

N-(4-Chlorobenzoyl)-4-methylbenzenesulfonamide

P. A. Suchetan,^a B. Thimme Gowda,^{a*} Sabine Foro^b and Hartmut Fuess^b

^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

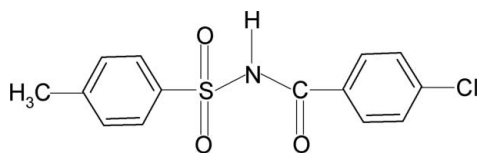
Received 23 December 2009; accepted 28 December 2009

Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.045; wR factor = 0.130; data-to-parameter ratio = 16.2.

The asymmetric unit of the title compound, $\text{C}_{14}\text{H}_{12}\text{ClNO}_3\text{S}$, contains two independent molecules. The dihedral angles between the two aromatic rings in each molecule are 81.0 (1) and 76.3 (1)°. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For background literature and similar structures, see: Gowda *et al.* (2009*a,b*); Suchetan *et al.* (2009).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{ClNO}_3\text{S}$

$M_r = 309.76$

Monoclinic, $C2/c$

$a = 25.675$ (3) Å

$b = 12.0508$ (8) Å

$c = 22.191$ (3) Å

$\beta = 122.16$ (1)°

$V = 5812.5$ (11) Å³

$Z = 16$

Mo $K\alpha$ radiation

$\mu = 0.41$ mm⁻¹

$T = 299$ K

$0.50 \times 0.48 \times 0.44$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)

$T_{\min} = 0.821$, $T_{\max} = 0.840$

12864 measured reflections

5931 independent reflections

3733 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.130$

$S = 1.07$

5931 reflections

367 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.22$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.35$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1N}\cdots\text{O5}^{\text{i}}$ | 0.84 (1) | 2.35 (1) | 3.133 (3) | 156 (2) |
| $\text{N2}-\text{H2N}\cdots\text{O2}^{\text{ii}}$ | 0.87 (1) | 2.03 (1) | 2.890 (3) | 170 (2) |

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

PAS thanks the Council of Scientific and Industrial Research (CSIR), Government of India, New Delhi, for the award of a research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5156).

References

- Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2009*a*). *Acta Cryst.* **E65**, o2516.
 Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2009*b*). *Acta Cryst.* **E65**, o2750.
 Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2009). *Acta Cryst.* **E65**, o3156.

supplementary materials

Acta Cryst. (2010). E66, o327 [doi:10.1107/S1600536809055585]

N-(4-Chlorobenzoyl)-4-methylbenzenesulfonamide

P. A. Suchetan, B. T. Gowda, S. Foro and H. Fuess

Comment

Diaryl acylsulfonamides are known as potent antitumor agents against a broad spectrum of human tumor xenografts in nude mice. As part of a study of the effect of ring and the side chain substituents on the crystal structures of *N*-aromatic sulfonamides (Gowda *et al.*, 2009*a,b*; Suchetan *et al.*, 2009), in the present work, the structure of *N*-(4-chlorobenzoyl)4-methylbenzenesulfonamide (I) has been determined (Fig.1). The conformations of the N—H bonds in the C—SO₂—NH—C(O) segments of the structure are *anti* to the C=O bonds, similar to that observed in *N*-(benzoyl)benzenesulfonamide (II) (Gowda *et al.*, 2009*a*) and *N*-(4-chlorobenzoyl) benzenesulfonamide (III)(Suchetan *et al.*, 2009).

The molecules are twisted at the *S* atom with the torsional angles of 67.1 (2)° and 67.7 (2)°, in the two molecules. The dihedral angles between the sulfonyl benzene ring and the —SO₂—NH—C—O segment are 83.6 (1)° and 81.0 (1)°, compared to the values of 86.5(0.1) in (II) and 75.7 (1)° in (III). Furthermore, the dihedral angle between the sulfonyl and the benzoyl benzene rings in (I) are 81.0 (1)° and 76.3 (1)°, compared to the values of 80.3(0.1) in (II) and 68.6 (1)° in (III).

The dihedral angle between the sulfonyl benzene rings of the two molecules in the asymmetric unit is 45.8 (1)°. The packing of molecules linked by of N—H···O(S) hydrogen bonds (Table 1) is shown in Fig. 2.

Experimental

N-(4-Chlorobenzoyl)4-methylbenzenesulfonamide was prepared by heating a mixture of 4-methylbenzenesulfonamide and 4-chlorobenzoyl chloride at 60° C for one hour. The reaction mixture was cooled and poured into ice cold water. The resulting solid was separated, washed thoroughly with water and dissolved in sodium hydrogen carbonate solution. The compound was precipitated by acidifying the filtered solution with dil. HCl. It was filtered and dried. The purity of the compound was checked by recording its melting point (168–170° C). Single crystals were obtained from slow evaporation of a solution of the compound in toluene. Prism like colourless single crystals of the title compound were obtained from a slow evaporation of its toluene solution at room temperature and the X-ray diffraction studies were also carried out at room temperature.

Refinement

The H atoms of the NH groups were located in a difference map and later restrained to N—H = 0.86 (1) %Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

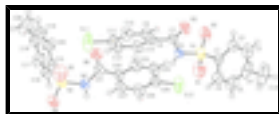


Fig. 1. Molecular structure of (I), showing the atom labelling scheme and the displacement ellipsoids are drawn at the 50% probability level.

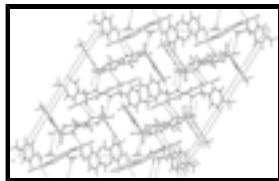


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

***N*-(4-Chlorobenzoyl)-4-methylbenzenesulfonamide**

Crystal data

| | |
|---------------------------------|---|
| $C_{14}H_{12}ClNO_3S$ | $F(000) = 2560$ |
| $M_r = 309.76$ | $D_x = 1.416 \text{ Mg m}^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: $-C 2yc$ | Cell parameters from 3720 reflections |
| $a = 25.675 (3) \text{ \AA}$ | $\theta = 2.4\text{--}27.8^\circ$ |
| $b = 12.0508 (8) \text{ \AA}$ | $\mu = 0.41 \text{ mm}^{-1}$ |
| $c = 22.191 (3) \text{ \AA}$ | $T = 299 \text{ K}$ |
| $\beta = 122.16 (1)^\circ$ | Prism, colourless |
| $V = 5812.5 (11) \text{ \AA}^3$ | $0.50 \times 0.48 \times 0.44 \text{ mm}$ |
| $Z = 16$ | |

Data collection

| | |
|---|--|
| Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector | 5931 independent reflections |
| Radiation source: fine-focus sealed tube graphite | 3733 reflections with $I > 2\sigma(I)$ |
| Rotation method data acquisition using ω and ϕ scans | $R_{\text{int}} = 0.018$ |
| Absorption correction: multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2009) | $\theta_{\text{max}} = 26.4^\circ$, $\theta_{\text{min}} = 2.5^\circ$ |
| $T_{\text{min}} = 0.821$, $T_{\text{max}} = 0.840$ | $h = -25 \rightarrow 32$ |
| 12864 measured reflections | $k = -10 \rightarrow 15$ |
| | $l = -27 \rightarrow 26$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.045$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.130$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.07$ | $w = 1/[\sigma^2(F_o^2) + (0.0686P)^2 + 0.2769P]$ |
| 5931 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 367 parameters | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 2 restraints | $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$ |
| | $\Delta\rho_{\text{min}} = -0.35 \text{ e \AA}^{-3}$ |

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|---------------|--------------|----------------------------------|
| Cl1 | 0.38789 (4) | 0.05434 (7) | 0.38377 (5) | 0.0983 (3) |
| S1 | 0.14332 (3) | -0.10122 (5) | 0.50954 (3) | 0.05366 (19) |
| O1 | 0.11216 (9) | -0.19893 (15) | 0.47163 (12) | 0.0881 (6) |
| O2 | 0.18104 (8) | -0.10514 (17) | 0.58521 (9) | 0.0788 (6) |
| O3 | 0.22829 (9) | 0.08559 (16) | 0.54283 (10) | 0.0806 (6) |
| N1 | 0.18659 (8) | -0.06886 (16) | 0.47816 (9) | 0.0452 (5) |
| H1N | 0.1773 (10) | -0.1095 (16) | 0.4431 (9) | 0.054* |
| C1 | 0.09034 (10) | 0.00583 (18) | 0.48616 (12) | 0.0442 (5) |
| C2 | 0.09089 (11) | 0.0707 (2) | 0.53769 (14) | 0.0585 (7) |
| H2 | 0.1209 | 0.0605 | 0.5855 | 0.070* |
| C3 | 0.04631 (14) | 0.1506 (2) | 0.51720 (18) | 0.0740 (8) |
| H3 | 0.0462 | 0.1941 | 0.5518 | 0.089* |
| C4 | 0.00175 (14) | 0.1678 (3) | 0.4465 (2) | 0.0793 (9) |
| C5 | 0.00290 (14) | 0.1015 (3) | 0.39662 (17) | 0.0886 (10) |
| H5 | -0.0269 | 0.1118 | 0.3487 | 0.106* |
| C6 | 0.04580 (12) | 0.0223 (2) | 0.41515 (14) | 0.0691 (7) |
| H6 | 0.0455 | -0.0211 | 0.3803 | 0.083* |
| C7 | 0.22739 (10) | 0.01850 (19) | 0.50176 (12) | 0.0477 (6) |
| C8 | 0.26807 (10) | 0.02355 (18) | 0.47333 (11) | 0.0417 (5) |
| C9 | 0.28899 (10) | 0.12670 (19) | 0.46694 (13) | 0.0532 (6) |
| H9 | 0.2778 | 0.1901 | 0.4812 | 0.064* |
| C10 | 0.32578 (11) | 0.1362 (2) | 0.44001 (13) | 0.0566 (7) |
| H10 | 0.3397 | 0.2055 | 0.4361 | 0.068* |
| C11 | 0.34187 (11) | 0.0432 (2) | 0.41897 (13) | 0.0547 (6) |
| C12 | 0.32329 (11) | -0.0603 (2) | 0.42626 (13) | 0.0580 (7) |
| H12 | 0.3360 | -0.1233 | 0.4133 | 0.070* |
| C13 | 0.28596 (10) | -0.06994 (19) | 0.45281 (12) | 0.0494 (6) |
| H13 | 0.2726 | -0.1396 | 0.4570 | 0.059* |
| C14 | -0.04607 (16) | 0.2566 (3) | 0.4263 (2) | 0.1319 (16) |
| H14A | -0.0263 | 0.3246 | 0.4500 | 0.158* |

supplementary materials

| | | | | |
|------|--------------|--------------|--------------|-------------|
| H14B | -0.0740 | 0.2341 | 0.4402 | 0.158* |
| H14C | -0.0684 | 0.2679 | 0.3757 | 0.158* |
| Cl2 | 0.10332 (4) | 0.14098 (7) | 0.31950 (5) | 0.0936 (3) |
| S2 | 0.33983 (3) | 0.23886 (6) | 0.17178 (3) | 0.0584 (2) |
| O4 | 0.31253 (8) | 0.18435 (17) | 0.10439 (8) | 0.0764 (6) |
| O5 | 0.35779 (10) | 0.35126 (16) | 0.17631 (11) | 0.0853 (6) |
| O6 | 0.33220 (8) | 0.33394 (15) | 0.29154 (9) | 0.0688 (5) |
| N2 | 0.28714 (9) | 0.22737 (16) | 0.19241 (10) | 0.0510 (5) |
| H2N | 0.2582 (8) | 0.1838 (16) | 0.1619 (10) | 0.061* |
| C15 | 0.40193 (10) | 0.1613 (2) | 0.23652 (12) | 0.0501 (6) |
| C16 | 0.39912 (11) | 0.0479 (2) | 0.23028 (13) | 0.0613 (7) |
| H16 | 0.3644 | 0.0142 | 0.1926 | 0.074* |
| C17 | 0.44735 (13) | -0.0156 (3) | 0.27945 (16) | 0.0755 (8) |
| H17 | 0.4450 | -0.0924 | 0.2745 | 0.091* |
| C18 | 0.49838 (13) | 0.0307 (4) | 0.33503 (16) | 0.0806 (9) |
| C19 | 0.50120 (13) | 0.1440 (4) | 0.34170 (15) | 0.0901 (11) |
| H19 | 0.5361 | 0.1766 | 0.3797 | 0.108* |
| C20 | 0.45307 (13) | 0.2109 (3) | 0.29301 (14) | 0.0734 (8) |
| H20 | 0.4552 | 0.2876 | 0.2984 | 0.088* |
| C21 | 0.29100 (11) | 0.27162 (19) | 0.25229 (12) | 0.0486 (6) |
| C22 | 0.24186 (10) | 0.23757 (18) | 0.26438 (11) | 0.0433 (5) |
| C23 | 0.22642 (11) | 0.3098 (2) | 0.30130 (12) | 0.0533 (6) |
| H23 | 0.2454 | 0.3788 | 0.3154 | 0.064* |
| C24 | 0.18339 (12) | 0.2803 (2) | 0.31707 (13) | 0.0585 (7) |
| H24 | 0.1725 | 0.3296 | 0.3408 | 0.070* |
| C25 | 0.15648 (11) | 0.1773 (2) | 0.29750 (13) | 0.0555 (6) |
| C26 | 0.17150 (12) | 0.1034 (2) | 0.26192 (13) | 0.0576 (6) |
| H26 | 0.1531 | 0.0338 | 0.2490 | 0.069* |
| C27 | 0.21412 (11) | 0.13404 (19) | 0.24571 (12) | 0.0517 (6) |
| H27 | 0.2246 | 0.0844 | 0.2218 | 0.062* |
| C28 | 0.55230 (14) | -0.0392 (4) | 0.38905 (19) | 0.1392 (17) |
| H28A | 0.5748 | -0.0653 | 0.3685 | 0.167* |
| H28B | 0.5374 | -0.1014 | 0.4026 | 0.167* |
| H28C | 0.5789 | 0.0050 | 0.4303 | 0.167* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| Cl1 | 0.1146 (7) | 0.0990 (6) | 0.1408 (8) | 0.0175 (5) | 0.1081 (6) | 0.0329 (5) |
| S1 | 0.0629 (4) | 0.0522 (4) | 0.0623 (4) | 0.0014 (3) | 0.0443 (3) | 0.0062 (3) |
| O1 | 0.1045 (15) | 0.0493 (11) | 0.1475 (19) | -0.0261 (11) | 0.0920 (15) | -0.0236 (12) |
| O2 | 0.0802 (12) | 0.1132 (16) | 0.0579 (12) | 0.0377 (12) | 0.0467 (10) | 0.0363 (11) |
| O3 | 0.0916 (14) | 0.0817 (14) | 0.0988 (15) | -0.0321 (11) | 0.0711 (13) | -0.0506 (12) |
| N1 | 0.0521 (11) | 0.0500 (12) | 0.0439 (11) | -0.0115 (9) | 0.0326 (10) | -0.0143 (9) |
| C1 | 0.0444 (12) | 0.0488 (14) | 0.0459 (14) | -0.0055 (10) | 0.0283 (11) | 0.0032 (10) |
| C2 | 0.0564 (15) | 0.0642 (17) | 0.0577 (16) | -0.0022 (13) | 0.0323 (13) | -0.0058 (13) |
| C3 | 0.080 (2) | 0.0623 (19) | 0.107 (3) | 0.0012 (16) | 0.069 (2) | -0.0054 (17) |
| C4 | 0.0615 (18) | 0.074 (2) | 0.124 (3) | 0.0162 (16) | 0.064 (2) | 0.042 (2) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C5 | 0.0615 (18) | 0.129 (3) | 0.074 (2) | 0.020 (2) | 0.0349 (17) | 0.041 (2) |
| C6 | 0.0667 (17) | 0.091 (2) | 0.0517 (17) | 0.0017 (16) | 0.0326 (14) | 0.0052 (15) |
| C7 | 0.0526 (14) | 0.0490 (14) | 0.0448 (14) | -0.0029 (12) | 0.0281 (11) | -0.0081 (11) |
| C8 | 0.0420 (12) | 0.0412 (13) | 0.0431 (13) | -0.0006 (10) | 0.0233 (10) | -0.0018 (10) |
| C9 | 0.0556 (14) | 0.0412 (14) | 0.0663 (16) | 0.0040 (11) | 0.0348 (13) | -0.0005 (11) |
| C10 | 0.0555 (15) | 0.0457 (15) | 0.0730 (18) | 0.0020 (12) | 0.0372 (14) | 0.0153 (12) |
| C11 | 0.0552 (14) | 0.0602 (17) | 0.0630 (16) | 0.0089 (13) | 0.0411 (13) | 0.0166 (13) |
| C12 | 0.0678 (16) | 0.0494 (15) | 0.0771 (18) | 0.0106 (13) | 0.0522 (15) | 0.0037 (13) |
| C13 | 0.0585 (14) | 0.0401 (14) | 0.0625 (16) | -0.0003 (11) | 0.0408 (13) | 0.0012 (11) |
| C14 | 0.101 (3) | 0.113 (3) | 0.227 (5) | 0.051 (2) | 0.118 (3) | 0.081 (3) |
| Cl2 | 0.0976 (6) | 0.1036 (7) | 0.1202 (7) | -0.0049 (5) | 0.0852 (6) | 0.0033 (5) |
| S2 | 0.0639 (4) | 0.0694 (5) | 0.0533 (4) | -0.0019 (3) | 0.0388 (3) | 0.0103 (3) |
| O4 | 0.0730 (12) | 0.1215 (16) | 0.0424 (10) | 0.0038 (12) | 0.0358 (9) | -0.0008 (10) |
| O5 | 0.1050 (15) | 0.0662 (13) | 0.1101 (17) | -0.0067 (11) | 0.0744 (14) | 0.0229 (11) |
| O6 | 0.0759 (12) | 0.0704 (12) | 0.0660 (12) | -0.0237 (10) | 0.0417 (10) | -0.0165 (10) |
| N2 | 0.0519 (12) | 0.0614 (14) | 0.0430 (11) | -0.0046 (10) | 0.0275 (10) | -0.0021 (9) |
| C15 | 0.0492 (14) | 0.0624 (17) | 0.0465 (14) | -0.0135 (12) | 0.0307 (12) | -0.0030 (12) |
| C16 | 0.0503 (15) | 0.0705 (19) | 0.0578 (17) | -0.0067 (14) | 0.0252 (13) | -0.0029 (14) |
| C17 | 0.0630 (18) | 0.082 (2) | 0.080 (2) | 0.0059 (16) | 0.0372 (18) | 0.0178 (17) |
| C18 | 0.0546 (18) | 0.127 (3) | 0.064 (2) | 0.000 (2) | 0.0343 (16) | 0.024 (2) |
| C19 | 0.0490 (17) | 0.157 (4) | 0.0515 (19) | -0.036 (2) | 0.0178 (15) | -0.010 (2) |
| C20 | 0.0704 (18) | 0.087 (2) | 0.0611 (18) | -0.0333 (17) | 0.0337 (16) | -0.0167 (16) |
| C21 | 0.0584 (15) | 0.0430 (14) | 0.0453 (14) | 0.0013 (12) | 0.0281 (12) | 0.0025 (11) |
| C22 | 0.0521 (13) | 0.0399 (13) | 0.0380 (12) | 0.0053 (11) | 0.0241 (11) | 0.0052 (10) |
| C23 | 0.0672 (16) | 0.0379 (13) | 0.0566 (15) | -0.0008 (12) | 0.0341 (13) | -0.0041 (11) |
| C24 | 0.0702 (17) | 0.0538 (16) | 0.0626 (17) | 0.0121 (14) | 0.0428 (14) | -0.0011 (13) |
| C25 | 0.0586 (15) | 0.0609 (17) | 0.0552 (15) | 0.0058 (13) | 0.0358 (13) | 0.0081 (13) |
| C26 | 0.0703 (16) | 0.0476 (15) | 0.0643 (16) | -0.0079 (13) | 0.0421 (14) | -0.0018 (13) |
| C27 | 0.0667 (16) | 0.0457 (14) | 0.0510 (15) | 0.0020 (12) | 0.0369 (13) | -0.0026 (11) |
| C28 | 0.069 (2) | 0.233 (5) | 0.096 (3) | 0.032 (3) | 0.030 (2) | 0.081 (3) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|---------|-------------|
| Cl1—C11 | 1.734 (2) | Cl2—C25 | 1.732 (2) |
| S1—O1 | 1.4205 (19) | S2—O5 | 1.4170 (19) |
| S1—O2 | 1.4242 (18) | S2—O4 | 1.4294 (18) |
| S1—N1 | 1.6417 (18) | S2—N2 | 1.6487 (19) |
| S1—C1 | 1.741 (2) | S2—C15 | 1.744 (2) |
| O3—C7 | 1.209 (2) | O6—C21 | 1.209 (3) |
| N1—C7 | 1.377 (3) | N2—C21 | 1.385 (3) |
| N1—H1N | 0.838 (9) | N2—H2N | 0.867 (9) |
| C1—C2 | 1.379 (3) | C15—C16 | 1.371 (3) |
| C1—C6 | 1.383 (3) | C15—C20 | 1.378 (3) |
| C2—C3 | 1.375 (3) | C16—C17 | 1.368 (4) |
| C2—H2 | 0.9300 | C16—H16 | 0.9300 |
| C3—C4 | 1.380 (4) | C17—C18 | 1.352 (4) |
| C3—H3 | 0.9300 | C17—H17 | 0.9300 |
| C4—C5 | 1.377 (5) | C18—C19 | 1.371 (4) |
| C4—C14 | 1.507 (4) | C18—C28 | 1.515 (4) |

supplementary materials

| | | | |
|-----------|-------------|-------------|-------------|
| C5—C6 | 1.345 (4) | C19—C20 | 1.387 (4) |
| C5—H5 | 0.9300 | C19—H19 | 0.9300 |
| C6—H6 | 0.9300 | C20—H20 | 0.9300 |
| C7—C8 | 1.481 (3) | C21—C22 | 1.481 (3) |
| C8—C13 | 1.382 (3) | C22—C27 | 1.386 (3) |
| C8—C9 | 1.391 (3) | C22—C23 | 1.390 (3) |
| C9—C10 | 1.365 (3) | C23—C24 | 1.372 (3) |
| C9—H9 | 0.9300 | C23—H23 | 0.9300 |
| C10—C11 | 1.361 (3) | C24—C25 | 1.374 (3) |
| C10—H10 | 0.9300 | C24—H24 | 0.9300 |
| C11—C12 | 1.375 (3) | C25—C26 | 1.374 (3) |
| C12—C13 | 1.371 (3) | C26—C27 | 1.372 (3) |
| C12—H12 | 0.9300 | C26—H26 | 0.9300 |
| C13—H13 | 0.9300 | C27—H27 | 0.9300 |
| C14—H14A | 0.9600 | C28—H28A | 0.9600 |
| C14—H14B | 0.9600 | C28—H28B | 0.9600 |
| C14—H14C | 0.9600 | C28—H28C | 0.9600 |
| O1—S1—O2 | 119.07 (13) | O5—S2—O4 | 118.25 (12) |
| O1—S1—N1 | 103.93 (10) | O5—S2—N2 | 110.16 (11) |
| O2—S1—N1 | 108.60 (10) | O4—S2—N2 | 103.53 (10) |
| O1—S1—C1 | 109.47 (12) | O5—S2—C15 | 109.49 (12) |
| O2—S1—C1 | 108.00 (11) | O4—S2—C15 | 109.86 (12) |
| N1—S1—C1 | 107.18 (10) | N2—S2—C15 | 104.58 (10) |
| C7—N1—S1 | 124.74 (15) | C21—N2—S2 | 125.70 (17) |
| C7—N1—H1N | 125.6 (16) | C21—N2—H2N | 125.2 (16) |
| S1—N1—H1N | 109.4 (16) | S2—N2—H2N | 108.9 (16) |
| C2—C1—C6 | 120.0 (2) | C16—C15—C20 | 119.8 (3) |
| C2—C1—S1 | 120.74 (19) | C16—C15—S2 | 118.36 (18) |
| C6—C1—S1 | 119.16 (19) | C20—C15—S2 | 121.8 (2) |
| C3—C2—C1 | 118.9 (3) | C17—C16—C15 | 120.0 (3) |
| C3—C2—H2 | 120.5 | C17—C16—H16 | 120.0 |
| C1—C2—H2 | 120.5 | C15—C16—H16 | 120.0 |
| C2—C3—C4 | 121.4 (3) | C18—C17—C16 | 121.5 (3) |
| C2—C3—H3 | 119.3 | C18—C17—H17 | 119.2 |
| C4—C3—H3 | 119.3 | C16—C17—H17 | 119.2 |
| C5—C4—C3 | 117.9 (3) | C17—C18—C19 | 118.6 (3) |
| C5—C4—C14 | 122.4 (4) | C17—C18—C28 | 121.8 (4) |
| C3—C4—C14 | 119.7 (4) | C19—C18—C28 | 119.6 (3) |
| C6—C5—C4 | 122.0 (3) | C18—C19—C20 | 121.4 (3) |
| C6—C5—H5 | 119.0 | C18—C19—H19 | 119.3 |
| C4—C5—H5 | 119.0 | C20—C19—H19 | 119.3 |
| C5—C6—C1 | 119.8 (3) | C15—C20—C19 | 118.6 (3) |
| C5—C6—H6 | 120.1 | C15—C20—H20 | 120.7 |
| C1—C6—H6 | 120.1 | C19—C20—H20 | 120.7 |
| O3—C7—N1 | 120.2 (2) | O6—C21—N2 | 121.2 (2) |
| O3—C7—C8 | 124.0 (2) | O6—C21—C22 | 123.0 (2) |
| N1—C7—C8 | 115.83 (18) | N2—C21—C22 | 115.7 (2) |
| C13—C8—C9 | 118.7 (2) | C27—C22—C23 | 118.5 (2) |
| C13—C8—C7 | 122.7 (2) | C27—C22—C21 | 123.2 (2) |

| | | | |
|---------------|--------------|-----------------|--------------|
| C9—C8—C7 | 118.6 (2) | C23—C22—C21 | 118.1 (2) |
| C10—C9—C8 | 121.0 (2) | C24—C23—C22 | 120.6 (2) |
| C10—C9—H9 | 119.5 | C24—C23—H23 | 119.7 |
| C8—C9—H9 | 119.5 | C22—C23—H23 | 119.7 |
| C11—C10—C9 | 119.2 (2) | C23—C24—C25 | 119.4 (2) |
| C11—C10—H10 | 120.4 | C23—C24—H24 | 120.3 |
| C9—C10—H10 | 120.4 | C25—C24—H24 | 120.3 |
| C10—C11—C12 | 121.3 (2) | C26—C25—C24 | 121.3 (2) |
| C10—C11—C11 | 119.64 (19) | C26—C25—C12 | 120.2 (2) |
| C12—C11—C11 | 119.06 (19) | C24—C25—C12 | 118.56 (19) |
| C13—C12—C11 | 119.5 (2) | C27—C26—C25 | 118.9 (2) |
| C13—C12—H12 | 120.2 | C27—C26—H26 | 120.5 |
| C11—C12—H12 | 120.2 | C25—C26—H26 | 120.5 |
| C12—C13—C8 | 120.2 (2) | C26—C27—C22 | 121.2 (2) |
| C12—C13—H13 | 119.9 | C26—C27—H27 | 119.4 |
| C8—C13—H13 | 119.9 | C22—C27—H27 | 119.4 |
| C4—C14—H14A | 109.5 | C18—C28—H28A | 109.5 |
| C4—C14—H14B | 109.5 | C18—C28—H28B | 109.5 |
| H14A—C14—H14B | 109.5 | H28A—C28—H28B | 109.5 |
| C4—C14—H14C | 109.5 | C18—C28—H28C | 109.5 |
| H14A—C14—H14C | 109.5 | H28A—C28—H28C | 109.5 |
| H14B—C14—H14C | 109.5 | H28B—C28—H28C | 109.5 |
| O1—S1—N1—C7 | -177.1 (2) | O5—S2—N2—C21 | -49.9 (2) |
| O2—S1—N1—C7 | -49.4 (2) | O4—S2—N2—C21 | -177.2 (2) |
| C1—S1—N1—C7 | 67.1 (2) | C15—S2—N2—C21 | 67.7 (2) |
| O1—S1—C1—C2 | 132.2 (2) | O5—S2—C15—C16 | -164.32 (18) |
| O2—S1—C1—C2 | 1.2 (2) | O4—S2—C15—C16 | -32.9 (2) |
| N1—S1—C1—C2 | -115.67 (19) | N2—S2—C15—C16 | 77.7 (2) |
| O1—S1—C1—C6 | -45.1 (2) | O5—S2—C15—C20 | 15.6 (2) |
| O2—S1—C1—C6 | -176.12 (19) | O4—S2—C15—C20 | 147.08 (19) |
| N1—S1—C1—C6 | 67.0 (2) | N2—S2—C15—C20 | -102.4 (2) |
| C6—C1—C2—C3 | 0.6 (4) | C20—C15—C16—C17 | -1.0 (4) |
| S1—C1—C2—C3 | -176.69 (19) | S2—C15—C16—C17 | 179.0 (2) |
| C1—C2—C3—C4 | -0.7 (4) | C15—C16—C17—C18 | 0.3 (4) |
| C2—C3—C4—C5 | 0.5 (4) | C16—C17—C18—C19 | 0.1 (4) |
| C2—C3—C4—C14 | -179.7 (2) | C16—C17—C18—C28 | -179.1 (3) |
| C3—C4—C5—C6 | -0.2 (4) | C17—C18—C19—C20 | 0.2 (5) |
| C14—C4—C5—C6 | 180.0 (3) | C28—C18—C19—C20 | 179.4 (3) |
| C4—C5—C6—C1 | 0.1 (4) | C16—C15—C20—C19 | 1.2 (4) |
| C2—C1—C6—C5 | -0.3 (4) | S2—C15—C20—C19 | -178.8 (2) |
| S1—C1—C6—C5 | 177.0 (2) | C18—C19—C20—C15 | -0.8 (4) |
| S1—N1—C7—O3 | -8.7 (3) | S2—N2—C21—O6 | 8.0 (3) |
| S1—N1—C7—C8 | 171.77 (16) | S2—N2—C21—C22 | -172.02 (15) |
| O3—C7—C8—C13 | 151.8 (3) | O6—C21—C22—C27 | -147.2 (2) |
| N1—C7—C8—C13 | -28.6 (3) | N2—C21—C22—C27 | 32.8 (3) |
| O3—C7—C8—C9 | -28.3 (4) | O6—C21—C22—C23 | 27.5 (3) |
| N1—C7—C8—C9 | 151.2 (2) | N2—C21—C22—C23 | -152.5 (2) |
| C13—C8—C9—C10 | 0.9 (3) | C27—C22—C23—C24 | -1.8 (3) |
| C7—C8—C9—C10 | -179.0 (2) | C21—C22—C23—C24 | -176.8 (2) |

supplementary materials

| | | | |
|-----------------|--------------|-----------------|--------------|
| C8—C9—C10—C11 | 0.2 (4) | C22—C23—C24—C25 | 1.5 (4) |
| C9—C10—C11—C12 | -1.9 (4) | C23—C24—C25—C26 | -0.5 (4) |
| C9—C10—C11—C11 | 179.30 (18) | C23—C24—C25—C12 | 178.81 (18) |
| C10—C11—C12—C13 | 2.4 (4) | C24—C25—C26—C27 | -0.2 (4) |
| C11—C11—C12—C13 | -178.81 (18) | C12—C25—C26—C27 | -179.45 (17) |
| C11—C12—C13—C8 | -1.2 (4) | C25—C26—C27—C22 | -0.2 (4) |
| C9—C8—C13—C12 | -0.4 (3) | C23—C22—C27—C26 | 1.2 (3) |
| C7—C8—C13—C12 | 179.4 (2) | C21—C22—C27—C26 | 175.9 (2) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|----------|-------------|-------------|---------------|
| N1—H1N \cdots O5 ⁱ | 0.84 (1) | 2.35 (1) | 3.133 (3) | 156 (2) |
| N2—H2N \cdots O2 ⁱⁱ | 0.87 (1) | 2.03 (1) | 2.890 (3) | 170 (2) |

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

Fig. 1

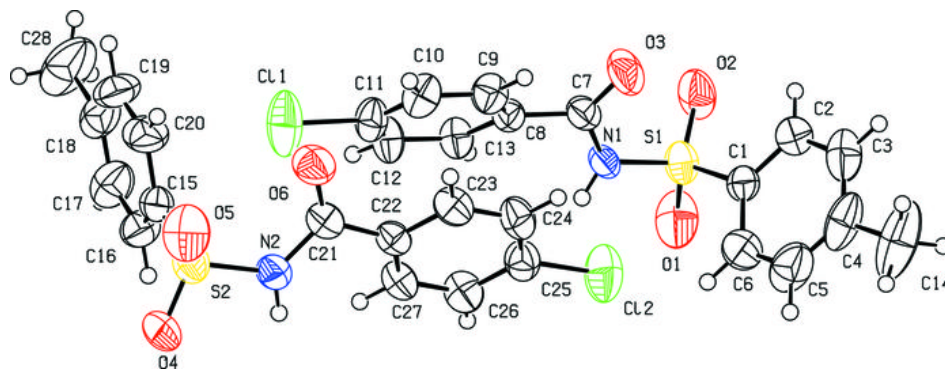


Fig. 2

