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3-Hydroxysalicylaldoxime

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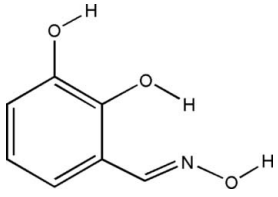
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.117; data-to-parameter ratio = 13.1.

The structure of the title compound, $C_7H_7NO_3$, is based on oximic hydrogen-bonded chains, which are linked together by further phenolic intermolecular hydrogen bonds that form an $R_4^4(14)$ ring motif.

Related literature

Salicylaldoximes without large side groups usually form hydrogen-bonded dimers [*e.g.* Cambridge Structural Database (Allen, 2002) refcodes SALOXM (Pfluger & Harlow, 1973) and ABULIT (Xu *et al.*, 2004)], whereas those bearing large substituents are generally found to form hydrogen-bonded chains [*e.g.* refcodes HEPKET10 (Koziol & Kosturkiewicz, 1984) and HELBOP (Maurin, 1994)], as shown by Smith *et al.* (2003). In common with 3-fluorosalicylaldoxime (Wood *et al.*, 2007) and salicylaldoxime-III (Wood *et al.*, 2006), the structure of 3-hydroxysalicylaldoxime (present work) is an exception to this rule.

For related literature, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_7H_7NO_3$
M_r = 153.14
Monoclinic', $P2_1/c$
a = 13.4603 (10) Å
b = 3.7507 (3) Å
c = 14.8398 (11) Å
α = 90°
β = 114.531 (5)°
γ = 90°
V = 681.57 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 150 K
0.46 × 0.13 × 0.11 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2006)
T_{min} = 0.51, T_{max} = 0.99
8183 measured reflections
1424 independent reflections
904 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.117
S = 0.99
1424 reflections
109 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5^{i}	0.87 (2)	1.95 (2)	2.7984 (18)	167 (3)
O5—H5···N2	0.89 (3)	1.85 (3)	2.651 (2)	148 (3)
O6—H6···O1^{ii}	0.81 (3)	2.16 (2)	2.8353 (18)	141 (3)

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

Data collection: *SMART* (Siemens, 1993); cell refinement: *SAINT*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2053).

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supplementary materials

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3-Hydroxysalicylaldoxime

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Comment

3-Hydroxysalicylaldoxime (I) crystallizes with one molecule in the asymmetric unit in the space group $P2_1/c$. The molecule forms an intramolecular phenolic OH···N hydrogen bond [O5···N2 = 2.651 (2) Å] (Figure 1) and an intermolecular oximic OH···O hydrogen bond [O1···O5 = 2.798 (2) Å] with a neighbouring molecule related by the 2_1 screw axis. These two interactions taken together form a secondary level C(5) chain running parallel to the crystallographic *b* axis (Figure 2). The molecules within the C(5) chain also interact with their next-but-one neighbours through π-π stacking contacts which are related by a translation in the direction of the *b* axis. The inter-plane separation in these stacking interactions, using a calculated least squares mean plane from the phenyl carbons in one molecule and measuring the distances to the phenyl carbons in another molecule, is between 3.458 (2) and 3.471 (2) Å and the dihedral angle between the two phenyl planes is 0° by symmetry.

Each chain interacts with a neighbouring chain through intermolecular OH···O hydrogen bonds [O6···O1 = 2.835 (2) Å] related through the c-glide perpendicular to the *b* axis. These interactions taken with the oximic OH···O contacts and their symmetry equivalents form hydrogen bonded ring motifs around an inversion centre for which the graph-set descriptor is $R^4_4(14)$ (Bernstein *et al.*, 1995). The hydrogen bonded rings connect the chains into slabs in the *bc* plane with the phenyl groups at the edges of the slabs (Figure 3). There are no hydrogen bonding interactions between the slabs and the only interactions are van der Waals contacts.

Experimental

All solvents and reagents were used as received from Aldrich and Fisher. ^{1}H and ^{13}C NMR were obtained using a Bruker AC250 spectrometer at ambient temperature. Chemical shifts (δ) are reported in parts per million (p.p.m.) relative to internal standards. Fast atom bombardment mass spectrometry (FABMS) was carried out using a Kratos MS50TC spectrometer with a thioglycerol matrix. Analytical data was obtained from the University of St Andrews Microanalytical Service.

KOH (1.347 g, 20.4 mmol) and $NH_2OH{\cdot}HCl$ (1.418 g, 20.4 mmol) were dissolved in EtOH, mixed thoroughly and a white KCl precipitate removed by filtration. 3-Hydroxysalicylaldehyde (2.500 g, 18.10 mmol) was added to the filtrate, and the mixture refluxed for 3 hr. The solvent was removed *in vacuo*, and the residue redissolved in $CHCl_3$, washed with water 3 times and dried over $MgSO_4$. The solvent was removed *in vacuo* to yield the crude product as a yellow powder which was then recrystallized from chloroform to give yellow needles (2.371 g, 85.6%). A yellow block suitable for *x*-ray diffraction was grown by slow evaporation of a hexane/chloroform solvent. (Anal. Calc. for $C_7H_7NO_3$: C, 54.9; H, 4.6; N, 9.2. Found: C, 54.9; H, 4.3; N, 9.1%); ^{1}H NMR (250 MHz, MeOD): δ(H) (p.p.m.) 6.80 (m, 3H, 3 *x* ArH), 8.22 (s, 1H, ArCHN); ^{13}C NMR (63 MHz, MeOD) δ(C) (p.p.m.) 118.0 (1 C, aromatic CH), 119.5 (1 C, aromatic C-CHN), 121.0 (1 C, aromatic CH), 122.0 (1 C, aromatic CH), 146.5 (1 C, aromatic C—OH), 152.5 (1 C, ArCHN); FABMS *m*/*z* 154 (MH)+, 48%.

Following data collection (see Table 1) an absorption correction was applied using the program *SADABS*. Tmax/Tmin is larger than calculated on the basis of the crystal dimensions. However, multi-scan procedures (such as *SADABS*) correct for all systematic errors that lead to disparities in the intensities of equivalent data. It is possible that the larger than expected range of transmission is accounted for by crystal decay or absorption by the mounting fibre.

Refinement

The hydrogen atoms were located in a Fourier difference map. The positional and isotropic displacement parameters were then refined subject to restraints [C—H = 0.93 (2) Å, O—H = 0.82 (2) Å and U_{iso}(H) = 1.5Ueq(C or O)]. In subsequent cycles of least squares the H-atoms attached to C were constrained to ride on their parent atoms. H1, H5 and H6 were refined subject to distance restraints equal to 0.84 (5) Å.

Figures

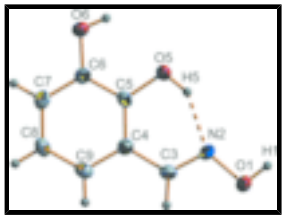

Fig. 1. Molecular structure of I with probability ellipsoids drawn at the 50% level.

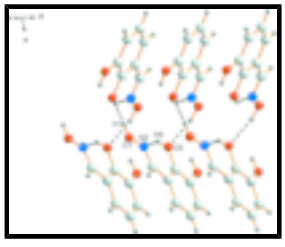

Fig. 2. H-bonded chain formation in the crystal structure of I.

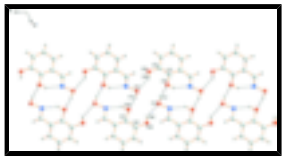

Fig. 3. H-bonded ring motif in the crystal structure of I that connects the structural chains into slabs in the *bc* plane. The extent of one such slab is shown.

3-Hydroxysalicylaldoxime

Crystal data

$C_7H_7N_1O_3$	$Z = 4$
$M_r = 153.14$	$F_{000} = 320$
Monoclinic', $P2_1/c$	$D_x = 1.492$ Mg m^{-3}
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 13.4603$ (10) Å	Cell parameters from 1756 reflections
$b = 3.7507$ (3) Å	$\theta = 3–25^o$
$c = 14.8398$ (11) Å	$\mu = 0.12$ mm^{-1}
$\alpha = 90^o$	$T = 150$ K

β = 114.531 (5)º	Block, yellow
γ = 90º	0.46 × 0.13 × 0.11 mm
V = 681.57 (9) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	904 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.029
T = 150 K	θ_{max} = 26.6º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 2006)	$h = -16 \rightarrow 16$
T_{min} = 0.51, T_{max} = 0.99	$k = -4 \rightarrow 4$
8183 measured reflections	$l = -17 \rightarrow 18$
1424 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: geom/difmap (OH)
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.117	Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 51.1 83.1 48.9 19.2 3.34 *P*rince, E. (1982). Mathematical Techniques in Crystallography and Materials Science. Ne*w* York: Springer-Verlag. Watkin, D. J. (1994). *Acta Cryst.* A**50**, 411–437.
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.0001
1424 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
109 parameters	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
3 restraints	Extinction correction: none

Special details

Experimental. Used Oxford Cryosystems low temperature device. Data collection strategy optimized with COSMO.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.44009 (11)	0.2386 (5)	0.06215 (10)	0.0333
N2	0.39503 (12)	0.3040 (5)	0.13107 (11)	0.0266
C3	0.30117 (14)	0.4508 (5)	0.08998 (13)	0.0258

C4	0.23874 (15)	0.5415 (5)	0.14676 (13)	0.0240
C5	0.28033 (14)	0.4911 (5)	0.24936 (13)	0.0226
O5	0.38212 (10)	0.3513 (4)	0.30415 (10)	0.0277
C6	0.21770 (14)	0.5813 (5)	0.30083 (13)	0.0241
O6	0.25519 (12)	0.5341 (4)	0.40046 (10)	0.0335
C7	0.11473 (15)	0.7240 (5)	0.25057 (15)	0.0272
C8	0.07223 (15)	0.7703 (5)	0.14860 (15)	0.0279
C9	0.13386 (15)	0.6814 (5)	0.09742 (14)	0.0276
H1	0.501 (2)	0.135 (8)	0.099 (2)	0.0512*
H3	0.2687	0.4989	0.0202	0.0300*
H5	0.410 (2)	0.296 (7)	0.261 (2)	0.0424*
H6	0.315 (2)	0.444 (8)	0.423 (2)	0.0511*
H7	0.0735	0.7879	0.2870	0.0341*
H8	0.0024	0.8643	0.1144	0.0319*
H9	0.1071	0.7129	0.0287	0.0318*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0246 (7)	0.0531 (10)	0.0249 (7)	0.0044 (7)	0.0129 (6)	0.0003 (7)
N2	0.0258 (8)	0.0353 (10)	0.0224 (8)	−0.0004 (7)	0.0136 (6)	−0.0014 (7)
C3	0.0262 (9)	0.0291 (10)	0.0207 (8)	−0.0015 (8)	0.0084 (7)	0.0015 (7)
C4	0.0237 (9)	0.0232 (9)	0.0260 (9)	−0.0023 (7)	0.0112 (7)	0.0000 (7)
C5	0.0178 (8)	0.0229 (10)	0.0249 (9)	−0.0022 (7)	0.0065 (7)	−0.0002 (7)
O5	0.0201 (6)	0.0395 (9)	0.0225 (6)	0.0029 (6)	0.0078 (5)	0.0002 (6)
C6	0.0235 (9)	0.0254 (9)	0.0240 (9)	−0.0029 (8)	0.0104 (7)	−0.0012 (8)
O6	0.0288 (7)	0.0516 (10)	0.0210 (7)	0.0063 (7)	0.0111 (6)	0.0014 (7)
C7	0.0262 (9)	0.0254 (9)	0.0333 (10)	−0.0018 (8)	0.0156 (8)	−0.0028 (8)
C8	0.0206 (9)	0.0267 (10)	0.0335 (10)	0.0038 (8)	0.0085 (8)	0.0021 (8)
C9	0.0276 (9)	0.0266 (10)	0.0250 (9)	0.0015 (8)	0.0073 (7)	0.0033 (8)

Geometric parameters (Å, °)

O1—N2	1.4103 (19)	O5—H5	0.89 (2)
O1—H1	0.87 (3)	C6—O6	1.361 (2)
N2—C3	1.277 (2)	C6—C7	1.380 (3)
C3—C4	1.456 (2)	O6—H6	0.81 (3)
C3—H3	0.959	C7—C8	1.389 (3)
C4—C5	1.400 (3)	C7—H7	0.952
C4—C9	1.395 (3)	C8—C9	1.378 (3)
C5—O5	1.374 (2)	C8—H8	0.932
C5—C6	1.394 (2)	C9—H9	0.938
N2—O1—H1	101.7 (18)	C5—C6—C7	119.86 (17)
O1—N2—C3	111.52 (14)	O6—C6—C7	118.27 (16)
N2—C3—C4	121.25 (16)	C6—O6—H6	111.2 (19)
N2—C3—H3	120.9	C6—C7—C8	120.29 (17)
C4—C3—H3	117.8	C6—C7—H7	118.8
C3—C4—C5	122.11 (17)	C8—C7—H7	120.9

C3—C4—C9	119.06 (16)	C7—C8—C9	120.02 (18)
C5—C4—C9	118.83 (17)	C7—C8—H8	120.4
C4—C5—O5	123.05 (16)	C9—C8—H8	119.5
C4—C5—C6	120.23 (17)	C4—C9—C8	120.75 (18)
O5—C5—C6	116.72 (16)	C4—C9—H9	117.8
C5—O5—H5	106.0 (17)	C8—C9—H9	121.4
C5—C6—O6	121.87 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O5^{i}	0.87 (2)	1.95 (2)	2.7984 (18)	167 (3)
O5—H5···N2	0.89 (3)	1.85 (3)	2.651 (2)	148 (3)
O6—H6···O1^{ii}	0.81 (3)	2.16 (2)	2.8353 (18)	141 (3)

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

Fig. 1

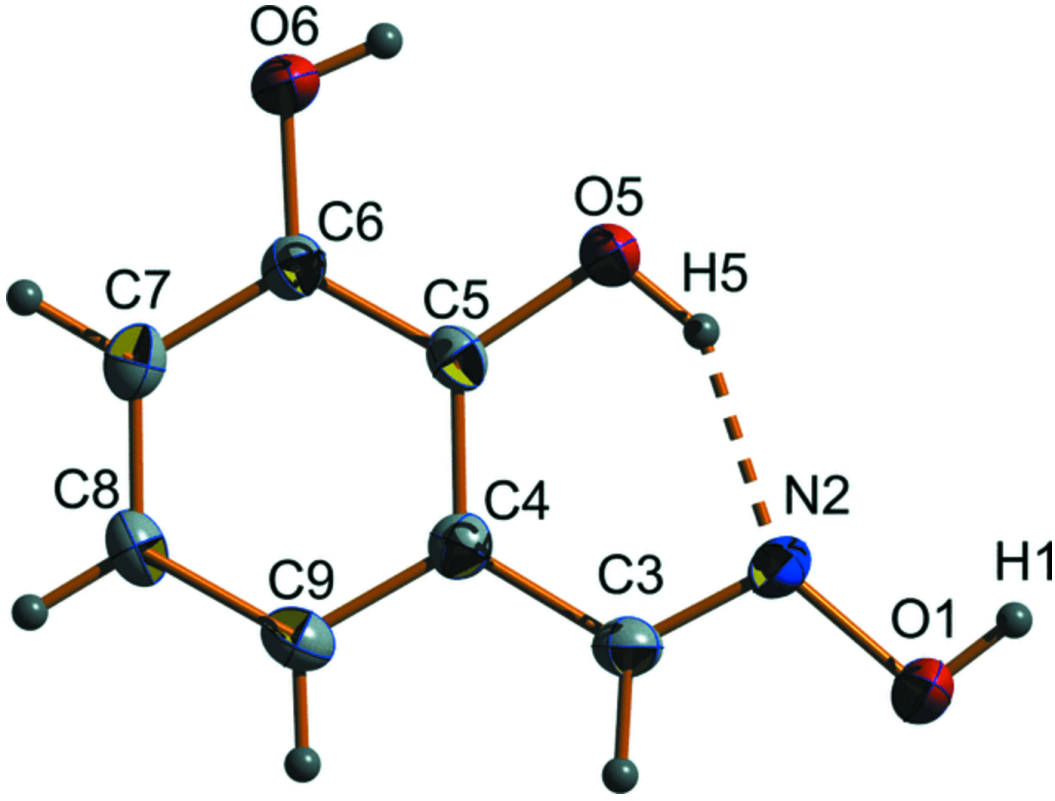

Fig. 2

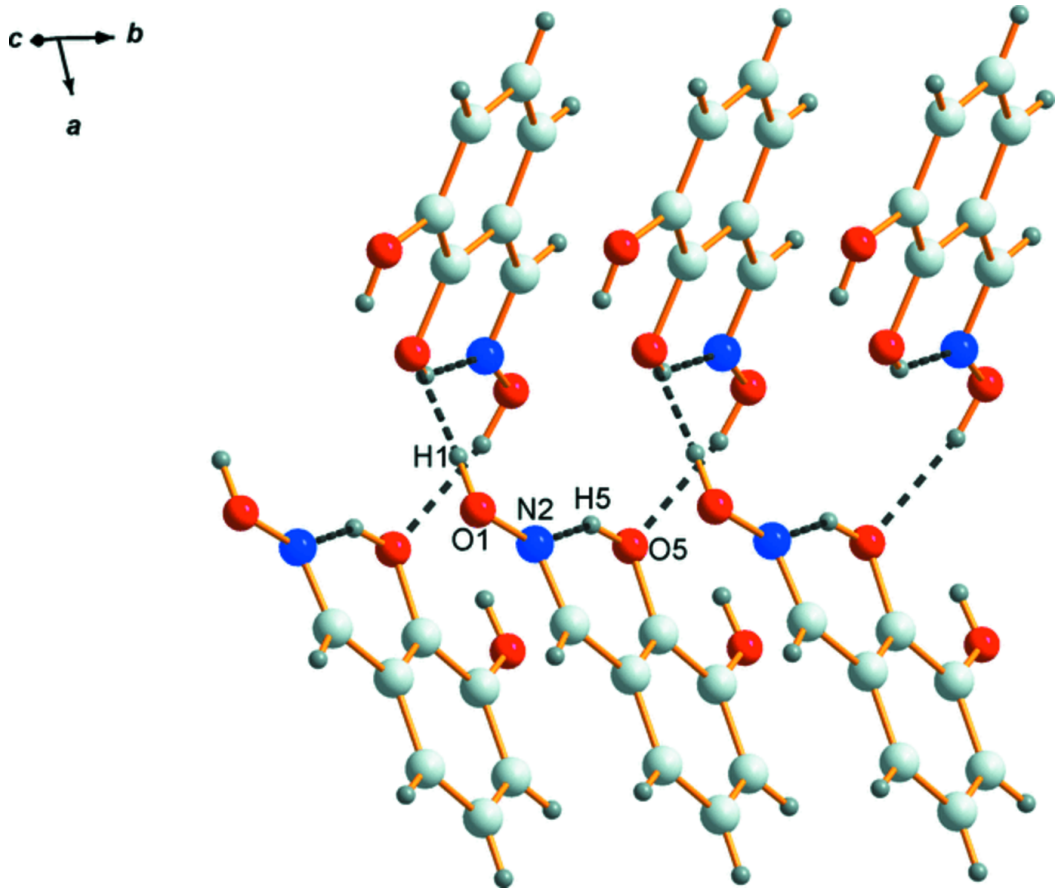
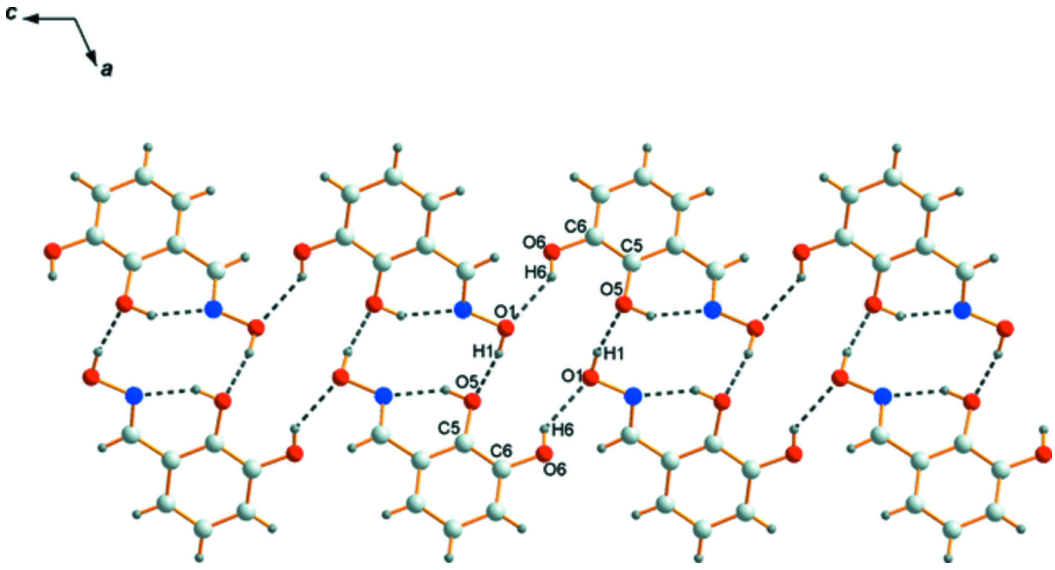
Fig. 3