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2-Chloro-*N*-(3-chlorobenzoyl)benzenesulfonamideB. Thimme Gowda,^{a*} Sabine Foro,^b P. A. Suchetan^a 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

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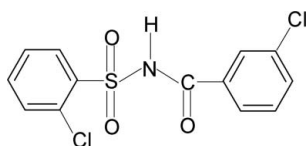
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.054; wR factor = 0.154; data-to-parameter ratio = 15.5.

The asymmetric unit of the title compound, $\text{C}_{13}\text{H}_9\text{Cl}_2\text{NO}_3\text{S}$, contains two independent molecules. The conformation of the $\text{C}=\text{O}$ bond is *anti* to the *meta*-Cl group in the chlorobenzoyl group of one of the molecules and *syn* in the other. The dihedral angles between the sulfonyl and benzoyl benzene rings are 77.8 (1) and 83.5 (1)°. In the crystal structure, two pairs of independent molecules are linked into a tetramer by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For background literature and related structures, see: Gowda *et al.* (2009, 2010); Suchetan *et al.* (2010*a,b*).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_9\text{Cl}_2\text{NO}_3\text{S}$ $M_r = 330.17$ Triclinic, $P\bar{1}$ $a = 7.4399$ (6) Å $b = 11.679$ (1) Å $c = 17.138$ (2) Å $\alpha = 75.346$ (7)° $\beta = 83.188$ (8)° $\gamma = 77.732$ (7)° $V = 1404.5$ (2) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.62$ mm⁻¹
 $T = 299$ K $0.20 \times 0.14 \times 0.08$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford)Diffraction, 2009)
 $T_{\min} = 0.887$, $T_{\max} = 0.952$
9768 measured reflections
5694 independent reflections
4267 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$ $wR(F^2) = 0.154$ $S = 1.04$

5694 reflections

367 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 1.10$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.50$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1N}\cdots\text{O6}$ | 0.84 (2) | 2.02 (2) | 2.836 (4) | 162 (4) |
| $\text{N2}-\text{H2N}\cdots\text{O5}^i$ | 0.85 (2) | 2.10 (2) | 2.937 (4) | 173 (4) |

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5091).

References

- Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2009). *Acta Cryst.* **E65**, o2516.
 Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2010). *Acta Cryst.* **E66**, o433.
 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. (2010*a*). *Acta Cryst.* **E66**, o1040.
 Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*b*). *Acta Cryst.* **E66**, o1292.

supplementary materials

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2-Chloro-*N*-(3-chlorobenzoyl)benzenesulfonamide

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Comment

As a part of studying the effect of ring and the side chain substitutions on the crystal structures of *N*-aryl sulfonamides (Gowda *et al.*, 2009, 2010; Suchetan *et al.*, 2010*a,b*), the structure of 2-chloro-*N*-(3-chlorobenzoyl)-benzenesulfonamide (I) has been determined. The asymmetric unit of (I) contains two independent molecules. In the C—SO₂—NH—C(O) segments, the N—H bonds are *anti* to the C=O bonds (Fig.1), similar to those observed in 2-chloro-*N*-(3-methylbenzoyl)-benzenesulfonamide (II) (Suchetan *et al.*, 2010*b*), 2-methyl-*N*-(3-methylbenzoyl)-benzenesulfonamide (III) (Gowda *et al.*, 2010), 2-chloro-*N*-(2-chlorobenzoyl)-benzenesulfonamide (IV) (Suchetan *et al.*, 2010*a*), *N*-(benzoyl)-benzenesulfonamide (V) (Gowda *et al.*, 2009).

The conformation of the C=O bond is *anti* to the *meta*-Cl group in the benzoyl ring of one of the molecules and *syn* in the other, compared to the *anti* conformation observed between C=O bond and *meta*-methyl group in the benzoyl ring of (II).

The chlorobenzoyl and sulfonyl-bound chlorophenyl units in the two molecules of (I) are twisted with respect to the S—N bond, with torsional angles of -62.6 (3)° [C7—N1—S1—C1] and -62.6 (3)° [C20—N2—S2—C14], compared to those of -66.5 (2)° in (II), -66.2 (3)° in (III), -66.5 (2)° in (IV) and -66.9 (3)° in (V). The dihedral angles between the sulfonyl and the benzoyl-bound benzene rings are 77.8 (1)° (molecule 1) and 83.5 (1)° (molecule 2), compared to the values of 74.7 (1)° in (II), 74.8 (1)° in (III), 76.9 (1)° in (IV) and 80.3 (1)° in (V).

Experimental

The title compound was prepared by refluxing a mixture of 3-chlorobenzoic acid, 2-chlorobenzenesulfonamide and phosphorous oxy chloride for 3 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid obtained was filtered, washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. It was filtered, dried and recrystallized. Rod like colourless single crystals of the title compound used in X-ray diffraction studies were obtained by a slow evaporation of its toluene solution at room temperature.

Refinement

The H atoms of the NH groups were located in a difference map and refined with the a N—H distance restraint of 0.86 (2) %A. 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). The residual electron-density features are located in the region of H25 and Cl4. The highest peak is 0.98 Å from H25 and the deepest hole is 0.68 Å from Cl4.

Figures

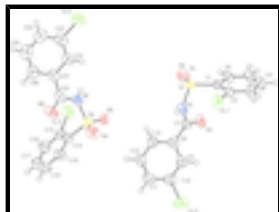


Fig. 1. The two independent molecules of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2-Chloro-*N*-(3-chlorobenzoyl)benzenesulfonamide

Crystal data

| | |
|--------------------------------|---|
| $C_{13}H_9Cl_2NO_3S$ | $Z = 4$ |
| $M_r = 330.17$ | $F(000) = 672$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.561 \text{ Mg m}^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 7.4399 (6) \text{ \AA}$ | Cell parameters from 3572 reflections |
| $b = 11.679 (1) \text{ \AA}$ | $\theta = 2.5\text{--}27.8^\circ$ |
| $c = 17.138 (2) \text{ \AA}$ | $\mu = 0.62 \text{ mm}^{-1}$ |
| $\alpha = 75.346 (7)^\circ$ | $T = 299 \text{ K}$ |
| $\beta = 83.188 (8)^\circ$ | Rod, colourless |
| $\gamma = 77.732 (7)^\circ$ | $0.20 \times 0.14 \times 0.08 \text{ mm}$ |
| $V = 1404.5 (2) \text{ \AA}^3$ | |

Data collection

| | |
|--|--|
| Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector | 5694 independent reflections |
| Radiation source: fine-focus sealed tube | 4267 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{\text{int}} = 0.018$ |
| Rotation method data acquisition using ω and φ scans | $\theta_{\text{max}} = 26.4^\circ$, $\theta_{\text{min}} = 2.5^\circ$ |
| Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009) | $h = -9 \rightarrow 5$ |
| $T_{\text{min}} = 0.887$, $T_{\text{max}} = 0.952$ | $k = -14 \rightarrow 14$ |
| 9768 measured reflections | $l = -19 \rightarrow 21$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.054$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.154$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.04$ | $w = 1/[\sigma^2(F_o^2) + (0.0682P)^2 + 1.64P]$ |

5694 reflections
367 parameters
2 restraints

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.014$$

$$\Delta\rho_{\max} = 1.10 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.50 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C1 | 0.2455 (4) | 1.0034 (3) | 0.13610 (19) | 0.0380 (7) |
| C2 | 0.4149 (4) | 1.0391 (3) | 0.1159 (2) | 0.0432 (8) |
| C3 | 0.4422 (5) | 1.1225 (3) | 0.0442 (2) | 0.0543 (9) |
| H3 | 0.5556 | 1.1464 | 0.0304 | 0.065* |
| C4 | 0.3009 (6) | 1.1700 (4) | -0.0067 (2) | 0.0594 (10) |
| H4 | 0.3199 | 1.2258 | -0.0548 | 0.071* |
| C5 | 0.1320 (5) | 1.1358 (3) | 0.0128 (2) | 0.0564 (10) |
| H5 | 0.0372 | 1.1690 | -0.0217 | 0.068* |
| C6 | 0.1041 (5) | 1.0522 (3) | 0.0839 (2) | 0.0473 (8) |
| H6 | -0.0095 | 1.0283 | 0.0970 | 0.057* |
| C7 | 0.3347 (4) | 0.7053 (3) | 0.1700 (2) | 0.0421 (7) |
| C8 | 0.4322 (4) | 0.5771 (3) | 0.1922 (2) | 0.0403 (7) |
| C9 | 0.5166 (5) | 0.5225 (3) | 0.1304 (2) | 0.0488 (8) |
| H9 | 0.5157 | 0.5659 | 0.0768 | 0.059* |
| C10 | 0.6012 (5) | 0.4034 (4) | 0.1500 (3) | 0.0539 (9) |
| C11 | 0.5995 (5) | 0.3361 (4) | 0.2282 (3) | 0.0596 (10) |
| H11 | 0.6549 | 0.2550 | 0.2399 | 0.072* |
| C12 | 0.5148 (6) | 0.3901 (4) | 0.2892 (3) | 0.0614 (10) |
| H12 | 0.5135 | 0.3450 | 0.3424 | 0.074* |
| C13 | 0.4315 (5) | 0.5106 (3) | 0.2723 (2) | 0.0487 (8) |
| H13 | 0.3757 | 0.5468 | 0.3138 | 0.058* |
| N1 | 0.3355 (4) | 0.7713 (2) | 0.22646 (18) | 0.0434 (7) |
| H1N | 0.423 (4) | 0.755 (3) | 0.2573 (19) | 0.052* |
| O1 | 0.0127 (3) | 0.8835 (2) | 0.22699 (17) | 0.0600 (7) |
| O2 | 0.2495 (4) | 0.9355 (2) | 0.29433 (14) | 0.0529 (6) |
| O3 | 0.2575 (4) | 0.7488 (2) | 0.10731 (15) | 0.0548 (6) |
| S1 | 0.19546 (11) | 0.90062 (8) | 0.22791 (5) | 0.0418 (2) |
| Cl1 | 0.59757 (13) | 0.98058 (10) | 0.17710 (6) | 0.0621 (3) |

supplementary materials

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|-----|--------------|--------------|--------------|-------------|
| C12 | 0.71150 (19) | 0.33696 (12) | 0.07272 (9) | 0.0864 (4) |
| C14 | 0.7395 (4) | 0.7963 (3) | 0.4444 (2) | 0.0418 (7) |
| C15 | 0.9034 (5) | 0.8353 (3) | 0.4444 (2) | 0.0469 (8) |
| C16 | 0.9072 (6) | 0.9573 (4) | 0.4233 (2) | 0.0573 (10) |
| H16 | 1.0162 | 0.9838 | 0.4240 | 0.069* |
| C17 | 0.7485 (7) | 1.0391 (4) | 0.4014 (3) | 0.0648 (11) |
| H17 | 0.7510 | 1.1211 | 0.3875 | 0.078* |
| C18 | 0.5875 (6) | 1.0018 (4) | 0.3996 (3) | 0.0633 (11) |
| H18 | 0.4816 | 1.0582 | 0.3841 | 0.076* |
| C19 | 0.5817 (5) | 0.8804 (3) | 0.4208 (2) | 0.0506 (9) |
| H19 | 0.4722 | 0.8550 | 0.4193 | 0.061* |
| C20 | 0.7982 (5) | 0.6143 (3) | 0.3232 (2) | 0.0473 (8) |
| C21 | 0.9079 (5) | 0.5420 (3) | 0.2688 (2) | 0.0470 (8) |
| C22 | 0.9803 (5) | 0.4209 (3) | 0.2962 (2) | 0.0511 (9) |
| H22 | 0.9681 | 0.3831 | 0.3507 | 0.061* |
| C23 | 1.0719 (5) | 0.3567 (4) | 0.2401 (3) | 0.0597 (10) |
| C24 | 1.0906 (6) | 0.4131 (5) | 0.1593 (3) | 0.0659 (11) |
| H24 | 1.1511 | 0.3691 | 0.1225 | 0.079* |
| C25 | 1.0212 (6) | 0.5326 (5) | 0.1332 (3) | 0.0721 (12) |
| H25 | 1.0363 | 0.5703 | 0.0788 | 0.087* |
| C26 | 0.9289 (5) | 0.5978 (4) | 0.1869 (2) | 0.0587 (10) |
| H26 | 0.8803 | 0.6793 | 0.1687 | 0.070* |
| N2 | 0.8416 (4) | 0.5803 (3) | 0.40323 (18) | 0.0477 (7) |
| H2N | 0.946 (3) | 0.537 (3) | 0.415 (2) | 0.057* |
| O4 | 0.5315 (3) | 0.6379 (3) | 0.4726 (2) | 0.0700 (8) |
| O5 | 0.8139 (3) | 0.5845 (2) | 0.54567 (15) | 0.0547 (6) |
| O6 | 0.6750 (4) | 0.6993 (2) | 0.30102 (18) | 0.0620 (8) |
| S2 | 0.71956 (11) | 0.64315 (8) | 0.47362 (6) | 0.0466 (2) |
| Cl3 | 1.10996 (13) | 0.73583 (10) | 0.46678 (7) | 0.0686 (3) |
| Cl4 | 1.1539 (2) | 0.20474 (11) | 0.27335 (10) | 0.0915 (4) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0388 (16) | 0.0328 (16) | 0.0392 (17) | -0.0003 (13) | -0.0070 (13) | -0.0059 (13) |
| C2 | 0.0401 (18) | 0.0421 (18) | 0.0462 (19) | -0.0046 (14) | -0.0122 (14) | -0.0067 (15) |
| C3 | 0.054 (2) | 0.054 (2) | 0.053 (2) | -0.0187 (18) | -0.0060 (17) | -0.0012 (17) |
| C4 | 0.075 (3) | 0.048 (2) | 0.049 (2) | -0.0125 (19) | -0.0116 (19) | 0.0042 (17) |
| C5 | 0.058 (2) | 0.050 (2) | 0.053 (2) | -0.0010 (18) | -0.0241 (18) | 0.0023 (17) |
| C6 | 0.0389 (18) | 0.0459 (19) | 0.055 (2) | -0.0031 (15) | -0.0151 (15) | -0.0060 (16) |
| C7 | 0.0380 (17) | 0.0400 (18) | 0.0467 (19) | -0.0116 (14) | -0.0031 (14) | -0.0040 (14) |
| C8 | 0.0340 (16) | 0.0381 (17) | 0.0496 (19) | -0.0107 (13) | -0.0064 (14) | -0.0067 (14) |
| C9 | 0.050 (2) | 0.048 (2) | 0.051 (2) | -0.0133 (16) | -0.0048 (16) | -0.0118 (16) |
| C10 | 0.047 (2) | 0.051 (2) | 0.069 (3) | -0.0105 (17) | -0.0015 (18) | -0.0247 (19) |
| C11 | 0.054 (2) | 0.042 (2) | 0.078 (3) | -0.0032 (17) | -0.006 (2) | -0.0086 (19) |
| C12 | 0.063 (2) | 0.047 (2) | 0.062 (3) | -0.0045 (19) | -0.003 (2) | 0.0032 (19) |
| C13 | 0.0467 (19) | 0.0434 (19) | 0.052 (2) | -0.0079 (15) | -0.0009 (16) | -0.0050 (16) |
| N1 | 0.0409 (15) | 0.0375 (15) | 0.0500 (17) | -0.0019 (12) | -0.0174 (12) | -0.0052 (13) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| O1 | 0.0351 (13) | 0.0638 (17) | 0.0706 (18) | -0.0073 (12) | -0.0041 (12) | 0.0016 (13) |
| O2 | 0.0630 (16) | 0.0529 (15) | 0.0388 (13) | -0.0027 (12) | -0.0037 (11) | -0.0104 (11) |
| O3 | 0.0632 (16) | 0.0486 (14) | 0.0504 (15) | -0.0082 (12) | -0.0212 (12) | -0.0019 (11) |
| S1 | 0.0359 (4) | 0.0405 (4) | 0.0432 (5) | -0.0018 (3) | -0.0037 (3) | -0.0033 (3) |
| C11 | 0.0421 (5) | 0.0781 (7) | 0.0618 (6) | -0.0153 (4) | -0.0194 (4) | 0.0017 (5) |
| C12 | 0.0947 (9) | 0.0792 (8) | 0.0946 (9) | -0.0088 (7) | 0.0062 (7) | -0.0494 (7) |
| C14 | 0.0397 (17) | 0.0442 (18) | 0.0381 (17) | -0.0008 (14) | -0.0055 (13) | -0.0082 (14) |
| C15 | 0.0420 (18) | 0.058 (2) | 0.0384 (18) | -0.0080 (16) | -0.0051 (14) | -0.0075 (16) |
| C16 | 0.063 (2) | 0.062 (2) | 0.051 (2) | -0.022 (2) | -0.0014 (18) | -0.0133 (18) |
| C17 | 0.087 (3) | 0.046 (2) | 0.059 (2) | -0.008 (2) | -0.006 (2) | -0.0116 (18) |
| C18 | 0.066 (3) | 0.052 (2) | 0.067 (3) | 0.008 (2) | -0.017 (2) | -0.014 (2) |
| C19 | 0.0440 (19) | 0.052 (2) | 0.056 (2) | 0.0047 (16) | -0.0138 (16) | -0.0199 (17) |
| C20 | 0.0471 (19) | 0.0395 (18) | 0.059 (2) | -0.0079 (15) | -0.0202 (16) | -0.0107 (16) |
| C21 | 0.0423 (18) | 0.0457 (19) | 0.056 (2) | -0.0091 (15) | -0.0205 (16) | -0.0082 (16) |
| C22 | 0.052 (2) | 0.046 (2) | 0.058 (2) | -0.0081 (16) | -0.0127 (17) | -0.0133 (17) |
| C23 | 0.051 (2) | 0.051 (2) | 0.083 (3) | -0.0113 (18) | -0.012 (2) | -0.022 (2) |
| C24 | 0.057 (2) | 0.086 (3) | 0.066 (3) | -0.024 (2) | 0.001 (2) | -0.032 (2) |
| C25 | 0.072 (3) | 0.086 (3) | 0.060 (3) | -0.027 (3) | -0.006 (2) | -0.010 (2) |
| C26 | 0.056 (2) | 0.060 (2) | 0.060 (2) | -0.0166 (19) | -0.0180 (19) | -0.0027 (19) |
| N2 | 0.0421 (16) | 0.0456 (17) | 0.0520 (18) | 0.0066 (13) | -0.0206 (13) | -0.0100 (13) |
| O4 | 0.0343 (13) | 0.0585 (17) | 0.112 (2) | -0.0072 (12) | -0.0075 (14) | -0.0100 (16) |
| O5 | 0.0500 (14) | 0.0572 (15) | 0.0441 (14) | 0.0009 (12) | -0.0030 (11) | 0.0020 (11) |
| O6 | 0.0612 (16) | 0.0461 (15) | 0.0787 (19) | 0.0104 (12) | -0.0416 (14) | -0.0149 (13) |
| S2 | 0.0334 (4) | 0.0445 (5) | 0.0555 (5) | -0.0009 (3) | -0.0062 (4) | -0.0041 (4) |
| C13 | 0.0357 (5) | 0.0757 (7) | 0.0822 (7) | -0.0091 (4) | -0.0112 (4) | 0.0057 (5) |
| C14 | 0.1048 (10) | 0.0482 (6) | 0.1177 (11) | 0.0030 (6) | -0.0070 (8) | -0.0277 (7) |

Geometric parameters (Å, °)

| | | | |
|---------|-----------|---------|-----------|
| C1—C2 | 1.389 (5) | C14—C19 | 1.388 (5) |
| C1—C6 | 1.394 (4) | C14—C15 | 1.390 (5) |
| C1—S1 | 1.775 (3) | C14—S2 | 1.765 (4) |
| C2—C3 | 1.386 (5) | C15—C16 | 1.384 (5) |
| C2—C11 | 1.730 (3) | C15—C13 | 1.736 (4) |
| C3—C4 | 1.377 (5) | C16—C17 | 1.375 (6) |
| C3—H3 | 0.93 | C16—H16 | 0.93 |
| C4—C5 | 1.377 (6) | C17—C18 | 1.365 (6) |
| C4—H4 | 0.93 | C17—H17 | 0.93 |
| C5—C6 | 1.381 (5) | C18—C19 | 1.380 (6) |
| C5—H5 | 0.93 | C18—H18 | 0.93 |
| C6—H6 | 0.93 | C19—H19 | 0.93 |
| C7—O3 | 1.220 (4) | C20—O6 | 1.214 (4) |
| C7—N1 | 1.382 (4) | C20—N2 | 1.384 (5) |
| C7—C8 | 1.493 (5) | C20—C21 | 1.475 (5) |
| C8—C9 | 1.391 (5) | C21—C22 | 1.382 (5) |
| C8—C13 | 1.396 (5) | C21—C26 | 1.396 (5) |
| C9—C10 | 1.374 (5) | C22—C23 | 1.393 (6) |
| C9—H9 | 0.93 | C22—H22 | 0.93 |
| C10—C11 | 1.372 (6) | C23—C24 | 1.379 (6) |

supplementary materials

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|-------------|------------|-------------|------------|
| C10—C12 | 1.738 (4) | C23—C14 | 1.719 (4) |
| C11—C12 | 1.377 (6) | C24—C25 | 1.359 (7) |
| C11—H11 | 0.93 | C24—H24 | 0.93 |
| C12—C13 | 1.383 (5) | C25—C26 | 1.371 (6) |
| C12—H12 | 0.93 | C25—H25 | 0.93 |
| C13—H13 | 0.93 | C26—H26 | 0.93 |
| N1—S1 | 1.647 (3) | N2—S2 | 1.650 (3) |
| N1—H1N | 0.845 (18) | N2—H2N | 0.846 (19) |
| O1—S1 | 1.418 (3) | O4—S2 | 1.416 (3) |
| O2—S1 | 1.427 (3) | O5—S2 | 1.435 (3) |
| C2—C1—C6 | 119.5 (3) | C19—C14—C15 | 119.4 (3) |
| C2—C1—S1 | 123.1 (2) | C19—C14—S2 | 117.3 (3) |
| C6—C1—S1 | 117.3 (3) | C15—C14—S2 | 123.2 (3) |
| C3—C2—C1 | 119.9 (3) | C16—C15—C14 | 120.0 (3) |
| C3—C2—C11 | 118.1 (3) | C16—C15—C13 | 117.6 (3) |
| C1—C2—C11 | 122.0 (3) | C14—C15—C13 | 122.4 (3) |
| C4—C3—C2 | 119.9 (4) | C17—C16—C15 | 119.5 (4) |
| C4—C3—H3 | 120.1 | C17—C16—H16 | 120.2 |
| C2—C3—H3 | 120.1 | C15—C16—H16 | 120.2 |
| C5—C4—C3 | 120.8 (4) | C18—C17—C16 | 121.0 (4) |
| C5—C4—H4 | 119.6 | C18—C17—H17 | 119.5 |
| C3—C4—H4 | 119.6 | C16—C17—H17 | 119.5 |
| C4—C5—C6 | 119.7 (3) | C17—C18—C19 | 120.0 (4) |
| C4—C5—H5 | 120.1 | C17—C18—H18 | 120.0 |
| C6—C5—H5 | 120.1 | C19—C18—H18 | 120.0 |
| C5—C6—C1 | 120.2 (3) | C18—C19—C14 | 120.0 (4) |
| C5—C6—H6 | 119.9 | C18—C19—H19 | 120.0 |
| C1—C6—H6 | 119.9 | C14—C19—H19 | 120.0 |
| O3—C7—N1 | 122.1 (3) | O6—C20—N2 | 120.3 (3) |
| O3—C7—C8 | 122.8 (3) | O6—C20—C21 | 123.3 (3) |
| N1—C7—C8 | 115.0 (3) | N2—C20—C21 | 116.4 (3) |
| C9—C8—C13 | 120.1 (3) | C22—C21—C26 | 120.2 (4) |
| C9—C8—C7 | 118.4 (3) | C22—C21—C20 | 121.8 (3) |
| C13—C8—C7 | 121.4 (3) | C26—C21—C20 | 117.9 (3) |
| C10—C9—C8 | 118.8 (4) | C21—C22—C23 | 118.5 (4) |
| C10—C9—H9 | 120.6 | C21—C22—H22 | 120.8 |
| C8—C9—H9 | 120.6 | C23—C22—H22 | 120.8 |
| C9—C10—C11 | 121.9 (4) | C24—C23—C22 | 120.6 (4) |
| C9—C10—C12 | 118.5 (3) | C24—C23—C14 | 120.8 (4) |
| C11—C10—C12 | 119.6 (3) | C22—C23—C14 | 118.6 (3) |
| C12—C11—C10 | 119.2 (4) | C25—C24—C23 | 120.5 (4) |
| C12—C11—H11 | 120.4 | C25—C24—H24 | 119.7 |
| C10—C11—H11 | 120.4 | C23—C24—H24 | 119.7 |
| C11—C12—C13 | 120.8 (4) | C24—C25—C26 | 120.2 (4) |
| C11—C12—H12 | 119.6 | C24—C25—H25 | 119.9 |
| C13—C12—H12 | 119.6 | C26—C25—H25 | 119.9 |
| C12—C13—C8 | 119.2 (4) | C25—C26—C21 | 120.1 (4) |
| C12—C13—H13 | 120.4 | C25—C26—H26 | 119.9 |
| C8—C13—H13 | 120.4 | C21—C26—H26 | 119.9 |

| | | | |
|-----------------|-------------|-----------------|-------------|
| C7—N1—S1 | 123.8 (2) | C20—N2—S2 | 122.5 (2) |
| C7—N1—H1N | 121 (3) | C20—N2—H2N | 119 (3) |
| S1—N1—H1N | 114 (3) | S2—N2—H2N | 117 (3) |
| O1—S1—O2 | 120.43 (17) | O4—S2—O5 | 119.14 (18) |
| O1—S1—N1 | 107.77 (16) | O4—S2—N2 | 109.45 (18) |
| O2—S1—N1 | 103.83 (15) | O5—S2—N2 | 104.14 (15) |
| O1—S1—C1 | 107.48 (16) | O4—S2—C14 | 107.70 (16) |
| O2—S1—C1 | 109.82 (15) | O5—S2—C14 | 110.08 (16) |
| N1—S1—C1 | 106.73 (15) | N2—S2—C14 | 105.52 (16) |
| C6—C1—C2—C3 | -0.2 (5) | C19—C14—C15—C16 | 2.0 (5) |
| S1—C1—C2—C3 | -178.1 (3) | S2—C14—C15—C16 | -177.8 (3) |
| C6—C1—C2—C11 | -179.0 (3) | C19—C14—C15—C13 | -175.8 (3) |
| S1—C1—C2—C11 | 3.1 (4) | S2—C14—C15—C13 | 4.5 (4) |
| C1—C2—C3—C4 | 0.2 (6) | C14—C15—C16—C17 | -1.0 (6) |
| C11—C2—C3—C4 | 179.1 (3) | C13—C15—C16—C17 | 176.9 (3) |
| C2—C3—C4—C5 | 0.2 (6) | C15—C16—C17—C18 | -0.4 (6) |
| C3—C4—C5—C6 | -0.6 (6) | C16—C17—C18—C19 | 0.6 (7) |
| C4—C5—C6—C1 | 0.7 (6) | C17—C18—C19—C14 | 0.4 (6) |
| C2—C1—C6—C5 | -0.3 (5) | C15—C14—C19—C18 | -1.7 (5) |
| S1—C1—C6—C5 | 177.7 (3) | S2—C14—C19—C18 | 178.1 (3) |
| O3—C7—C8—C9 | 31.9 (5) | O6—C20—C21—C22 | -149.0 (4) |
| N1—C7—C8—C9 | -149.0 (3) | N2—C20—C21—C22 | 30.7 (5) |
| O3—C7—C8—C13 | -144.7 (4) | O6—C20—C21—C26 | 27.5 (5) |
| N1—C7—C8—C13 | 34.4 (4) | N2—C20—C21—C26 | -152.8 (3) |
| C13—C8—C9—C10 | -1.1 (5) | C26—C21—C22—C23 | -0.7 (5) |
| C7—C8—C9—C10 | -177.8 (3) | C20—C21—C22—C23 | 175.8 (3) |
| C8—C9—C10—C11 | 2.2 (6) | C21—C22—C23—C24 | 0.4 (6) |
| C8—C9—C10—C12 | -178.3 (3) | C21—C22—C23—C14 | -177.4 (3) |
| C9—C10—C11—C12 | -1.7 (6) | C22—C23—C24—C25 | 0.5 (6) |
| C12—C10—C11—C12 | 178.8 (3) | C14—C23—C24—C25 | 178.3 (3) |
| C10—C11—C12—C13 | 0.2 (6) | C23—C24—C25—C26 | -1.1 (7) |
| C11—C12—C13—C8 | 0.8 (6) | C24—C25—C26—C21 | 0.8 (6) |
| C9—C8—C13—C12 | -0.3 (5) | C22—C21—C26—C25 | 0.1 (5) |
| C7—C8—C13—C12 | 176.3 (3) | C20—C21—C26—C25 | -176.5 (3) |
| O3—C7—N1—S1 | 15.6 (5) | O6—C20—N2—S2 | 5.7 (5) |
| C8—C7—N1—S1 | -163.5 (2) | C21—C20—N2—S2 | -173.9 (2) |
| C7—N1—S1—O1 | 52.6 (3) | C20—N2—S2—O4 | 53.1 (3) |
| C7—N1—S1—O2 | -178.6 (3) | C20—N2—S2—O5 | -178.5 (3) |
| C7—N1—S1—C1 | -62.6 (3) | C20—N2—S2—C14 | -62.6 (3) |
| C2—C1—S1—O1 | -178.8 (3) | C19—C14—S2—O4 | -4.5 (3) |
| C6—C1—S1—O1 | 3.3 (3) | C15—C14—S2—O4 | 175.2 (3) |
| C2—C1—S1—O2 | 48.6 (3) | C19—C14—S2—O5 | -135.9 (3) |
| C6—C1—S1—O2 | -129.4 (3) | C15—C14—S2—O5 | 43.9 (3) |
| C2—C1—S1—N1 | -63.4 (3) | C19—C14—S2—N2 | 112.3 (3) |
| C6—C1—S1—N1 | 118.7 (3) | C15—C14—S2—N2 | -67.9 (3) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------|-------|-------------|-------------|---------------|
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supplementary materials

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|--------------------------|----------|----------|-----------|---------|
| N1—H1N···O6 | 0.84 (2) | 2.02 (2) | 2.836 (4) | 162 (4) |
| N2—H2N···O5 ⁱ | 0.85 (2) | 2.10 (2) | 2.937 (4) | 173 (4) |

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

Fig. 1

