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## Structure Reports

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# (2E)-2-(2,4-Dichlorophenylsulfonyl)-3-(4-methylanilino)-3-(methylsulfonyl)acrylonitrile

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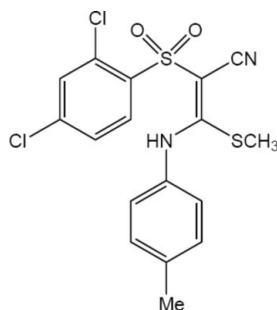
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 Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.117; data-to-parameter ratio = 19.9.

The title compound,  $\text{C}_{17}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_2\text{S}_2$ , and the 3-methoxyanilino analogue reported in the preceding paper have been used as starting materials to develop benzothiazine derivatives with antimalarial activity. The molecule displays an *E* (*trans*) configuration about the central double bond. Due to conjugation in the  $\text{C}=\text{C}-\text{C}\equiv\text{N}$  group, the putative single bond shows a significant shortening [1.418 (3) Å]. The molecule has a six-membered ring involving an intramolecular  $\text{N}-\text{H}\cdots\text{O}(\text{sulfonyl})$  bond, which is an example of resonance-assisted hydrogen bonding. In the crystal structure, bonds of the  $\text{C}-\text{H}\cdots\text{O}(\text{sulfonyl})$  and  $\text{C}-\text{H}\cdots\text{N}(\text{cyano})$  types form double layers of molecules parallel to (101). Within these layers there are  $\pi-\pi$  interactions between benzene rings of pairs of centrosymmetrically related molecules, with distances of 3.7969 (12) Å between centroids.  $\text{C}-\text{H}\cdots\pi$  interactions are also present.

## Related literature

For related literature, see: Allen (2002); Allen *et al.* (1987); Barazarte *et al.* (2008); Capparelli *et al.* (2008); Charris *et al.* (2005, 2007); Gilli *et al.* (1989); Hirshfeld (1976); Kennard *et al.* (2003); Krivokolysko *et al.* (2002); Song *et al.* (2005); Tominaga *et al.* (1989, 2002).



## Experimental

## Crystal data

 $\text{C}_{17}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_2\text{S}_2$   
 $M_r = 413.32$   
 Monoclinic,  $P2_1/n$   
 $a = 11.3975$  (6) Å  
 $b = 14.8147$  (8) Å  
 $c = 11.7215$  (6) Å  
 $\beta = 109.444$  (1)°

 $V = 1866.30$  (17) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.58$  mm<sup>-1</sup>  
 $T = 296$  (2) K  
 $0.55 \times 0.36 \times 0.27$  mm

## Data collection

 Bruker SMART APEX  
 diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2001)  
 $T_{\min} = 0.765$ ,  $T_{\max} = 0.851$ 

 12685 measured reflections  
 4562 independent reflections  
 3674 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.015$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.117$   
 $S = 1.03$   
 4562 reflections  
 229 parameters

 2 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.30$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O2}$	0.86	2.20	2.756 (2)	123
$\text{C4}-\text{H4A}\cdots\text{N1}^i$	0.96	2.61	3.504 (3)	156
$\text{C5}-\text{H5C}\cdots\text{O2}^{ii}$	0.96	2.50	3.442 (3)	167
$\text{C4}-\text{H4B}\cdots\text{Cg2}$	0.96	2.76	3.582 (3)	144

Symmetry codes: (i)  $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$ ; (ii)  $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$ . Cg2 is the centroid of the C21-C26 ring.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2185).

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**supplementary materials**

*Acta Cryst.* (2008). E64, o983-o984 [ doi:10.1107/S1600536808012531 ]

## (2E)-2-(2,4-Dichlorophenylsulfonyl)-3-(4-methylanilino)-3-(methylsulfanyl)acrylonitrile

M. V. Capparelli, A. R. Barazarte and J. E. Charris

### Comment

The exploration of simple molecules with different functionalities for the synthesis of heterocycles is a worthwhile contribution to the chemistry of these compounds. The title compound (II), and the 3"-methoxy analogue (I) [see previous paper: Capparelli *et al.*, 2008], and similar derivatives, have been used as effective synthons in the syntheses of some 1H-pyrrole-2,5-diones (Tominaga *et al.*, 2002), pyrimidine derivatives (Tominaga *et al.*, 1989) and 4H-1,4-benzothiazine-1,1-dioxides (Charris *et al.*, 2005). We used (I) and (II) as starting materials to develop benzothiazine derivatives with antimalarial activity (Charris *et al.*, 2007; Barazarte *et al.*, 2008).

The X-ray structure determination showed that there is one molecule per asymmetric unit (Fig. 1), which displays E (*trans*) configuration about the C2=C3 double bond. A search of the Cambridge Structural Database (version 5.29, updated Jan 2008) (Allen, 2002) produced no structures with the same central fragment (*i.e.* excluding the phenyl rings) of (II) for proper comparison, but a search for the more restricted fragment  $X-C(CN)=C(SMe)-N(H)-Y$  gave three comparable structures, *viz.* TAKDOZ (Krivokolysko *et al.*, 2002), AJULUM (Kennard *et al.*, 2003) and DALVES (Song *et al.*, 2005). Due to conjugation in the C3=C2—C1≡N1 moiety, the putative single bond C2—C1 (1.418 (3) Å) shows a significant shortening, similar to the range 1.415 (7)–1.437 (4) Å observed in the aforementioned structures. Bond lengths (see Supplementary Materials) are in good agreement with the expected values (Allen *et al.*, 1987). Within experimental error (*i.e.* 3 e.s.d.'s) all of the corresponding bond angles and most of the bond lengths are equal in (I) and (II).

Aside from the  $\sigma$ -bonded (*i.e.* free rotating) phenyl and SMe groups, the molecules of (I) and (II) display a significant difference in the rigid moieties formed by the double bonds C2=C3 and their neighboring atoms (S1, C1, S2, N2). The planes defined by S1—C2—C1 and N2—C3—S2 make dihedral angles of 17.08 (17)° in (I) and 11.2 (3)° in (II), but they are twisted about C2=C3 in different directions (Fig. 2), probably due to packing forces.

As in (I), the title compound displays a six-membered ring involving an intramolecular N—H...O(sulfonyl) bond (Table 1), which is an example of resonance-assisted hydrogen bonding (RAHB) (Gilli *et al.*, 1989), as suggested by the ring bond lengths. Comparison with AJULUM and DALVES, which display similar rings [with N—H...O(carbonyl) and N—H...O(carboxyl) bonds respectively] and TAKDOZ, which does not have RAHB, reveal lengthenings of the C=C distances [AJULUM, 1.371 (2) Å; DALVES, 1.386 (7) Å; TAKDOZ, 1.345 (4) Å] and shortenings of the C—N bonds [AJULUM, 1.360 (2) Å; DALVES, 1.333 (6) Å; TAKDOZ, 1.404 (4) Å]. On the other hand, the bond length of S1=O2, involved in RAHB, (1.4345 (15) Å) is nearly identical to the S1=O1 (1.4343 (15) Å). The molecular geometry also allows for a possible C4—H4b...Cg2 intramolecular interaction (Cg2: see below).

In the crystal structure (Fig. 3) bonds of the C—H...O(sulfonyl) and C—H...N(cyano) type form double layers of molecules parallel to (-1 0 1). Within these layers there are  $\pi$ - $\pi$  interactions between phenyl rings of pairs of centrosymmetrically related molecules, with Cg1...Cg1(-x, 1 - y, -z), 3.7969 (12) Å (Cgm: centroid of ring Cm1—Cm6, m = 1, 2).

## Experimental

To a solution of 2,4-dichlorobenzenesulfonylacetonitrile (1.50 g, 6.1 mmol) and KOH (0.46 g, 8.1 mmol) in anhydrous dioxane (10 ml) at 0°C, was added dropwise the 4-methylphenylisothiocyanate (0.91 g, 6.1 mmol) dissolved in anhydrous dioxane (3 ml). The resulting solution was stirred for 4 h at room temperature and then was added iodomethane (0.87 g, 6.1 mmol) and the mixture was stirred by 5 h at room temperature. The solvent was removed under reduced pressure and the residue was dissolved in dichloromethane (20 ml), washed with water, brine, dried over anhydrous sodium sulfate, filtered and concentrated to dryness. The solid was purified by recrystallization from ethanol. Yield: 1.91 g (74%). Crystals suitable for X-ray analysis were obtained during the recrystallization.

The IR spectrum was determined on a Shimadzu model 470 spectrophotometer. IR data [KBr pellets,  $\nu$  ( $\text{cm}^{-1}$ ): 3264 (NH), 2192 (CN), 1341 ( $\text{SO}_2$ ), 1133 ( $\text{SO}_2$ ).

The  $^1\text{H}$  NMR spectrum was recorded using a Jeol Eclipse 270 MHz [ $\text{CDCl}_3/\text{TMS}$ ,  $\delta$  (p.p.m.), atomic numbering as in Fig. 1]: 2.20 (s, 3H, SCH3), 2.37 (s, 3H,  $\text{CH}_3$ ), 7.13 (d, 2H, H22, H26; J: 8.3 Hz), 7.21 (d, 2H, H23, H25; J: 8.3 Hz), 7.46 (dd, 1H, H15; J: 8.6, 1.9 Hz), 7.56 (d, 1H, H13; J: 1.9 Hz), 8.14 (d, 1H, H16; J: 8.6 Hz), 9.90 (brs, 1H, NH).

The elemental analysis was performed on a Perkin Elmer 2400 CHN analyzer; results (%) were within  $\pm 0.4$  of predicted values. Calculated for  $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_2\text{Cl}_2\text{S}_2$ : C, 49.40; H, 3.41; N, 6.78. Found: C, 49.35; H, 3.40; N, 6.83.

The melting point (uncorrected) was measured with a Fischer-Johns micro hot-stage apparatus: 182–184 °C.

## Refinement

Hydrogen atoms were placed in calculated positions using a riding atom model with fixed C—H distances [0.86 Å for N, 0.93 Å for  $\text{C}(sp^2)$ , 0.96 Å for  $\text{C}(sp^3)$ ] and  $U_{\text{iso}} = p U_{\text{eq}}(\text{parent atom})$  [ $p = 1.2$  for N and  $\text{C}(sp^2)$ , 1.5 for  $\text{C}(sp^3)$ ].

DELU restraints (Sheldrick, 2008) were applied to C1—C2 and S1—O2 bonds which (before restraints) had values of  $\Delta U/\sigma = 6.00$  Å and 5.13 Å, somewhat above the standard maximum (*i.e.*  $\Delta U/\sigma \leq 5$  Å, Spek, 1998) for the rigid-bond test (Hirshfeld, 1976).

## Figures

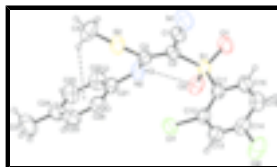


Fig. 1. Molecular structure of (II) showing the atomic numbering. Displacement ellipsoids are drawn at 50% probability level. Possible hydrogen bonds are shown as dashed lines.

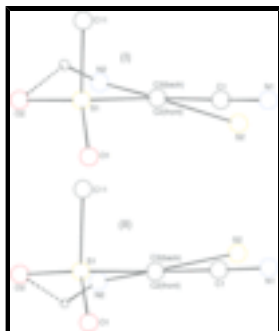


Fig. 2. View of the molecules of (I) and (II) along the C2=C3 bonds. Possible hydrogen bonds are shown as dashed lines. For clarity, the phenyl groups (except the C11 atoms) and the S-bonded Me groups were omitted.

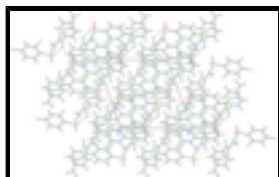


Fig. 3. Crystal structure of (II) viewed down the *b* axis. Possible hydrogen bonds are shown as dashed lines.

**(2E)-2-(2,4-Dichlorophenylsulfonyl)-3-(4-methylanilino)-3-(methylsulfonyl)acrylonitrile**

*Crystal data*

$C_{17}H_{14}Cl_2N_2O_2S_2$

$M_r = 413.32$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 11.3975\ (6)\ \text{\AA}$

$b = 14.8147\ (8)\ \text{\AA}$

$c = 11.7215\ (6)\ \text{\AA}$

$\beta = 109.444\ (1)^\circ$

$V = 1866.30\ (17)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 848$

$D_x = 1.471\ \text{Mg m}^{-3}$

Melting point = 455–457 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5367 reflections

$\theta = 2.3\text{--}28.5^\circ$

$\mu = 0.59\ \text{mm}^{-1}$

$T = 296\ (2)\ \text{K}$

Prism, colorless

$0.55 \times 0.36 \times 0.27\ \text{mm}$

*Data collection*

Bruker SMART APEX  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 8.13 pixels  $\text{mm}^{-1}$

$T = 296\ (2)\ \text{K}$

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2001)

$T_{\min} = 0.765$ ,  $T_{\max} = 0.851$

12685 measured reflections

4562 independent reflections

3674 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\text{max}} = 29.0^\circ$

$\theta_{\text{min}} = 2.2^\circ$

$h = -14 \rightarrow 12$

$k = -19 \rightarrow 19$

$l = -15 \rightarrow 14$

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.5083P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
4562 reflections	$(\Delta/\sigma)_{\max} = 0.001$
229 parameters	$\Delta\rho_{\max} = 0.38 \text{ e } \text{\AA}^{-3}$
2 restraints	$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

## 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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.36812 (4)	0.42470 (4)	0.06767 (4)	0.04761 (14)
S2	0.64407 (5)	0.37305 (4)	0.41840 (4)	0.05631 (15)
C11	0.30933 (5)	0.54484 (4)	0.27681 (5)	0.05801 (15)
C12	-0.14871 (5)	0.40783 (6)	0.14708 (6)	0.0824 (2)
O1	0.35547 (15)	0.36287 (12)	-0.02987 (13)	0.0681 (4)
O2	0.40074 (13)	0.51660 (10)	0.05390 (12)	0.0553 (3)
N1	0.4303 (2)	0.21530 (14)	0.23381 (18)	0.0737 (6)
N2	0.60865 (15)	0.51086 (11)	0.25871 (13)	0.0494 (4)
H2	0.5904	0.5266	0.1842	0.059*
C11	0.22335 (17)	0.42192 (12)	0.09442 (15)	0.0438 (4)
C12	0.19883 (17)	0.47436 (12)	0.18235 (16)	0.0445 (4)
C13	0.08383 (18)	0.47058 (14)	0.19803 (18)	0.0522 (4)
H13	0.0672	0.5062	0.2562	0.063*
C14	-0.00560 (18)	0.41325 (15)	0.12618 (18)	0.0542 (5)
C15	0.0156 (2)	0.36108 (15)	0.0379 (2)	0.0610 (5)
H15	-0.0462	0.3234	-0.0105	0.073*
C16	0.1306 (2)	0.36578 (14)	0.02256 (18)	0.0554 (5)

H16	0.1460	0.3308	-0.0368	0.066*
C21	0.67534 (18)	0.57496 (13)	0.34797 (16)	0.0466 (4)
C22	0.7764 (2)	0.61972 (15)	0.33448 (19)	0.0577 (5)
H22	0.7990	0.6098	0.2662	0.061 (6)*
C23	0.8437 (2)	0.67925 (16)	0.4231 (2)	0.0639 (6)
H23	0.9115	0.7092	0.4133	0.077*
C24	0.8129 (2)	0.69545 (14)	0.5260 (2)	0.0586 (5)
C25	0.7089 (2)	0.65227 (14)	0.53511 (19)	0.0583 (5)
H25	0.6848	0.6634	0.6022	0.070*
C26	0.6398 (2)	0.59308 (14)	0.44742 (19)	0.0534 (5)
H26	0.5695	0.5655	0.4553	0.064*
C1	0.45350 (19)	0.28900 (14)	0.22053 (17)	0.0524 (4)
C2	0.47563 (17)	0.38061 (13)	0.19901 (15)	0.0453 (4)
C3	0.57197 (17)	0.42866 (13)	0.28006 (15)	0.0438 (4)
C4	0.8069 (2)	0.3985 (2)	0.4575 (2)	0.0769 (7)
H4A	0.8543	0.3577	0.5193	0.115*
H4B	0.8219	0.4594	0.4869	0.115*
H4C	0.8316	0.3922	0.3872	0.115*
C5	0.8896 (3)	0.75847 (17)	0.6233 (3)	0.0873 (8)
H5A	0.9753	0.7403	0.6484	0.131*
H5B	0.8605	0.7563	0.6912	0.131*
H5C	0.8819	0.8189	0.5921	0.131*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0439 (2)	0.0669 (3)	0.0322 (2)	0.0030 (2)	0.01291 (17)	-0.00344 (19)
S2	0.0637 (3)	0.0598 (3)	0.0387 (2)	-0.0013 (2)	0.0081 (2)	0.0079 (2)
Cl1	0.0546 (3)	0.0655 (3)	0.0544 (3)	-0.0084 (2)	0.0188 (2)	-0.0189 (2)
Cl2	0.0437 (3)	0.1218 (6)	0.0828 (4)	-0.0037 (3)	0.0227 (3)	0.0103 (4)
O1	0.0607 (9)	0.1015 (12)	0.0413 (7)	0.0062 (8)	0.0161 (6)	-0.0211 (7)
O2	0.0475 (7)	0.0724 (9)	0.0458 (7)	-0.0004 (6)	0.0153 (6)	0.0141 (6)
N1	0.0953 (15)	0.0615 (12)	0.0611 (11)	-0.0123 (11)	0.0217 (11)	-0.0057 (9)
N2	0.0524 (9)	0.0587 (9)	0.0349 (7)	-0.0031 (7)	0.0116 (6)	0.0047 (6)
C11	0.0420 (9)	0.0517 (10)	0.0366 (8)	0.0000 (7)	0.0115 (7)	0.0002 (7)
C12	0.0428 (9)	0.0503 (10)	0.0379 (8)	-0.0005 (7)	0.0100 (7)	-0.0004 (7)
C13	0.0476 (10)	0.0653 (12)	0.0455 (10)	0.0058 (9)	0.0177 (8)	0.0005 (9)
C14	0.0409 (10)	0.0707 (13)	0.0496 (10)	0.0016 (9)	0.0132 (8)	0.0121 (9)
C15	0.0523 (12)	0.0683 (13)	0.0556 (12)	-0.0123 (10)	0.0088 (9)	-0.0044 (10)
C16	0.0544 (11)	0.0624 (12)	0.0465 (10)	-0.0041 (9)	0.0130 (9)	-0.0104 (9)
C21	0.0468 (10)	0.0504 (10)	0.0412 (9)	0.0003 (8)	0.0129 (7)	0.0037 (7)
C22	0.0588 (12)	0.0696 (13)	0.0495 (11)	-0.0061 (10)	0.0243 (9)	0.0057 (9)
C23	0.0618 (13)	0.0639 (13)	0.0672 (13)	-0.0165 (10)	0.0228 (11)	0.0040 (10)
C24	0.0699 (13)	0.0439 (10)	0.0570 (11)	-0.0023 (9)	0.0145 (10)	0.0035 (9)
C25	0.0749 (14)	0.0521 (11)	0.0532 (11)	0.0017 (10)	0.0282 (10)	-0.0035 (9)
C26	0.0534 (11)	0.0566 (11)	0.0554 (11)	-0.0028 (9)	0.0251 (9)	-0.0004 (9)
C1	0.0586 (12)	0.0573 (10)	0.0413 (9)	0.0000 (9)	0.0167 (8)	-0.0073 (8)
C2	0.0472 (10)	0.0520 (9)	0.0358 (8)	0.0051 (8)	0.0128 (7)	-0.0026 (7)

## supplementary materials

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C3	0.0445 (9)	0.0543 (10)	0.0334 (8)	0.0070 (8)	0.0139 (7)	0.0004 (7)
C4	0.0582 (14)	0.1022 (19)	0.0608 (14)	0.0158 (13)	0.0070 (11)	0.0216 (13)
C5	0.112 (2)	0.0604 (14)	0.0782 (17)	-0.0191 (14)	0.0166 (16)	-0.0122 (12)

### *Geometric parameters (Å, °)*

S1—O1	1.4343 (15)	C16—H16	0.9300
S1—O2	1.4345 (15)	C21—C26	1.382 (3)
S1—C2	1.7442 (18)	C21—C22	1.383 (3)
S1—C11	1.7796 (19)	C22—C23	1.384 (3)
S2—C3	1.7596 (18)	C22—H22	0.9300
S2—C4	1.798 (3)	C23—C24	1.385 (3)
C11—C12	1.7245 (18)	C23—H23	0.9300
C12—C14	1.730 (2)	C24—C25	1.382 (3)
N1—C1	1.146 (3)	C24—C5	1.508 (3)
N2—C3	1.338 (2)	C25—C26	1.381 (3)
N2—C21	1.430 (2)	C25—H25	0.9300
N2—H2	0.8600	C26—H26	0.9300
C11—C16	1.388 (3)	C1—C2	1.418 (3)
C11—C12	1.391 (3)	C2—C3	1.386 (3)
C12—C13	1.384 (3)	C4—H4A	0.9600
C13—C14	1.378 (3)	C4—H4B	0.9600
C13—H13	0.9300	C4—H4C	0.9600
C14—C15	1.375 (3)	C5—H5A	0.9600
C15—C16	1.383 (3)	C5—H5B	0.9600
C15—H15	0.9300	C5—H5C	0.9600
O1—S1—O2	118.52 (10)	C23—C22—H22	120.2
O1—S1—C2	108.61 (9)	C22—C23—C24	121.7 (2)
O2—S1—C2	108.81 (9)	C22—C23—H23	119.1
O1—S1—C11	105.82 (9)	C24—C23—H23	119.1
O2—S1—C11	109.39 (9)	C25—C24—C23	117.4 (2)
C2—S1—C11	104.83 (9)	C25—C24—C5	121.6 (2)
C3—S2—C4	105.22 (10)	C23—C24—C5	121.0 (2)
C3—N2—C21	126.20 (15)	C26—C25—C24	121.8 (2)
C3—N2—H2	116.9	C26—C25—H25	119.1
C21—N2—H2	116.9	C24—C25—H25	119.1
C16—C11—C12	118.95 (17)	C25—C26—C21	119.76 (19)
C16—C11—S1	118.07 (14)	C25—C26—H26	120.1
C12—C11—S1	122.97 (14)	C21—C26—H26	120.1
C13—C12—C11	120.56 (17)	N1—C1—C2	176.8 (2)
C13—C12—C11	117.44 (14)	C3—C2—C1	121.12 (17)
C11—C12—C11	122.00 (14)	C3—C2—S1	125.19 (14)
C14—C13—C12	118.97 (18)	C1—C2—S1	113.64 (14)
C14—C13—H13	120.5	N2—C3—C2	124.40 (16)
C12—C13—H13	120.5	N2—C3—S2	121.29 (14)
C15—C14—C13	121.78 (19)	C2—C3—S2	114.31 (14)
C15—C14—C12	119.36 (17)	S2—C4—H4A	109.5
C13—C14—C12	118.85 (16)	S2—C4—H4B	109.5
C14—C15—C16	118.76 (19)	H4A—C4—H4B	109.5

C14—C15—H15	120.6	S2—C4—H4C	109.5
C16—C15—H15	120.6	H4A—C4—H4C	109.5
C15—C16—C11	120.96 (19)	H4B—C4—H4C	109.5
C15—C16—H16	119.5	C24—C5—H5A	109.5
C11—C16—H16	119.5	C24—C5—H5B	109.5
C26—C21—C22	119.52 (19)	H5A—C5—H5B	109.5
C26—C21—N2	120.82 (17)	C24—C5—H5C	109.5
C22—C21—N2	119.66 (17)	H5A—C5—H5C	109.5
C21—C22—C23	119.66 (19)	H5B—C5—H5C	109.5
C21—C22—H22	120.2		
O1—S1—C11—C16	-0.27 (19)	C21—C22—C23—C24	0.1 (4)
O2—S1—C11—C16	128.48 (16)	C22—C23—C24—C25	-2.3 (3)
C2—S1—C11—C16	-114.99 (16)	C22—C23—C24—C5	178.2 (2)
O1—S1—C11—C12	-179.20 (16)	C23—C24—C25—C26	1.9 (3)
O2—S1—C11—C12	-50.45 (17)	C5—C24—C25—C26	-178.7 (2)
C2—S1—C11—C12	66.08 (17)	C24—C25—C26—C21	0.8 (3)
C16—C11—C12—C13	0.1 (3)	C22—C21—C26—C25	-3.1 (3)
S1—C11—C12—C13	178.98 (15)	N2—C21—C26—C25	177.15 (18)
C16—C11—C12—C11	179.16 (15)	O1—S1—C2—C3	133.90 (16)
S1—C11—C12—C11	-1.9 (2)	O2—S1—C2—C3	3.58 (19)
C11—C12—C13—C14	0.8 (3)	C11—S1—C2—C3	-113.35 (17)
C11—C12—C13—C14	-178.37 (15)	O1—S1—C2—C1	-48.61 (17)
C12—C13—C14—C15	-1.3 (3)	O2—S1—C2—C1	-178.93 (14)
C12—C13—C14—C12	179.34 (15)	C11—S1—C2—C1	64.14 (15)
C13—C14—C15—C16	0.9 (3)	C21—N2—C3—C2	157.68 (18)
C12—C14—C15—C16	-179.72 (17)	C21—N2—C3—S2	-21.5 (3)
C14—C15—C16—C11	0.0 (3)	C1—C2—C3—N2	170.85 (18)
C12—C11—C16—C15	-0.4 (3)	S1—C2—C3—N2	-11.8 (3)
S1—C11—C16—C15	-179.42 (17)	C1—C2—C3—S2	-9.9 (2)
C3—N2—C21—C26	-46.0 (3)	S1—C2—C3—S2	167.40 (11)
C3—N2—C21—C22	134.3 (2)	C4—S2—C3—N2	-39.06 (19)
C26—C21—C22—C23	2.6 (3)	C4—S2—C3—C2	141.68 (16)
N2—C21—C22—C23	-177.62 (19)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 $\cdots$ O2	0.86	2.20	2.756 (2)	123
C4—H4A $\cdots$ N1 <sup>i</sup>	0.96	2.61	3.504 (3)	156
C5—H5C $\cdots$ O2 <sup>ii</sup>	0.96	2.50	3.442 (3)	167
C16—H16 $\cdots$ O1	0.93	2.41	2.828 (3)	107
C4—H4B $\cdots$ Cg2	0.96	2.76	3.582 (3)	144

Symmetry codes: (i)  $x+1/2, -y+1/2, z+1/2$ ; (ii)  $x+1/2, -y+3/2, z+1/2$ .

Fig. 1

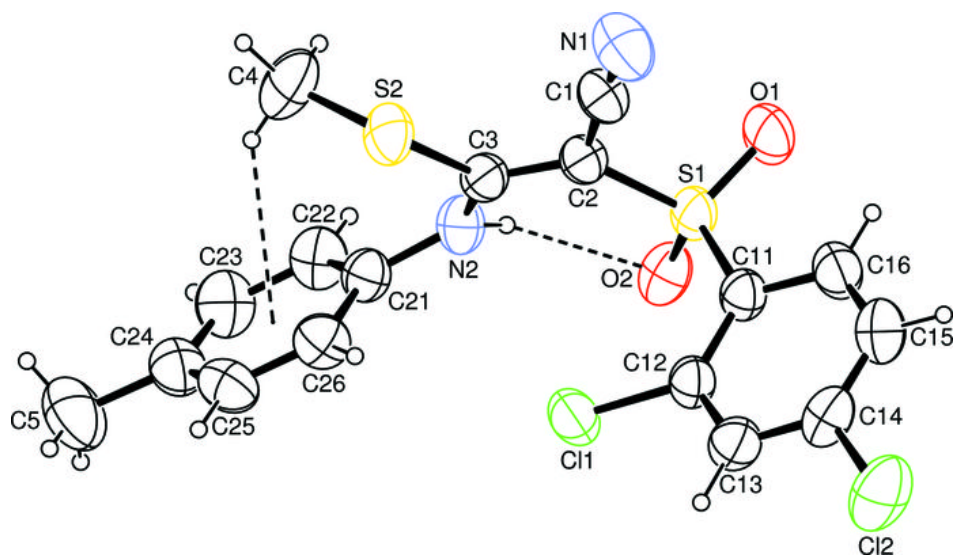


Fig. 2

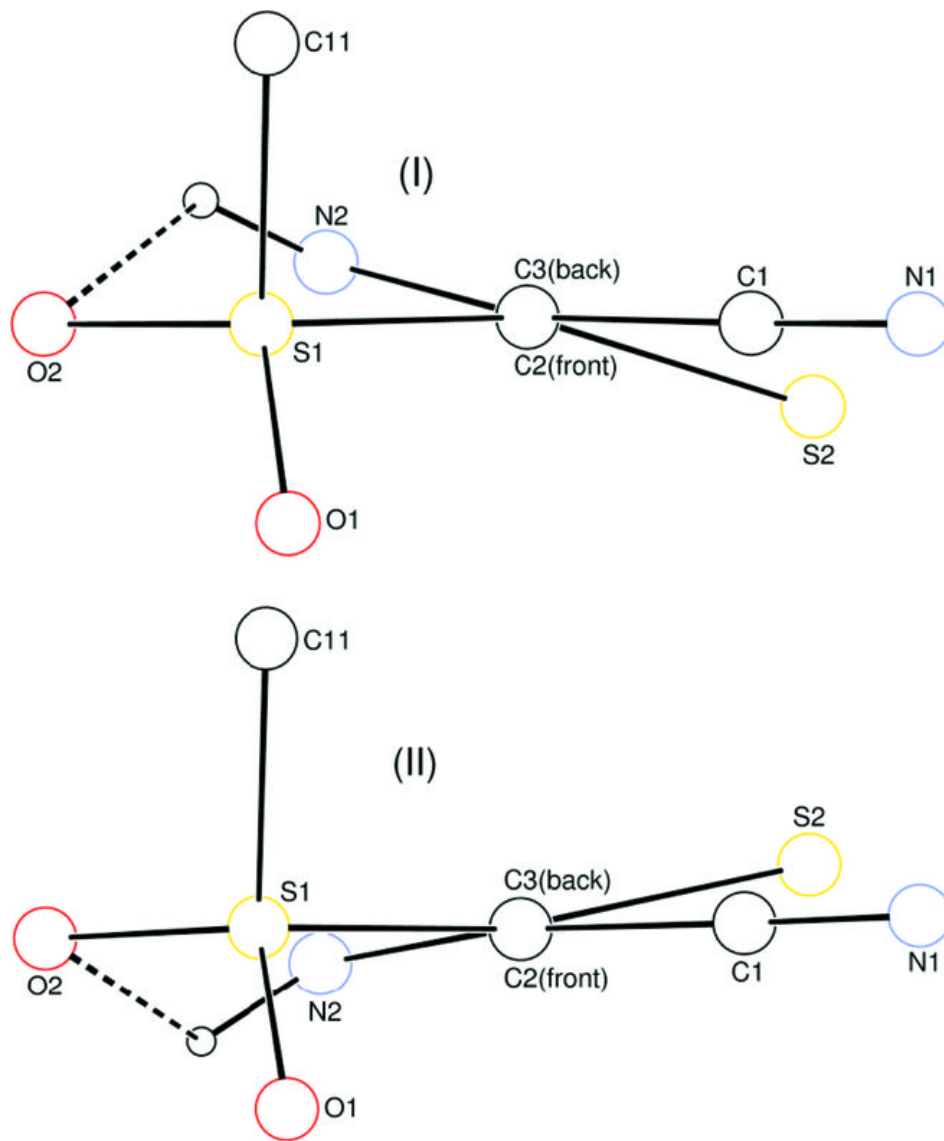


Fig. 3

