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4-Methyl-*N*-(3-oxo-1,3-dihydroisobenzofuran-1-yl)benzenesulfonamide[1]

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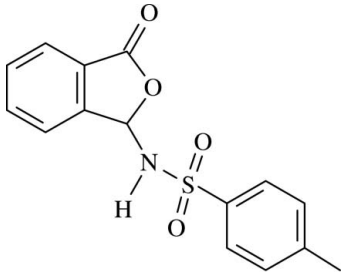
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.041; wR factor = 0.110; data-to-parameter ratio = 14.3.

The title compound, $C_{15}H_{13}NO_4S$, exhibits N—H···O and C—H···O hydrogen-bond interactions that generate $S(5)$ and $R_2^2(9)$ ring motifs arranged as molecular chains along the c axis. The phthalide part of the molecule is planar and is inclined by 67.66 (12)° to the substituted aromatic ring.

Related literature

For related structures (3-halogenophenyl phthalides, halogens = I, Br, Cl, F), see: Odabaşoğlu & Büyükgüngör (2006, 2007*a*, 2007*b*). For ring motif details, see: Bernstein *et al.* (1995); Etter (1990). For general background, see: Aoki *et al.* (1973); Elderfield (1951); Tsi & Tan (1997); Roy & Sarkar (2005); Kubota & Tatsuno (1971).



Experimental

Crystal data

$C_{15}H_{13}NO_4S$
M_r = 303.32
Orthorhombic, $Pca2_1$
a = 17.6356 (14) Å
b = 8.4593 (6) Å
c = 9.3809 (6) Å
V = 1399.49 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 296 K
0.78 × 0.68 × 0.52 mm

Data collection

Stoe IPDS II diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.850, T_{max} = 0.898
11123 measured reflections
2758 independent reflections
2262 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.110
S = 0.98
2758 reflections
193 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), with 1287 Friedel pairs
Flack parameter: −0.08 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O3	0.98	2.40	2.885 (4)	110
N1—H1···O1^{i}	0.87 (4)	2.04 (4)	2.904 (3)	168 (3)

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2415).

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[1] 3-Substituted phthalides. XXXII. For part XXXI, see: Odabaşoğlu & Büyükgüngör (2007*b*).

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supplementary materials

Acta Cryst. (2007). E**63**, o4730 [**doi:10.1107/S1600536807057595**]

4-Methyl-*N*-(3-oxo-1,3-dihydroisobenzofuran-1-yl)benzenesulfonamide

M. Odabasoglu and O. Büyükgüngör

Comment

Phthalides are known to show diverse biological activities as hormones, pheromones and antibiotics (Aoki *et al.*, 1973; Kubota & Tatsuno, 1971; Tsi & Tan, 1997). As part of our on going research on 3-substituted phthalides, the title compound, (I), has been synthesized and its crystal structure is reported here.

The molecule of (I) is built up from a phthalide unit connected to a *p*-toluensulfonyl group through an amino group (Fig. 1). The phthalide part (atoms C1–C8) is essentially planar, the largest deviation from the mean plane being −0.065 (3) Å for atom C8. The dihedral angle between the substituted aromatic ring and the mean plane of the phthalide group is 67.66 (12)°. In (I), the crystal packing is stabilized by N—H···O and C—H···O hydrogen bond interactions that these interactions generate S(5) and $R_2^2(9)$ ring motifs (Bernstein *et al.*, 1995; Etter, 1990). These motifs are arranged in the molecular chains along the *c* axis (Fig. 2, 3 and Table 1).

Experimental

The title compound was prepared according to the method described by Odabaşoğlu & Büyükgüngör (2006), using phthalaldehydic acid and 2-bromoaniline as starting materials (yield 70%; m.p. 430–432 K). Crystals of (I) suitable for *x*-ray analysis were obtained by slow evaporation of an ethanol–DMF (*v*/*v*, 1/1) solution at room temperature.

Refinement

H1 was found in a difference Fourier map and refined freely with U_{iso}(H) = 1.2U_{eq}(N). All other H atoms were included in calculated positions and refined using a riding model approximation. Constrained C—H bond lengths and isotropic U parameters: 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for Csp^2—H; 0.96 Å and U_{iso}(H) = 1.2U_{eq}(C) for methyl C—H; 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) for methine C—H;

Figures

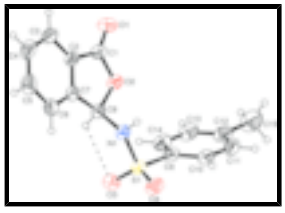

Fig. 1. A view of (I) with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is drawn as a dashed line.

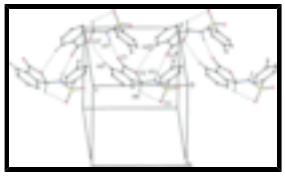

Fig. 2. Part of the crystal structure of (I), showing the formation of S(5) and $R_2^2(9)$ ring motifs. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) 1 − *x*, −*y*, *z* + 1/2; (ii) 1 − *x*, −*y*, *z* − 1/2].

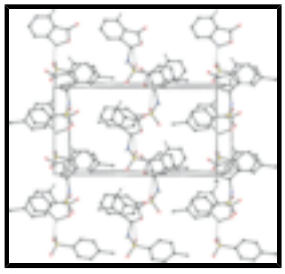

Fig. 3. A packing diagram of (I), with hydrogen bonds drawn as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

4-Methyl-*N*-(3-oxo-1,3-dihydroisobenzofuran-1-yl)benzenesulfonamide

Crystal data

$C_{15}H_{13}NO_4S$	F_{000} = 632
M_r = 303.32	D_x = 1.440 Mg m^{-3}
Orthorhombic, *Pca*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 11123 reflections
a = 17.6356 (14) Å	θ = 2.2–27.1º
b = 8.4593 (6) Å	μ = 0.25 mm^{-1}
c = 9.3809 (6) Å	*T* = 296 K
V = 1399.49 (17) Å^3	Block, colorless
Z = 4	0.78 × 0.68 × 0.52 mm

Data collection

Stoe IPDS II diffractometer	2758 independent reflections
Monochromator: plane graphite	2262 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	R_{int} = 0.063
T = 296 K	θ_{max} = 26.0º
ω scan rotation method	θ_{min} = 2.3º
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	*h* = −21→21
T_{min} = 0.850, T_{max} = 0.898	*k* = −10→10
11123 measured reflections	*l* = −11→11

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0758P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.110$	$(\Delta/\sigma)_{max} = <0.001$
$S = 0.98$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
2758 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
193 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 1287 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.08 (9)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2$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}
C1	0.50446 (16)	0.1283 (3)	0.6188 (3)	0.0549 (6)
C2	0.42320 (17)	0.1352 (3)	0.6529 (3)	0.0548 (6)
C3	0.38379 (19)	0.0680 (4)	0.7648 (3)	0.0704 (8)
H3	0.4081	0.0112	0.8363	0.084*
C4	0.3069 (2)	0.0897 (5)	0.7644 (4)	0.0843 (10)
H4	0.2781	0.0459	0.8375	0.101*
C5	0.27106 (19)	0.1746 (5)	0.6587 (5)	0.0853 (10)
H5	0.2186	0.1858	0.6621	0.102*
C6	0.31076 (17)	0.2436 (4)	0.5477 (3)	0.0712 (8)
H6	0.2866	0.3016	0.4769	0.085*
C7	0.38812 (15)	0.2216 (3)	0.5482 (3)	0.0533 (6)
C8	0.44652 (15)	0.2846 (4)	0.4467 (3)	0.0541 (6)
H8	0.4510	0.3991	0.4601	0.065*
C9	0.55597 (17)	0.3469 (3)	0.1450 (3)	0.0583 (7)
C10	0.57909 (17)	0.2585 (4)	0.0291 (3)	0.0635 (7)
H10	0.5433	0.2155	−0.0326	0.076*
C11	0.65528 (17)	0.2342 (4)	0.0053 (3)	0.0744 (8)
H11	0.6706	0.1751	−0.0732	0.089*
C12	0.7093 (2)	0.2965 (5)	0.0963 (4)	0.0790 (9)
C13	0.68489 (19)	0.3860 (4)	0.2101 (4)	0.0792 (10)
H13	0.7206	0.4296	0.2715	0.095*

C14	0.60886 (19)	0.4129 (4)	0.2355 (3)	0.0710 (8)
H14	0.5936	0.4746	0.3123	0.085*
C15	0.7928 (2)	0.2635 (7)	0.0736 (6)	0.1181 (16)
H15A	0.8217	0.3136	0.1477	0.177*
H15B	0.8082	0.3046	−0.0173	0.177*
H15C	0.8015	0.1515	0.0760	0.177*
N1	0.43189 (14)	0.2530 (3)	0.3006 (2)	0.0565 (5)
O1	0.55549 (11)	0.0622 (2)	0.6801 (3)	0.0732 (6)
O2	0.51692 (10)	0.2083 (2)	0.49564 (18)	0.0565 (5)
O3	0.45049 (14)	0.5308 (3)	0.2340 (3)	0.0826 (7)
O4	0.41807 (13)	0.3302 (3)	0.0519 (2)	0.0806 (7)
S1	0.45901 (4)	0.37722 (8)	0.17674 (8)	0.0604 (2)
H1	0.4318 (19)	0.154 (4)	0.275 (4)	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0634 (17)	0.0530 (14)	0.0484 (12)	−0.0054 (12)	−0.0059 (11)	0.0036 (12)
C2	0.0698 (15)	0.0511 (13)	0.0437 (16)	−0.0015 (11)	0.0050 (11)	−0.0041 (11)
C3	0.086 (2)	0.0747 (19)	0.0501 (15)	−0.0029 (16)	0.0070 (13)	0.0096 (14)
C4	0.084 (2)	0.101 (3)	0.068 (2)	−0.0044 (18)	0.0281 (17)	0.0039 (19)
C5	0.0626 (17)	0.109 (3)	0.084 (2)	0.0072 (15)	0.0189 (18)	−0.013 (2)
C6	0.0687 (18)	0.082 (2)	0.0626 (18)	0.0150 (14)	0.0033 (14)	−0.0082 (15)
C7	0.0611 (14)	0.0559 (14)	0.0429 (12)	0.0031 (12)	0.0042 (11)	−0.0060 (11)
C8	0.0662 (16)	0.0537 (14)	0.0423 (13)	0.0050 (11)	−0.0038 (11)	−0.0034 (11)
C9	0.0696 (17)	0.0575 (15)	0.0479 (18)	−0.0043 (12)	−0.0009 (11)	0.0121 (11)
C10	0.0699 (17)	0.0698 (18)	0.0510 (14)	−0.0009 (14)	−0.0029 (13)	0.0052 (12)
C11	0.0726 (19)	0.091 (2)	0.0599 (17)	0.0042 (16)	0.0078 (15)	0.0041 (16)
C12	0.0708 (19)	0.098 (2)	0.068 (2)	−0.0084 (17)	0.0042 (16)	0.0262 (19)
C13	0.0732 (19)	0.095 (2)	0.069 (2)	−0.0242 (17)	−0.0102 (15)	0.0147 (17)
C14	0.081 (2)	0.074 (2)	0.0571 (15)	−0.0119 (16)	−0.0037 (15)	0.0019 (14)
C15	0.064 (2)	0.183 (5)	0.107 (3)	−0.006 (3)	0.006 (2)	0.030 (3)
N1	0.0689 (13)	0.0603 (14)	0.0403 (11)	0.0019 (12)	−0.0018 (10)	−0.0046 (10)
O1	0.0701 (12)	0.0725 (12)	0.0770 (13)	−0.0017 (9)	−0.0159 (13)	0.0212 (14)
O2	0.0548 (9)	0.0659 (12)	0.0488 (10)	−0.0025 (8)	−0.0037 (8)	0.0062 (9)
O3	0.1074 (18)	0.0654 (13)	0.0748 (14)	0.0200 (11)	0.0085 (12)	0.0102 (11)
O4	0.0702 (13)	0.1198 (19)	0.0519 (11)	0.0065 (12)	−0.0136 (10)	0.0065 (12)
S1	0.0688 (4)	0.0671 (4)	0.0454 (3)	0.0099 (3)	−0.0024 (3)	0.0113 (4)

Geometric parameters (Å, °)

C1—O1	1.206 (3)	C9—C10	1.381 (4)
C1—O2	1.357 (3)	C9—S1	1.754 (3)
C1—C2	1.469 (4)	C10—C11	1.378 (4)
C2—C7	1.372 (4)	C10—H10	0.9300
C2—C3	1.381 (4)	C11—C12	1.383 (5)
C3—C4	1.368 (5)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.377 (5)
C4—C5	1.378 (6)	C12—C15	1.514 (5)

C4—H4	0.9300	C13—C14	1.381 (5)
C5—C6	1.384 (5)	C13—H13	0.9300
C5—H5	0.9300	C14—H14	0.9300
C6—C7	1.377 (4)	C15—H15A	0.9600
C6—H6	0.9300	C15—H15B	0.9600
C7—C8	1.501 (4)	C15—H15C	0.9600
C8—N1	1.419 (3)	N1—S1	1.638 (3)
C8—O2	1.473 (3)	N1—H1	0.87 (4)
C8—H8	0.9800	O3—S1	1.414 (3)
C9—C14	1.379 (4)	O4—S1	1.432 (2)
O1—C1—O2	121.1 (3)	C11—C10—H10	120.1
O1—C1—C2	130.0 (3)	C9—C10—H10	120.1
O2—C1—C2	108.9 (2)	C10—C11—C12	120.9 (3)
C7—C2—C3	122.4 (3)	C10—C11—H11	119.5
C7—C2—C1	107.8 (2)	C12—C11—H11	119.5
C3—C2—C1	129.7 (3)	C13—C12—C11	118.2 (3)
C4—C3—C2	116.2 (3)	C13—C12—C15	120.9 (4)
C4—C3—H3	121.9	C11—C12—C15	120.8 (4)
C2—C3—H3	121.9	C12—C13—C14	121.8 (3)
C3—C4—C5	121.8 (3)	C12—C13—H13	119.1
C3—C4—H4	119.1	C14—C13—H13	119.1
C5—C4—H4	119.1	C9—C14—C13	119.0 (3)
C4—C5—C6	121.9 (3)	C9—C14—H14	120.5
C4—C5—H5	119.0	C13—C14—H14	120.5
C6—C5—H5	119.0	C12—C15—H15A	109.5
C7—C6—C5	116.2 (3)	C12—C15—H15B	109.5
C7—C6—H6	121.9	H15A—C15—H15B	109.5
C5—C6—H6	121.9	C12—C15—H15C	109.5
C2—C7—C6	121.4 (3)	H15A—C15—H15C	109.5
C2—C7—C8	109.5 (2)	H15B—C15—H15C	109.5
C6—C7—C8	129.1 (3)	C8—N1—S1	120.7 (2)
N1—C8—O2	111.8 (2)	C8—N1—H1	116 (3)
N1—C8—C7	114.9 (2)	S1—N1—H1	115 (2)
O2—C8—C7	103.0 (2)	C1—O2—C8	110.3 (2)
N1—C8—H8	109.0	O3—S1—O4	120.82 (15)
O2—C8—H8	109.0	O3—S1—N1	106.81 (14)
C7—C8—H8	109.0	O4—S1—N1	104.76 (14)
C14—C9—C10	120.2 (3)	O3—S1—C9	107.59 (14)
C14—C9—S1	119.7 (2)	O4—S1—C9	108.17 (13)
C10—C9—S1	120.0 (2)	N1—S1—C9	108.14 (12)
C11—C10—C9	119.8 (3)		
O1—C1—C2—C7	−178.2 (3)	C10—C11—C12—C13	−1.2 (5)
O2—C1—C2—C7	−0.5 (3)	C10—C11—C12—C15	177.3 (4)
O1—C1—C2—C3	0.3 (5)	C11—C12—C13—C14	0.7 (5)
O2—C1—C2—C3	178.0 (3)	C15—C12—C13—C14	−177.8 (4)
C7—C2—C3—C4	1.2 (5)	C10—C9—C14—C13	−1.5 (4)
C1—C2—C3—C4	−177.1 (3)	S1—C9—C14—C13	179.1 (2)
C2—C3—C4—C5	−0.4 (5)	C12—C13—C14—C9	0.6 (5)

C3—C4—C5—C6	−0.5 (6)	O2—C8—N1—S1	−94.8 (3)
C4—C5—C6—C7	0.5 (5)	C7—C8—N1—S1	148.3 (2)
C3—C2—C7—C6	−1.2 (4)	O1—C1—O2—C8	−177.1 (3)
C1—C2—C7—C6	177.4 (3)	C2—C1—O2—C8	4.9 (3)
C3—C2—C7—C8	177.3 (3)	N1—C8—O2—C1	−130.8 (2)
C1—C2—C7—C8	−4.1 (3)	C7—C8—O2—C1	−7.0 (3)
C5—C6—C7—C2	0.3 (4)	C8—N1—S1—O3	−35.7 (3)
C5—C6—C7—C8	−177.9 (3)	C8—N1—S1—O4	−165.0 (2)
C2—C7—C8—N1	128.5 (3)	C8—N1—S1—C9	79.8 (2)
C6—C7—C8—N1	−53.1 (4)	C14—C9—S1—O3	34.3 (3)
C2—C7—C8—O2	6.7 (3)	C10—C9—S1—O3	−145.1 (2)
C6—C7—C8—O2	−175.0 (3)	C14—C9—S1—O4	166.4 (2)
C14—C9—C10—C11	1.0 (4)	C10—C9—S1—O4	−13.1 (3)
S1—C9—C10—C11	−179.6 (2)	C14—C9—S1—N1	−80.7 (3)
C9—C10—C11—C12	0.4 (5)	C10—C9—S1—N1	99.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O3	0.98	2.40	2.885 (4)	110
N1—H1···O1^{i}	0.87 (4)	2.04 (4)	2.904 (3)	168 (3)

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

Fig. 1

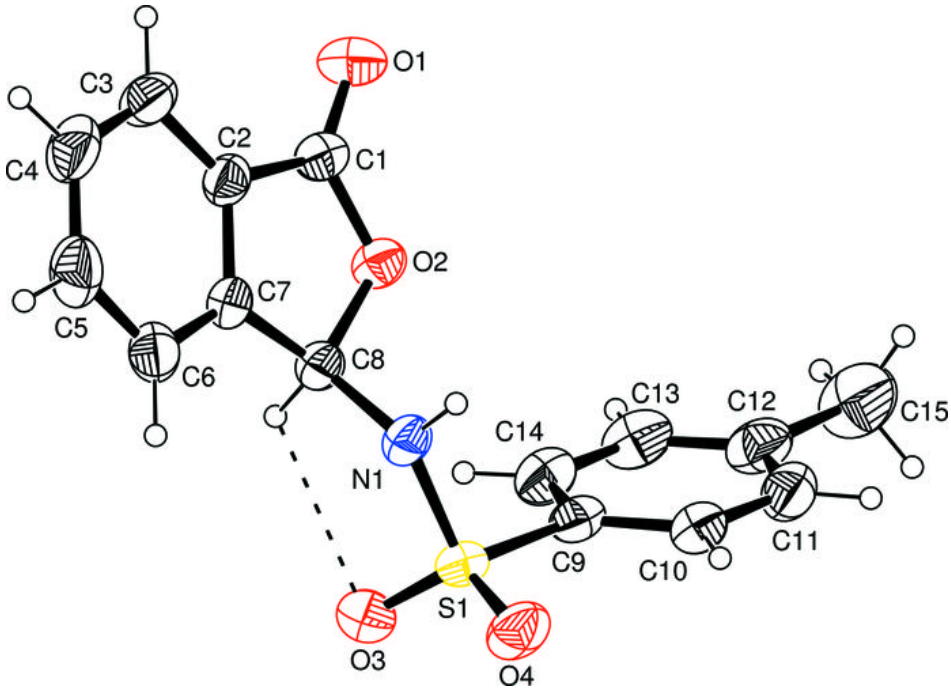

Fig. 2

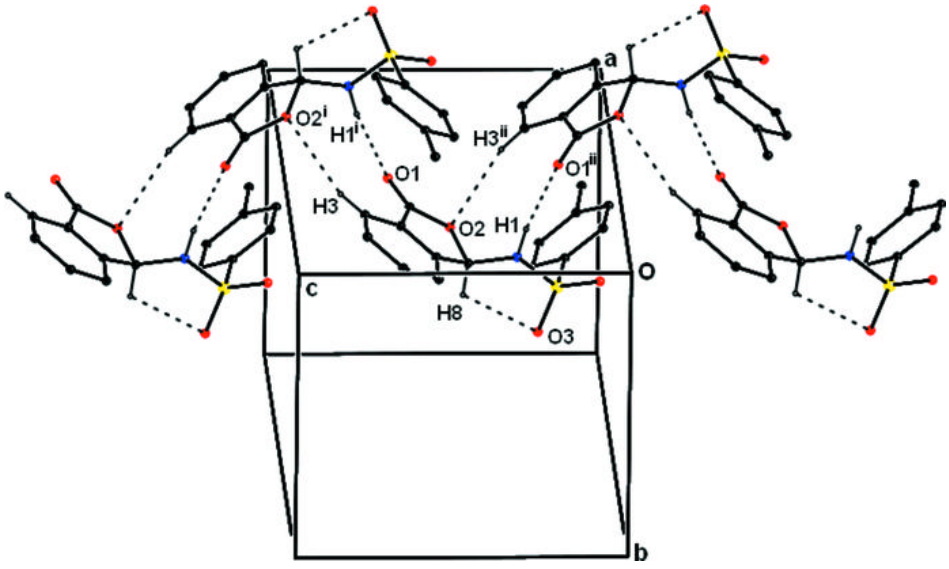

Fig. 3