4)	C7—O8	1.308 (6)
C1—C2	1.394 (6)	O8—H8	0.80 (5)
C1—C6	1.398 (6)	C10—O12	1.223 (6)
C1—C7	1.504 (6)	C10—O11	1.299 (6)
C2—C3	1.396 (6)	O11—H11	0.79 (5)
C3—C4	1.391 (6)	N13—H13A	0.88 (4)
C3—C10	1.509 (6)	N13—H13B	0.88 (4)
C4—C5	1.418 (6)	O14—H14A	0.81 (4)
C5—C6	1.393 (6)	O14—H14B	0.81 (4)
C2—C1—C6	119.4 (4)	C5—C6—C1	122.3 (4)
C2—C1—C7	121.0 (4)	C5—C6—I3	119.3 (3)

C6—C1—C7	119.6 (4)	C1—C6—I3	118.4 (3)
C1—C2—C3	119.8 (4)	O9—C7—O8	125.0 (4)
C1—C2—I1	120.0 (3)	O9—C7—C1	122.7 (4)
C3—C2—I1	120.2 (3)	O8—C7—C1	112.3 (4)
C4—C3—C2	120.0 (4)	C7—O8—H8	115 (5)
C4—C3—C10	119.6 (4)	O12—C10—O11	125.3 (4)
C2—C3—C10	120.4 (4)	O12—C10—C3	122.5 (4)
C3—C4—C5	121.3 (4)	O11—C10—C3	112.3 (4)
C3—C4—I2	119.6 (3)	C10—O11—H11	114 (5)
C5—C4—I2	119.0 (3)	C5—N13—H13A	117 (4)
C6—C5—N13	122.0 (4)	C5—N13—H13B	113 (4)
C6—C5—C4	116.9 (4)	H13A—N13—H13B	105 (6)
N13—C5—C4	121.0 (4)	H14A—O14—H14B	108 (6)

C6—C1—C2—C3	−2.2 (6)	N13—C5—C6—C1	−177.4 (4)
C7—C1—C2—C3	179.0 (4)	C4—C5—C6—C1	6.0 (6)
C6—C1—C2—I1	176.7 (3)	N13—C5—C6—I3	5.2 (5)
C7—C1—C2—I1	−2.0 (5)	C4—C5—C6—I3	−171.4 (3)
C1—C2—C3—C4	3.5 (6)	C2—C1—C6—C5	−2.8 (6)
I1—C2—C3—C4	−175.4 (3)	C7—C1—C6—C5	176.0 (4)
C1—C2—C3—C10	−175.6 (4)	C2—C1—C6—I3	174.7 (3)
I1—C2—C3—C10	5.5 (5)	C7—C1—C6—I3	−6.5 (5)
C2—C3—C4—C5	−0.1 (6)	C2—C1—C7—O9	−84.0 (6)
C10—C3—C4—C5	179.1 (4)	C6—C1—C7—O9	97.2 (5)
C2—C3—C4—I2	−176.2 (3)	C2—C1—C7—O8	97.3 (5)
C10—C3—C4—I2	3.0 (5)	C6—C1—C7—O8	−81.5 (5)
C3—C4—C5—C6	−4.6 (6)	C4—C3—C10—O12	75.4 (5)
I2—C4—C5—C6	171.5 (3)	C2—C3—C10—O12	−105.5 (5)
C3—C4—C5—N13	178.8 (4)	C4—C3—C10—O11	−103.6 (4)
I2—C4—C5—N13	−5.1 (5)	C2—C3—C10—O11	75.5 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H11···O14	0.79 (5)	1.75 (5)	2.540 (5)	173 (7)
O8—H8···O12^{i}	0.80 (5)	1.90 (5)	2.662 (5)	161 (7)
O14—H14A···O9^{ii}	0.81 (4)	1.95 (4)	2.751 (5)	170 (6)
O14—H14B···N13^{iii}	0.81 (4)	2.05 (4)	2.841 (5)	166 (6)
N13—H13A···O14^{iv}	0.88 (4)	2.30 (5)	3.067 (6)	147 (5)
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Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*; (ii) −*x*+5/2, *y*−1/2, *z*; (iii) −*x*+2, *y*−1/2, −*z*+1/2; (iv) −*x*+3/2, *y*+1/2, *z*.

Fig. 1

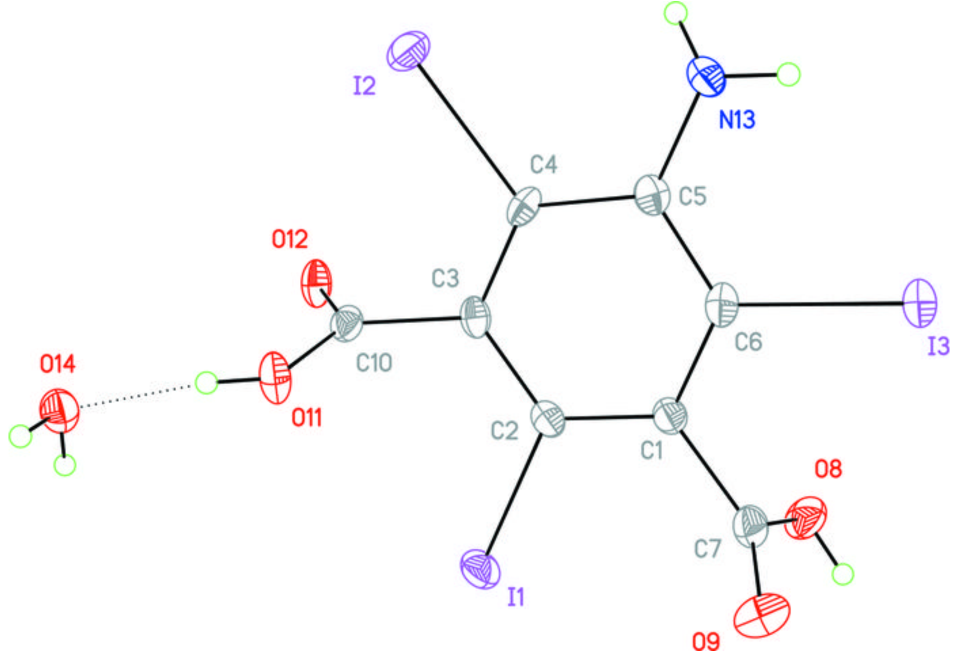

Fig. 2

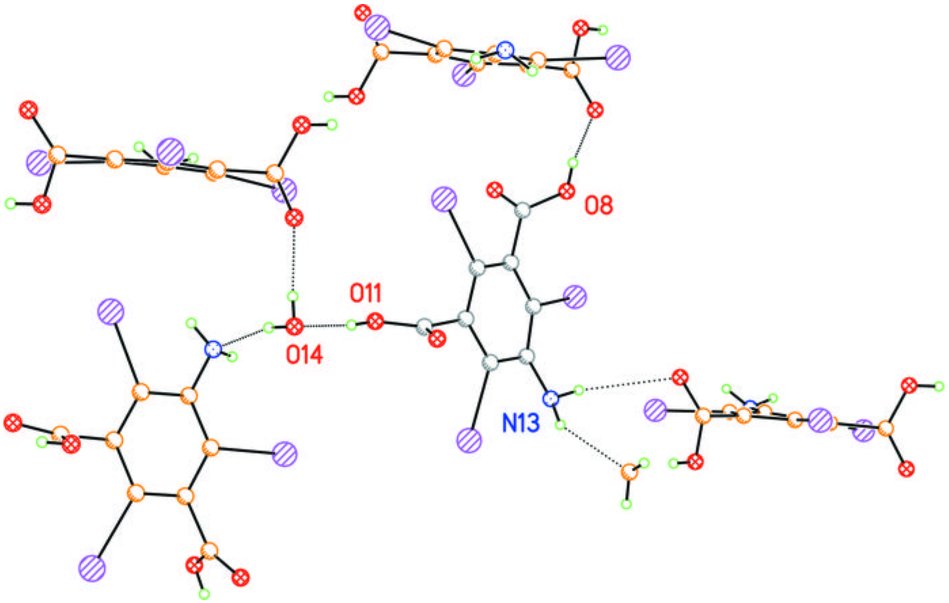

Fig. 3

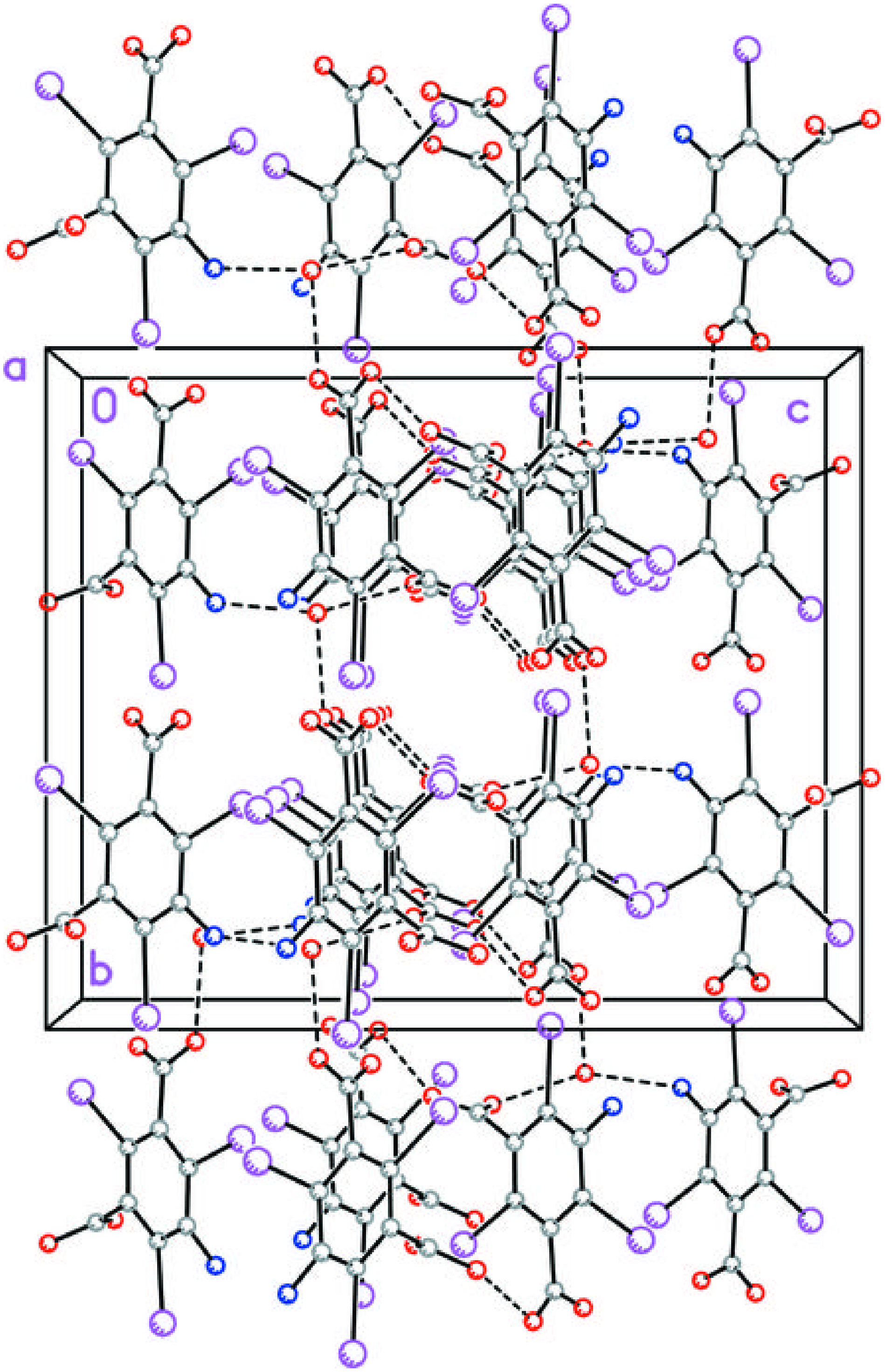

Fig. 4