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3,3-Dichloro-1-ethyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

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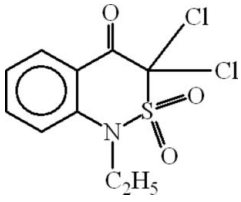
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.110; data-to-parameter ratio = 20.0.

In the title compound, $C_{10}H_9Cl_2NO_3S$, the S atom, which is a component atom of a heterocyclic ring, shows tetrahedral coordination. The heterocyclic ring is not planar.

Related literature

For related compounds, see: Arshad *et al.* (2008); Shafiq, Khan *et al.* (2008); Shafiq, Tahir *et al.* (2008); Tahir *et al.* (2008).



Experimental

Crystal data

$C_{10}H_9Cl_2NO_3S$
M_r = 294.14
Monoclinic, $P2_1/c$
a = 7.7416 (2) Å
b = 11.9185 (3) Å
c = 12.9614 (3) Å
β = 95.995 (2)°
V = 1189.39 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.72 mm^{-1}
T = 296 (2) K
0.24 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.838, T_{max} = 0.881
12499 measured reflections
3082 independent reflections
1872 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.110
S = 1.01
3082 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2539).

References

Arshad, M. N., Tahir, M. N., Khan, I. U., Shafiq, M. & Siddiqui, W. A. (2008). *Acta Cryst.* E**64**, o2045.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Shafiq, M., Khan, I. U., Tahir, M. N. & Siddiqui, W. A. (2008). *Acta Cryst.* E**64**, o558.
Shafiq, M., Tahir, M. N., Khan, I. U., Ahmad, S. & Siddiqui, W. A. (2008). *Acta Cryst.* E**64**, o1270.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tahir, M. N., Shafiq, M., Khan, I. U., Siddiqui, W. A. & Arshad, M. N. (2008). *Acta Cryst.* E**64**, o557.

supplementary materials

Acta Cryst. (2009). E**65**, o430 [**doi:10.1107/S1600536809003079**]

3,3-Dichloro-1-ethyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

M. Shafiq, M. N. Tahir, I. U. Khan, S. Ahmad and M. N. Arshad

Comment

In continuation to the formation of different 2,1-Benzothiazine (Shafiq, Khan *et al.*, 2008), (Tahir *et al.*, 2008), (Arshad *et al.*, 2008), the title compound (I), (Fig 1), has been prepared.

We compare the bond distances and bond angles realised in (I) with the corresponding values observed in 3,3-Dibromo-1-ethyl-1*H*-2,1-benzothiazin- 4(3*H*)-one 2,2-dioxide (II) (Shafiq, Tahir *et al.*, 2008), which is structural isomer of (I). The bond distances S1—C8 [1.817 (2) Å] and S1—N1 [1.625 (2) Å] are larger as compared to 1.792 (8) and 1.617 (6) Å, respectively. This change in the thiazine ring is observed due to the reduction of C–Cl [1.744 (2), 1.766 (2) Å] bonds as compared with C—Br [1.898 (7), 1.947 (8) Å] bonds. The dihedral angle of benzene ring with *N*-ethyl moiety and the SO_2 group is 78.08 (25)° and 77.99 (11)°, respectively. There exist intermolecular H-bonds (Table 1), due to which the molecules are connected in helical way along the *c* axis.

Experimental

The title copound was prepared following the same method as in Shafiq, Tahir *et al.* (2008). A mixture of 1-Ethyl-1*H*-2,1 benzothiazin-4(3*H*)-one 2,2 dioxide (Shafiq, Khan *et al.*, 2008)(34 mg, 0.151 mmol), *N*-Chloro Succinamide (40.2 mg, 0.302 mmol) and Benzoylperoxide (2.11 mg, 0.009 mmol) in Carbon Tetra Chloride (10 ml), was heated under reflux for two hours. CCl_4 was evaporated under reduced pressure and the residue was recrystallized in ethanol for X-ray diffraction studies.

Figures

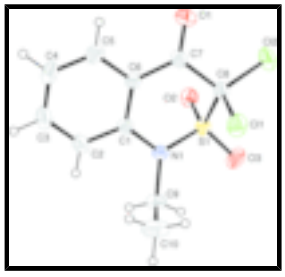

Fig. 1. *ORTEP* drawing of the title compound, with the atom numbering scheme. The thermal ellipsoids are drawn at the 30% probability level. H-atoms are shown by small circles of arbitrary radii. The dotted lines show the intramolecular H-bonds.

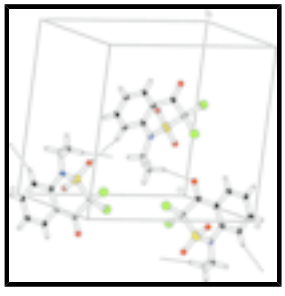

Fig. 2. The partial packing figure (*PLATON*: Spek, 2003) which shows that molecules are connected through intermolecular H-bonds along the *c* axis in helical way.

3,3-Dichloro-1-ethyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

Crystal data

$C_{10}H_9Cl_2NO_3S$	$F_{000} = 600$
$M_r = 294.14$	$D_x = 1.643$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3082 reflections
$a = 7.7416$ (2) Å	$\theta = 2.3$–28.7º
$b = 11.9185$ (3) Å	$\mu = 0.72$ mm^{-1}
$c = 12.9614$ (3) Å	$T = 296$ (2) K
$\beta = 95.995$ (2)º	Prismatic, colorless
$V = 1189.39$ (5) Å^3	0.24 × 0.20 × 0.18 mm
$Z = 4$	

Data collection

Bruker Kappa APEXII CCD diffractometer	3082 independent reflections
Radiation source: fine-focus sealed tube	1872 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.041$
Detector resolution: 7.40 pixels mm^{-1}	$\theta_{max} = 28.7$º
$T = 296(2)$ K	$\theta_{min} = 2.3$º
ω scans	$h = -10 \rightarrow 10$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -16 \rightarrow 16$
$T_{min} = 0.838$, $T_{max} = 0.881$	$l = -17 \rightarrow 15$
12499 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.3265P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3082 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > \sigma(F^2)$ 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}
Cl1	0.45866 (9)	0.41954 (6)	0.18953 (6)	0.0592 (3)
Cl2	0.21735 (10)	0.55261 (7)	0.05418 (5)	0.0649 (3)
S1	0.08395 (8)	0.40174 (5)	0.20115 (5)	0.0421 (2)
O1	0.2953 (3)	0.68290 (16)	0.23915 (16)	0.0637 (8)
O2	−0.0624 (2)	0.47361 (15)	0.19971 (13)	0.0498 (6)
O3	0.0781 (3)	0.30825 (16)	0.13333 (15)	0.0635 (7)
N1	0.1508 (3)	0.36020 (16)	0.31795 (15)	0.0420 (7)
C1	0.1973 (3)	0.4423 (2)	0.39506 (17)	0.0359 (7)
C2	0.1918 (3)	0.4159 (2)	0.49864 (19)	0.0472 (9)
C3	0.2348 (4)	0.4945 (3)	0.5745 (2)	0.0579 (10)
C4	0.2803 (4)	0.6011 (3)	0.5502 (2)	0.0592 (10)
C5	0.2877 (3)	0.6290 (2)	0.4482 (2)	0.0510 (9)
C6	0.2506 (3)	0.5507 (2)	0.36908 (18)	0.0369 (7)
C7	0.2704 (3)	0.5872 (2)	0.26343 (19)	0.0418 (8)
C8	0.2613 (3)	0.4951 (2)	0.17788 (17)	0.0419 (8)
C9	0.1481 (4)	0.2393 (2)	0.3441 (2)	0.0549 (10)
C10	0.3251 (4)	0.1952 (3)	0.3774 (3)	0.0811 (14)
H2	0.15879	0.34423	0.51707	0.0567*
H3	0.23274	0.47473	0.64379	0.0693*
H4	0.30605	0.65411	0.60215	0.0711*
H5	0.31804	0.70172	0.43143	0.0612*
H9A	0.09821	0.19756	0.28397	0.0658*
H9B	0.07472	0.22786	0.39944	0.0658*
H10A	0.31778	0.11692	0.39385	0.1217*
H10B	0.37431	0.23552	0.43749	0.1217*
H10C	0.39743	0.20468	0.32215	0.1217*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0513 (4)	0.0709 (5)	0.0564 (4)	0.0130 (3)	0.0109 (3)	0.0003 (4)
Cl2	0.0770 (5)	0.0835 (6)	0.0339 (3)	0.0038 (4)	0.0045 (3)	0.0121 (3)
S1	0.0482 (3)	0.0423 (4)	0.0347 (3)	−0.0006 (3)	−0.0011 (3)	−0.0072 (3)

O1	0.0897 (15)	0.0416 (12)	0.0612 (13)	−0.0127 (10)	0.0142 (11)	0.0090 (10)
O2	0.0442 (10)	0.0585 (12)	0.0450 (10)	0.0060 (8)	−0.0028 (8)	−0.0015 (9)
O3	0.0797 (14)	0.0552 (12)	0.0542 (12)	−0.0042 (10)	−0.0001 (10)	−0.0249 (10)
N1	0.0558 (13)	0.0286 (11)	0.0403 (12)	−0.0035 (9)	−0.0005 (9)	0.0020 (9)
C1	0.0369 (12)	0.0374 (13)	0.0327 (12)	0.0025 (10)	0.0006 (9)	−0.0007 (10)
C2	0.0503 (14)	0.0531 (17)	0.0383 (14)	0.0035 (12)	0.0047 (11)	0.0094 (12)
C3	0.0592 (18)	0.082 (2)	0.0317 (14)	0.0094 (16)	0.0013 (12)	−0.0010 (14)
C4	0.0635 (18)	0.073 (2)	0.0393 (16)	0.0020 (16)	−0.0035 (13)	−0.0213 (15)
C5	0.0576 (17)	0.0452 (16)	0.0488 (16)	−0.0049 (12)	−0.0008 (12)	−0.0114 (13)
C6	0.0403 (13)	0.0362 (13)	0.0336 (12)	0.0004 (10)	0.0004 (10)	−0.0020 (10)
C7	0.0415 (13)	0.0429 (15)	0.0408 (14)	−0.0032 (11)	0.0032 (10)	0.0014 (12)
C8	0.0464 (14)	0.0494 (15)	0.0299 (13)	0.0041 (11)	0.0034 (10)	0.0034 (11)
C9	0.0631 (18)	0.0348 (15)	0.0662 (19)	−0.0044 (13)	0.0045 (14)	0.0063 (13)
C10	0.075 (2)	0.0483 (19)	0.121 (3)	0.0103 (16)	0.015 (2)	0.0158 (19)

Geometric parameters (Å, °)

Cl1—C8	1.766 (2)	C5—C6	1.394 (3)
Cl2—C8	1.744 (2)	C6—C7	1.460 (3)
S1—O2	1.4189 (18)	C7—C8	1.557 (3)
S1—O3	1.417 (2)	C9—C10	1.489 (4)
S1—N1	1.625 (2)	C2—H2	0.9300
S1—C8	1.817 (2)	C3—H3	0.9300
O1—C7	1.204 (3)	C4—H4	0.9300
N1—C1	1.418 (3)	C5—H5	0.9300
N1—C9	1.481 (3)	C9—H9A	0.9700
C1—C2	1.384 (3)	C9—H9B	0.9700
C1—C6	1.408 (3)	C10—H10A	0.9600
C2—C3	1.374 (4)	C10—H10B	0.9600
C3—C4	1.364 (5)	C10—H10C	0.9600
C4—C5	1.370 (4)		
Cl1···O1	3.469 (2)	C2···C3^{iv}	3.506 (4)
Cl1···O3	3.244 (2)	C2···C2^{iv}	3.586 (3)
Cl1···N1	3.127 (2)	C3···C2^{iv}	3.506 (4)
Cl1···C1	3.520 (2)	C3···O2^{iv}	3.363 (3)
Cl2···O3	3.306 (2)	C3···C1^{iv}	3.491 (4)
Cl2···O1	2.867 (2)	C6···O2	3.229 (3)
Cl2···O2	3.1604 (18)	C9···O2^{viii}	3.273 (3)
Cl2···O2^{i}	3.3972 (18)	C10···C2	3.285 (4)
Cl1···H10C	3.1500	C10···O2^{viii}	3.418 (4)
Cl2···H10A^{ii}	3.0600	C1···H10B	2.8500
O1···Cl1	3.469 (2)	C2···H10B	2.7400
O1···Cl2	2.867 (2)	C2···H9B	2.6900
O2···Cl2	3.1604 (18)	C9···H2	2.5600
O2···C6	3.229 (3)	C10···H2	2.9300
O2···Cl2^{i}	3.3972 (18)	H2···C9	2.5600
O2···C10^{iii}	3.418 (4)	H2···C10	2.9300

O2···C3^{iv}	3.363 (3)	H2···H9B	2.1100
O2···C9^{iii}	3.273 (3)	H2···H10B	2.4300
O3···C2^{ii}	3.359 (3)	H2···O3^{vii}	2.4800
O3···Cl2	3.306 (2)	H3···O2^{iv}	2.6100
O3···Cl1	3.244 (2)	H4···O1^{ix}	2.6400
O1···H5	2.4900	H5···O1	2.4900
O1···H10C^{v}	2.6000	H9A···O3	2.3500
O1···H4^{vi}	2.6400	H9A···O2^{viii}	2.6900
O2···H3^{iv}	2.6100	H9B···C2	2.6900
O2···H9A^{iii}	2.6900	H9B···H2	2.1100
O2···H10A^{iii}	2.7900	H10A···O2^{viii}	2.7900
O3···H9A	2.3500	H10A···Cl2^{vii}	3.0600
O3···H2^{ii}	2.4800	H10B···C1	2.8500
N1···Cl1	3.127 (2)	H10B···C2	2.7400
C1···Cl1	3.520 (2)	H10B···H2	2.4300
C1···C3^{iv}	3.491 (4)	H10C···Cl1	3.1500
C2···O3^{vii}	3.359 (3)	H10C···O1^{x}	2.6000
C2···C10	3.285 (4)		
O2—S1—O3	119.52 (12)	Cl1—C8—C7	108.92 (16)
O2—S1—N1	111.86 (11)	Cl2—C8—S1	108.36 (12)
O2—S1—C8	104.14 (11)	Cl2—C8—C7	111.57 (17)
O3—S1—N1	108.95 (11)	S1—C8—C7	107.00 (15)
O3—S1—C8	110.75 (12)	N1—C9—C10	112.0 (2)
N1—S1—C8	99.71 (11)	C1—C2—H2	120.00
S1—N1—C1	118.62 (16)	C3—C2—H2	120.00
S1—N1—C9	119.93 (16)	C2—C3—H3	119.00
C1—N1—C9	121.25 (19)	C4—C3—H3	119.00
N1—C1—C2	119.7 (2)	C3—C4—H4	120.00
N1—C1—C6	121.6 (2)	C5—C4—H4	120.00
C2—C1—C6	118.7 (2)	C4—C5—H5	119.00
C1—C2—C3	120.6 (2)	C6—C5—H5	119.00
C2—C3—C4	121.3 (2)	N1—C9—H9A	109.00
C3—C4—C5	119.1 (3)	N1—C9—H9B	109.00
C4—C5—C6	121.4 (2)	C10—C9—H9A	109.00
C1—C6—C5	118.8 (2)	C10—C9—H9B	109.00
C1—C6—C7	124.1 (2)	H9A—C9—H9B	108.00
C5—C6—C7	117.2 (2)	C9—C10—H10A	109.00
O1—C7—C6	124.2 (2)	C9—C10—H10B	110.00
O1—C7—C8	118.6 (2)	C9—C10—H10C	109.00
C6—C7—C8	117.2 (2)	H10A—C10—H10B	110.00
Cl1—C8—Cl2	111.28 (13)	H10A—C10—H10C	109.00
Cl1—C8—S1	109.60 (13)	H10B—C10—H10C	109.00
O2—S1—N1—C1	−57.1 (2)	N1—C1—C2—C3	−179.4 (2)
O2—S1—N1—C9	117.7 (2)	C6—C1—C2—C3	1.0 (4)
O3—S1—N1—C1	168.51 (19)	N1—C1—C6—C5	177.4 (2)
O3—S1—N1—C9	−16.6 (2)	N1—C1—C6—C7	−2.8 (4)

C8—S1—N1—C1	52.5 (2)	C2—C1—C6—C5	−3.1 (3)
C8—S1—N1—C9	−132.7 (2)	C2—C1—C6—C7	176.8 (2)
O2—S1—C8—Cl1	174.51 (11)	C1—C2—C3—C4	1.5 (4)
O2—S1—C8—Cl2	−63.88 (14)	C2—C3—C4—C5	−1.8 (5)
O2—S1—C8—C7	56.55 (17)	C3—C4—C5—C6	−0.3 (4)
O3—S1—C8—Cl1	−55.78 (15)	C4—C5—C6—C1	2.8 (4)
O3—S1—C8—Cl2	65.83 (16)	C4—C5—C6—C7	−177.1 (2)
O3—S1—C8—C7	−173.73 (16)	C1—C6—C7—O1	170.6 (3)
N1—S1—C8—Cl1	58.88 (14)	C1—C6—C7—C8	−10.4 (3)
N1—S1—C8—Cl2	−179.52 (12)	C5—C6—C7—O1	−9.6 (4)
N1—S1—C8—C7	−59.08 (17)	C5—C6—C7—C8	169.4 (2)
S1—N1—C1—C2	156.00 (19)	O1—C7—C8—Cl1	103.2 (2)
S1—N1—C1—C6	−24.5 (3)	O1—C7—C8—Cl2	−20.0 (3)
C9—N1—C1—C2	−18.8 (4)	O1—C7—C8—S1	−138.4 (2)
C9—N1—C1—C6	160.7 (2)	C6—C7—C8—Cl1	−75.9 (2)
S1—N1—C9—C10	118.6 (2)	C6—C7—C8—Cl2	160.91 (17)
C1—N1—C9—C10	−66.7 (3)	C6—C7—C8—S1	42.5 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O3^{vii}	0.9300	2.4800	3.359 (3)	157.00
C10—H10C···O1^{x}	0.9600	2.6000	3.445 (4)	147.00

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

Fig. 1

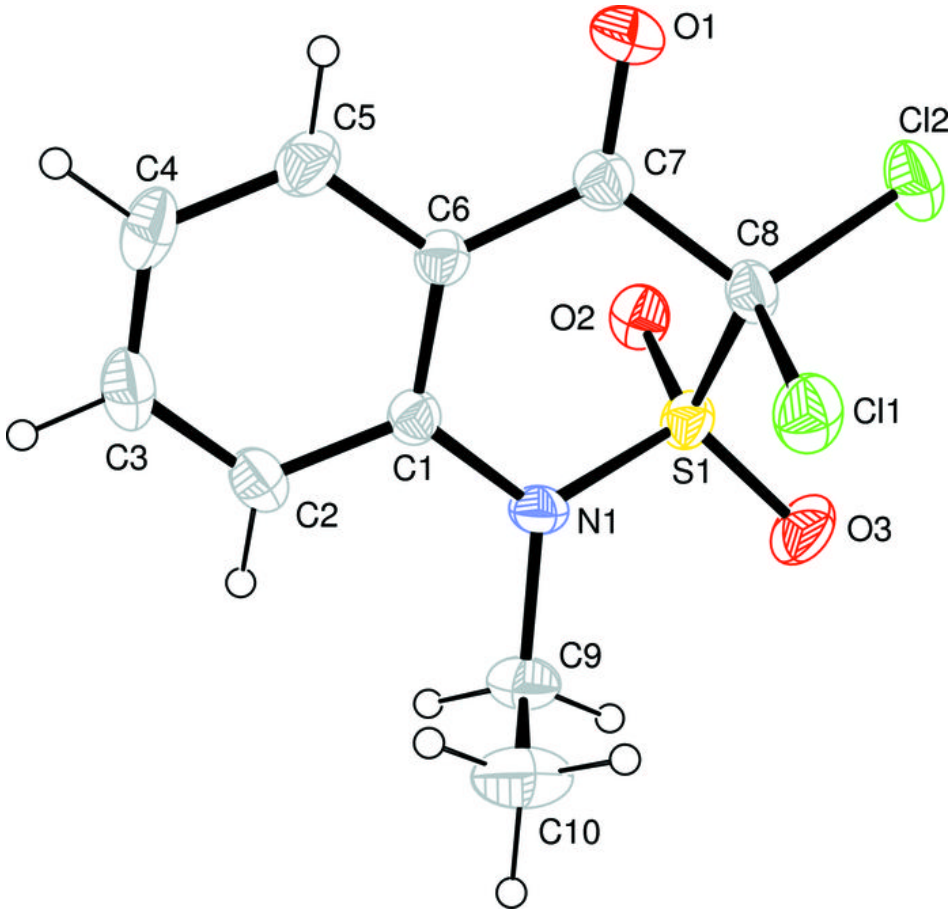

Fig. 2