

## N-(4-Methylbenzoyl)-2-nitrobenzenesulfonamide

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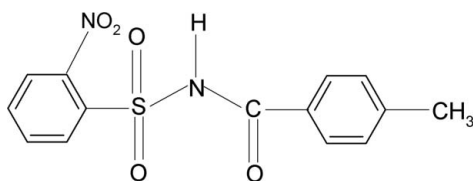
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.049;  $wR$  factor = 0.116; data-to-parameter ratio = 14.3.

The asymmetric unit of the title compound,  $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_5\text{S}$ , contains two independent molecules. The dihedral angles between the aromatic rings are  $82.03$  (9) and  $79.47$  (8)° in the two independent molecules. In the crystal, the two molecules in the asymmetric unit are linked into dimers *via* pairs of  $\text{N}-\text{H}\cdots\text{O}(\text{S})$  hydrogen bonds to generate  $C(4)$  chains.

### Related literature

For studies, including ours, on the effects of substituents on the structures and other aspects of *N*-(aryl)amides, see: Bowes *et al.* (2003); Gowda *et al.* (1999, 2003). For *N*-(aryl)methanesulfonamides, see: Gowda *et al.* (2007). For *N*-(aryl)arylsulfonamides, see: Shetty & Gowda (2005). For *N*-(substituted benzoyl)arylsulfonamides, see: Suchetan *et al.* (2012). For *N*-chloroarylamides, see: Jyothi & Gowda (2004). For *N*-bromoarylsulfonamides, see: Usha & Gowda (2006).



### Experimental

#### Crystal data

$\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_5\text{S}$   
 $M_r = 320.32$   
 Triclinic,  $P\bar{1}$   
 $a = 10.860$  (1) Å

$b = 11.716$  (2) Å  
 $c = 12.841$  (2) Å  
 $\alpha = 114.51$  (2)°  
 $\beta = 102.99$  (2)°

$\gamma = 91.16$  (1)°  
 $V = 1436.6$  (4) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.25$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.44 \times 0.44 \times 0.24$  mm

#### Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector  
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)  
 $T_{\min} = 0.898$ ,  $T_{\max} = 0.942$   
 8993 measured reflections  
 5772 independent reflections  
 4069 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.116$   
 $S = 1.06$   
 5772 reflections  
 405 parameters  
 2 restraints

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.29$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.31$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                         | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|----------|-------------|-------------|---------------|
| $\text{N3}-\text{H3N}\cdots\text{O2}$ | 0.85 (1) | 2.30 (1)    | 3.141 (3)   | 168 (3)       |

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: BT5801).

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## supplementary materials

*Acta Cryst.* (2012). E68, o572 [doi:10.1107/S1600536812003522]

***N*-(4-Methylbenzoyl)-2-nitrobenzenesulfonamide**

P. A. Suchetan, Sabine Foro and B. Thimme Gowda

**Comment**

Diaryl acylsulfonamides are known as potent antitumor agents. As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Bowes *et al.*, 2003; Gowda *et al.*, 1999, 2003), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007), *N*-(aryl)-arylsulfonamides (Shetty & Gowda, 2005); *N*-(substitutedbenzoyl)-arylsulfonamides (Suchetan *et al.*, 2012); *N*-chloroarylsulfonamides (Jyothi & Gowda, 2004) and *N*-bromoarylsulfonamides (Usha & Gowda, 2006), in the present work, the crystal structure of *N*-(4-methylbenzoyl)-2-nitrobenzenesulfonamide (I) has been determined (Fig.1).

The asymmetric unit of the structure contains two independent molecules. In one of the molecules, the N—C bond in the C—SO<sub>2</sub>—NH—C segment has *gauche* torsion with respect to the S=O bonds. The conformation between the N—H and C=O bonds in the C—SO<sub>2</sub>—NH—C(O) segments are *anti*. Further, the conformations between the N—H bonds and the *ortho*-nitro groups in the sulfonyl benzene rings are *syn*, similar to that observed in *N*-(3-methylbenzoyl)-2-nitrobenzenesulfonamide (II)(Suchetan *et al.*, 2012).

The molecules are twisted at the S—N bonds with the torsional angles of 64.42 (25)° and -55.37 (245)°, compared to the value of 64.32 (20)° in (II).

The dihedral angles between the sulfonyl benzene rings and the —SO<sub>2</sub>—NH—C—O segments are 76.73 (8)° and 79.47 (8)°, compared to the value of 75.7 (1)° in (II). Furthermore, the dihedral angles between the sulfonyl and the benzoyl benzene rings are 82.03 (9)° and 79.47 (8)°, compared to the value of 89.5 (1)° in (II).

In the crystal, the intermolecular N—H···O (S) hydrogen bonds (Table 1) link the molecules into chains. Part of the crystal structure is shown in Fig. 2.

**Experimental**

The title compound was prepared by refluxing a mixture of *p*-methylbenzoic acid (0.02 mole), 2-nitrobenzenesulfonamide (0.02 mole) and excess phosphorous oxychloride for 3 h on a water bath. The resultant mixture was cooled and poured into crushed ice. The solid, *N*-(4-methylbenzoyl)-2-nitrobenzenesulfonamide, 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.

Prism like colourless single crystals of the title compound used in X-ray diffraction studies were obtained by slow evaporation of its toluene solution at room temperature.

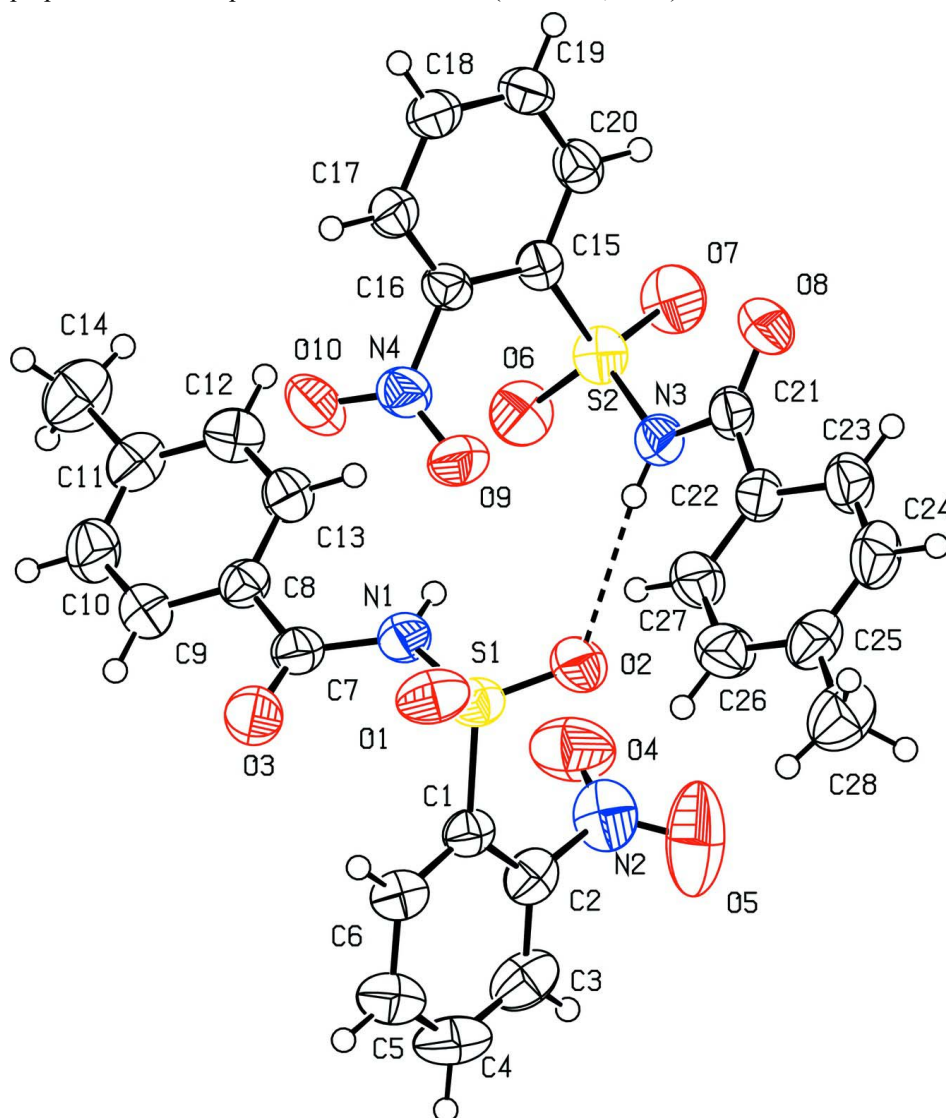
**Refinement**

The H atom of the NH group was located in a difference map and later restrained to N—H = 0.86 (2) Å. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å and the methyl C—H = 0.93 Å. All H atoms were refined with isotropic displacement parameters set at 1.2  $U_{eq}$ (C-aromatic, N) and 1.5

$U_{eq}(\text{C-methyl})$ .

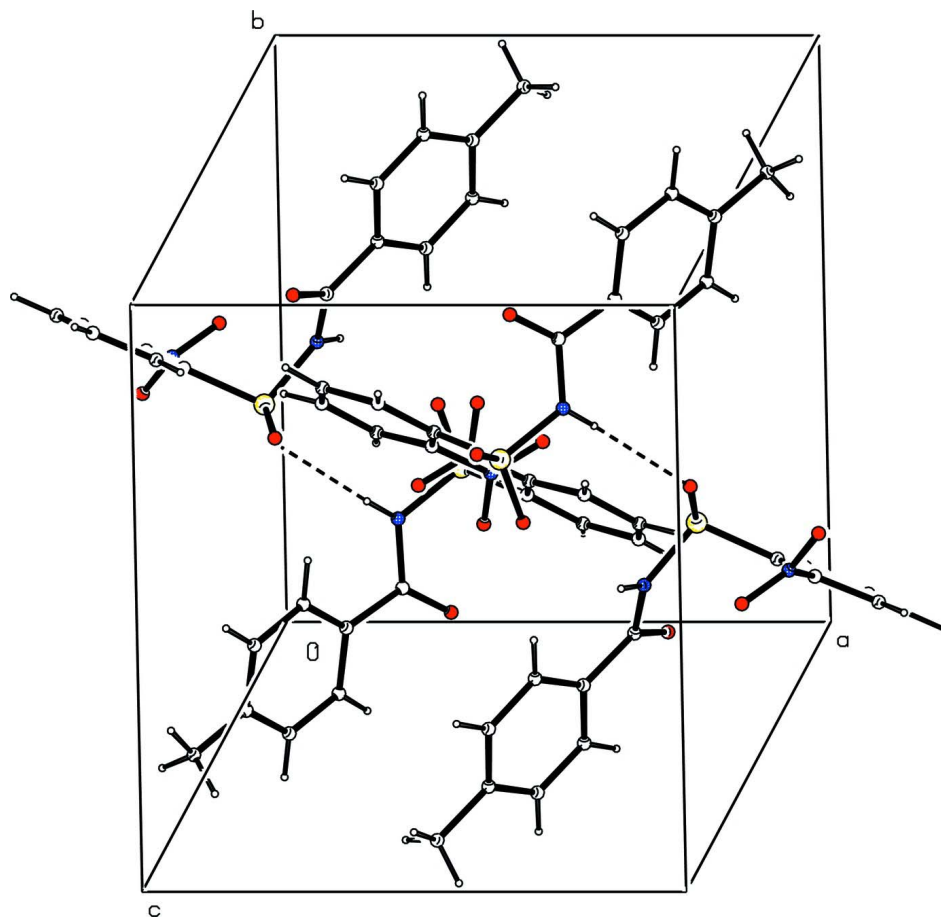
### Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); 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* (Sheldrick, 2008).



**Figure 1**

Molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

Molecular packing in the title compound. Hydrogen bonds are shown as dashed lines.

### *N*-(4-Methylbenzoyl)-2-nitrobenzenesulfonamide

#### Crystal data

$C_{14}H_{12}N_2O_5S$

$M_r = 320.32$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 10.860$  (1) Å

$b = 11.716$  (2) Å

$c = 12.841$  (2) Å

$\alpha = 114.51$  (2)°

$\beta = 102.99$  (2)°

$\gamma = 91.16$  (1)°

$V = 1436.6$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 664$

$D_x = 1.481$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 3497 reflections

$\theta = 2.6$ – $27.7$ °

$\mu = 0.25$  mm<sup>-1</sup>

$T = 293$  K

Prism, colourless

$0.44 \times 0.44 \times 0.24$  mm

#### Data collection

Oxford Diffraction Xcalibur

diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

Graphite monochromator

Rotation method data acquisition using  $\omega$  scans

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.898$ ,  $T_{\max} = 0.942$

8993 measured reflections

5772 independent reflections

4069 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$   
 $\theta_{\text{max}} = 26.4^\circ$ ,  $\theta_{\text{min}} = 2.6^\circ$   
 $h = -13 \rightarrow 13$

$k = -12 \rightarrow 14$   
 $l = -14 \rightarrow 16$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.116$   
 $S = 1.06$   
 5772 reflections  
 405 parameters  
 2 restraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0321P)^2 + 1.1761P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.018$   
 $\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.31 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

|     | $x$         | $y$        | $z$        | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|------------|------------|----------------------------------|
| C1  | -0.0965 (2) | 0.5718 (2) | 0.3025 (2) | 0.0424 (6)                       |
| C2  | -0.1921 (3) | 0.5564 (2) | 0.2042 (3) | 0.0485 (6)                       |
| C3  | -0.3051 (3) | 0.6057 (3) | 0.2163 (3) | 0.0642 (9)                       |
| H3  | -0.3696     | 0.5933     | 0.1498     | 0.077*                           |
| C4  | -0.3212 (3) | 0.6736 (3) | 0.3285 (4) | 0.0713 (10)                      |
| H4  | -0.3959     | 0.7095     | 0.3375     | 0.086*                           |
| C5  | -0.2284 (3) | 0.6885 (3) | 0.4263 (3) | 0.0661 (9)                       |
| H5  | -0.2405     | 0.7333     | 0.5015     | 0.079*                           |
| C6  | -0.1166 (3) | 0.6369 (3) | 0.4134 (3) | 0.0505 (7)                       |
| H6  | -0.0541     | 0.6462     | 0.4802     | 0.061*                           |
| C7  | 0.1874 (2)  | 0.7296 (3) | 0.3702 (2) | 0.0467 (6)                       |
| C8  | 0.2763 (2)  | 0.8070 (2) | 0.3448 (2) | 0.0428 (6)                       |
| C9  | 0.2955 (3)  | 0.9366 (3) | 0.4126 (3) | 0.0587 (8)                       |
| H9  | 0.2529      | 0.9730     | 0.4714     | 0.070*                           |
| C10 | 0.3781 (3)  | 1.0120 (3) | 0.3926 (3) | 0.0659 (9)                       |
| H10 | 0.3898      | 1.0990     | 0.4384     | 0.079*                           |
| C11 | 0.4436 (3)  | 0.9617 (3) | 0.3068 (3) | 0.0545 (7)                       |
| C12 | 0.4233 (3)  | 0.8330 (3) | 0.2396 (3) | 0.0525 (7)                       |
| H12 | 0.4656      | 0.7971     | 0.1805     | 0.063*                           |
| C13 | 0.3409 (3)  | 0.7557 (3) | 0.2582 (2) | 0.0473 (6)                       |

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|      |              |              |               |              |
|------|--------------|--------------|---------------|--------------|
| H13  | 0.3292       | 0.6688       | 0.2121        | 0.057*       |
| C14  | 0.5351 (3)   | 1.0447 (3)   | 0.2876 (3)    | 0.0790 (10)  |
| H14A | 0.6115       | 1.0720       | 0.3509        | 0.095*       |
| H14B | 0.4966       | 1.1172       | 0.2861        | 0.095*       |
| H14C | 0.5556       | 0.9979       | 0.2136        | 0.095*       |
| N1   | 0.1440 (2)   | 0.6072 (2)   | 0.2821 (2)    | 0.0488 (5)   |
| H1N  | 0.157 (3)    | 0.583 (3)    | 0.2132 (13)   | 0.059*       |
| N2   | -0.1779 (3)  | 0.4904 (3)   | 0.0836 (3)    | 0.0704 (8)   |
| O1   | 0.09629 (18) | 0.4991 (2)   | 0.40379 (19)  | 0.0633 (6)   |
| O2   | 0.02871 (19) | 0.39485 (17) | 0.18596 (19)  | 0.0601 (5)   |
| O3   | 0.1506 (2)   | 0.7687 (2)   | 0.45992 (18)  | 0.0659 (6)   |
| O4   | -0.0976 (3)  | 0.5353 (3)   | 0.0563 (2)    | 0.0944 (8)   |
| O5   | -0.2522 (4)  | 0.3971 (3)   | 0.0170 (2)    | 0.1311 (13)  |
| S1   | 0.04803 (6)  | 0.50626 (6)  | 0.29466 (7)   | 0.04812 (18) |
| C15  | 0.4933 (2)   | 0.2942 (2)   | 0.1683 (2)    | 0.0380 (5)   |
| C16  | 0.5306 (2)   | 0.3732 (2)   | 0.2893 (2)    | 0.0414 (6)   |
| C17  | 0.6504 (2)   | 0.3816 (3)   | 0.3571 (2)    | 0.0468 (6)   |
| H17  | 0.6740       | 0.4362       | 0.4374        | 0.056*       |
| C18  | 0.7355 (3)   | 0.3071 (3)   | 0.3037 (3)    | 0.0503 (7)   |
| H18  | 0.8171       | 0.3115       | 0.3485        | 0.060*       |
| C19  | 0.7009 (3)   | 0.2267 (3)   | 0.1852 (3)    | 0.0507 (7)   |
| H19  | 0.7585       | 0.1757       | 0.1506        | 0.061*       |
| C20  | 0.5805 (3)   | 0.2213 (2)   | 0.1172 (2)    | 0.0477 (6)   |
| H20  | 0.5583       | 0.1683       | 0.0365        | 0.057*       |
| C21  | 0.2377 (3)   | 0.0971 (2)   | 0.0854 (2)    | 0.0479 (6)   |
| C22  | 0.1465 (2)   | 0.0519 (2)   | 0.1349 (2)    | 0.0457 (6)   |
| C23  | 0.1197 (3)   | -0.0777 (3)  | 0.0968 (3)    | 0.0570 (7)   |
| H23  | 0.1556       | -0.1334      | 0.0392        | 0.068*       |
| C24  | 0.0401 (3)   | -0.1238 (3)  | 0.1443 (3)    | 0.0623 (8)   |
| H24  | 0.0222       | -0.2108      | 0.1168        | 0.075*       |
| C25  | -0.0139 (3)  | -0.0445 (3)  | 0.2314 (3)    | 0.0572 (7)   |
| C26  | 0.0137 (3)   | 0.0836 (3)   | 0.2686 (3)    | 0.0613 (8)   |
| H26  | -0.0217      | 0.1391       | 0.3268        | 0.074*       |
| C27  | 0.0925 (3)   | 0.1320 (3)   | 0.2217 (3)    | 0.0560 (7)   |
| H27  | 0.1093       | 0.2191       | 0.2487        | 0.067*       |
| C28  | -0.0969 (3)  | -0.0950 (3)  | 0.2860 (4)    | 0.0813 (10)  |
| H28A | -0.0514      | -0.1478      | 0.3175        | 0.098*       |
| H28B | -0.1728      | -0.1437      | 0.2267        | 0.098*       |
| H28C | -0.1197      | -0.0257      | 0.3486        | 0.098*       |
| N3   | 0.2353 (2)   | 0.2211 (2)   | 0.0981 (2)    | 0.0467 (5)   |
| H3N  | 0.185 (2)    | 0.269 (2)    | 0.133 (2)     | 0.056*       |
| N4   | 0.4405 (2)   | 0.4484 (2)   | 0.3514 (2)    | 0.0524 (6)   |
| O6   | 0.3190 (2)   | 0.42572 (18) | 0.11978 (19)  | 0.0637 (6)   |
| O7   | 0.3564 (2)   | 0.2349 (2)   | -0.04299 (17) | 0.0708 (6)   |
| O8   | 0.3129 (2)   | 0.03374 (18) | 0.0378 (2)    | 0.0677 (6)   |
| O9   | 0.3422 (2)   | 0.3918 (2)   | 0.34577 (19)  | 0.0780 (7)   |
| O10  | 0.4714 (2)   | 0.5617 (2)   | 0.4097 (2)    | 0.0713 (6)   |
| S2   | 0.34704 (7)  | 0.29805 (7)  | 0.07566 (6)   | 0.04910 (19) |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$    |
|-----|-------------|-------------|-------------|--------------|-------------|-------------|
| C1  | 0.0391 (14) | 0.0388 (13) | 0.0565 (16) | 0.0053 (11)  | 0.0140 (12) | 0.0266 (12) |
| C2  | 0.0499 (16) | 0.0463 (15) | 0.0566 (17) | 0.0015 (12)  | 0.0095 (13) | 0.0316 (14) |
| C3  | 0.0475 (17) | 0.076 (2)   | 0.083 (2)   | 0.0057 (15)  | 0.0037 (16) | 0.055 (2)   |
| C4  | 0.0478 (18) | 0.091 (2)   | 0.103 (3)   | 0.0269 (17)  | 0.0312 (19) | 0.062 (2)   |
| C5  | 0.060 (2)   | 0.075 (2)   | 0.078 (2)   | 0.0204 (17)  | 0.0329 (18) | 0.0380 (19) |
| C6  | 0.0441 (15) | 0.0573 (17) | 0.0577 (18) | 0.0087 (13)  | 0.0150 (13) | 0.0309 (14) |
| C7  | 0.0390 (14) | 0.0520 (16) | 0.0475 (16) | 0.0079 (12)  | 0.0113 (12) | 0.0198 (13) |
| C8  | 0.0377 (13) | 0.0456 (15) | 0.0452 (15) | 0.0063 (11)  | 0.0072 (11) | 0.0214 (12) |
| C9  | 0.0572 (18) | 0.0503 (17) | 0.0599 (19) | 0.0060 (14)  | 0.0195 (15) | 0.0132 (14) |
| C10 | 0.068 (2)   | 0.0407 (16) | 0.081 (2)   | 0.0025 (15)  | 0.0139 (18) | 0.0211 (16) |
| C11 | 0.0503 (16) | 0.0547 (17) | 0.0661 (19) | 0.0029 (13)  | 0.0075 (14) | 0.0373 (15) |
| C12 | 0.0562 (17) | 0.0580 (17) | 0.0555 (17) | 0.0113 (14)  | 0.0205 (14) | 0.0329 (15) |
| C13 | 0.0536 (16) | 0.0420 (14) | 0.0487 (16) | 0.0073 (12)  | 0.0136 (13) | 0.0216 (12) |
| C14 | 0.079 (2)   | 0.073 (2)   | 0.100 (3)   | -0.0053 (19) | 0.018 (2)   | 0.054 (2)   |
| N1  | 0.0477 (13) | 0.0477 (13) | 0.0526 (14) | 0.0023 (10)  | 0.0190 (11) | 0.0202 (12) |
| N2  | 0.084 (2)   | 0.0717 (19) | 0.0577 (18) | 0.0124 (16)  | 0.0071 (16) | 0.0360 (16) |
| O1  | 0.0498 (12) | 0.0827 (15) | 0.0815 (15) | 0.0207 (11)  | 0.0173 (11) | 0.0576 (13) |
| O2  | 0.0604 (13) | 0.0395 (10) | 0.0803 (15) | 0.0126 (9)   | 0.0240 (11) | 0.0224 (10) |
| O3  | 0.0666 (14) | 0.0700 (14) | 0.0563 (13) | -0.0036 (11) | 0.0273 (11) | 0.0171 (11) |
| O4  | 0.0899 (19) | 0.132 (2)   | 0.0839 (19) | 0.0306 (18)  | 0.0437 (16) | 0.0570 (18) |
| O5  | 0.191 (4)   | 0.098 (2)   | 0.0616 (18) | -0.042 (2)   | -0.013 (2)  | 0.0194 (16) |
| S1  | 0.0431 (4)  | 0.0460 (4)  | 0.0649 (5)  | 0.0112 (3)   | 0.0174 (3)  | 0.0311 (3)  |
| C15 | 0.0444 (14) | 0.0359 (13) | 0.0373 (13) | 0.0020 (11)  | 0.0130 (11) | 0.0180 (11) |
| C16 | 0.0447 (14) | 0.0406 (14) | 0.0440 (15) | 0.0077 (11)  | 0.0192 (12) | 0.0189 (12) |
| C17 | 0.0442 (15) | 0.0498 (15) | 0.0416 (15) | 0.0018 (12)  | 0.0118 (12) | 0.0150 (12) |
| C18 | 0.0403 (15) | 0.0529 (16) | 0.0591 (18) | 0.0062 (12)  | 0.0140 (13) | 0.0249 (14) |
| C19 | 0.0490 (16) | 0.0483 (15) | 0.0584 (18) | 0.0124 (13)  | 0.0251 (14) | 0.0205 (14) |
| C20 | 0.0583 (17) | 0.0423 (14) | 0.0434 (15) | 0.0048 (12)  | 0.0208 (13) | 0.0153 (12) |
| C21 | 0.0466 (15) | 0.0353 (14) | 0.0478 (16) | 0.0053 (12)  | 0.0049 (12) | 0.0081 (12) |
| C22 | 0.0418 (14) | 0.0371 (13) | 0.0495 (15) | 0.0060 (11)  | 0.0019 (12) | 0.0152 (12) |
| C23 | 0.0618 (18) | 0.0384 (15) | 0.0591 (18) | 0.0053 (13)  | 0.0095 (15) | 0.0128 (13) |
| C24 | 0.068 (2)   | 0.0386 (15) | 0.071 (2)   | -0.0025 (14) | 0.0025 (17) | 0.0222 (15) |
| C25 | 0.0461 (16) | 0.0550 (17) | 0.069 (2)   | 0.0004 (13)  | 0.0009 (14) | 0.0326 (16) |
| C26 | 0.0589 (18) | 0.0525 (17) | 0.076 (2)   | 0.0148 (14)  | 0.0243 (16) | 0.0271 (16) |
| C27 | 0.0614 (18) | 0.0370 (14) | 0.071 (2)   | 0.0116 (13)  | 0.0217 (16) | 0.0219 (14) |
| C28 | 0.072 (2)   | 0.077 (2)   | 0.099 (3)   | -0.0049 (19) | 0.017 (2)   | 0.046 (2)   |
| N3  | 0.0446 (13) | 0.0407 (12) | 0.0497 (14) | 0.0066 (10)  | 0.0092 (10) | 0.0160 (11) |
| N4  | 0.0483 (14) | 0.0625 (16) | 0.0407 (13) | 0.0112 (12)  | 0.0136 (11) | 0.0155 (12) |
| O6  | 0.0703 (14) | 0.0520 (12) | 0.0798 (15) | 0.0140 (10)  | 0.0135 (11) | 0.0415 (11) |
| O7  | 0.0799 (15) | 0.0911 (16) | 0.0404 (11) | 0.0088 (13)  | 0.0109 (10) | 0.0296 (11) |
| O8  | 0.0688 (14) | 0.0444 (11) | 0.0808 (15) | 0.0134 (10)  | 0.0312 (12) | 0.0119 (10) |
| O9  | 0.0462 (12) | 0.0970 (18) | 0.0665 (15) | -0.0018 (12) | 0.0232 (11) | 0.0081 (13) |
| O10 | 0.0874 (16) | 0.0527 (13) | 0.0704 (15) | 0.0213 (12)  | 0.0374 (13) | 0.0138 (11) |
| S2  | 0.0561 (4)  | 0.0505 (4)  | 0.0443 (4)  | 0.0073 (3)   | 0.0089 (3)  | 0.0258 (3)  |

Geometric parameters (Å, °)

|          |            |             |             |
|----------|------------|-------------|-------------|
| C1—C6    | 1.379 (4)  | C15—C20     | 1.381 (3)   |
| C1—C2    | 1.385 (4)  | C15—C16     | 1.394 (3)   |
| C1—S1    | 1.766 (3)  | C15—S2      | 1.771 (3)   |
| C2—C3    | 1.380 (4)  | C16—C17     | 1.370 (4)   |
| C2—N2    | 1.462 (4)  | C16—N4      | 1.474 (3)   |
| C3—C4    | 1.380 (5)  | C17—C18     | 1.381 (4)   |
| C3—H3    | 0.9300     | C17—H17     | 0.9300      |
| C4—C5    | 1.366 (5)  | C18—C19     | 1.372 (4)   |
| C4—H4    | 0.9300     | C18—H18     | 0.9300      |
| C5—C6    | 1.379 (4)  | C19—C20     | 1.383 (4)   |
| C5—H5    | 0.9300     | C19—H19     | 0.9300      |
| C6—H6    | 0.9300     | C20—H20     | 0.9300      |
| C7—O3    | 1.214 (3)  | C21—O8      | 1.210 (3)   |
| C7—N1    | 1.395 (3)  | C21—N3      | 1.395 (3)   |
| C7—C8    | 1.487 (4)  | C21—C22     | 1.486 (4)   |
| C8—C13   | 1.380 (4)  | C22—C27     | 1.384 (4)   |
| C8—C9    | 1.384 (4)  | C22—C23     | 1.389 (4)   |
| C9—C10   | 1.381 (4)  | C23—C24     | 1.379 (4)   |
| C9—H9    | 0.9300     | C23—H23     | 0.9300      |
| C10—C11  | 1.378 (4)  | C24—C25     | 1.384 (4)   |
| C10—H10  | 0.9300     | C24—H24     | 0.9300      |
| C11—C12  | 1.374 (4)  | C25—C26     | 1.376 (4)   |
| C11—C14  | 1.505 (4)  | C25—C28     | 1.503 (4)   |
| C12—C13  | 1.386 (4)  | C26—C27     | 1.382 (4)   |
| 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      |
| N1—S1    | 1.641 (2)  | N3—S2       | 1.642 (2)   |
| N1—H1N   | 0.855 (10) | N3—H3N      | 0.853 (10)  |
| N2—O4    | 1.196 (4)  | N4—O10      | 1.216 (3)   |
| N2—O5    | 1.207 (4)  | N4—O9       | 1.216 (3)   |
| O1—S1    | 1.419 (2)  | O6—S2       | 1.430 (2)   |
| O2—S1    | 1.429 (2)  | O7—S2       | 1.420 (2)   |
|          |            |             |             |
| C6—C1—C2 | 118.7 (2)  | C20—C15—C16 | 118.1 (2)   |
| C6—C1—S1 | 117.4 (2)  | C20—C15—S2  | 118.89 (19) |
| C2—C1—S1 | 123.8 (2)  | C16—C15—S2  | 122.58 (19) |
| C3—C2—C1 | 120.9 (3)  | C17—C16—C15 | 122.1 (2)   |
| C3—C2—N2 | 116.8 (3)  | C17—C16—N4  | 116.7 (2)   |
| C1—C2—N2 | 122.3 (3)  | C15—C16—N4  | 121.2 (2)   |
| C2—C3—C4 | 119.1 (3)  | C16—C17—C18 | 118.6 (2)   |
| C2—C3—H3 | 120.4      | C16—C17—H17 | 120.7       |
| C4—C3—H3 | 120.4      | C18—C17—H17 | 120.7       |
| C5—C4—C3 | 120.6 (3)  | C19—C18—C17 | 120.8 (3)   |
| C5—C4—H4 | 119.7      | C19—C18—H18 | 119.6       |
| C3—C4—H4 | 119.7      | C17—C18—H18 | 119.6       |



|               |             |                 |             |
|---------------|-------------|-----------------|-------------|
| C4—C5—C6      | 119.9 (3)   | C18—C19—C20     | 120.1 (3)   |
| C4—C5—H5      | 120.0       | C18—C19—H19     | 120.0       |
| C6—C5—H5      | 120.0       | C20—C19—H19     | 120.0       |
| C1—C6—C5      | 120.6 (3)   | C15—C20—C19     | 120.4 (2)   |
| C1—C6—H6      | 119.7       | C15—C20—H20     | 119.8       |
| C5—C6—H6      | 119.7       | C19—C20—H20     | 119.8       |
| O3—C7—N1      | 120.4 (2)   | O8—C21—N3       | 119.8 (3)   |
| O3—C7—C8      | 123.6 (3)   | O8—C21—C22      | 123.9 (2)   |
| N1—C7—C8      | 116.0 (2)   | N3—C21—C22      | 116.3 (2)   |
| C13—C8—C9     | 118.8 (3)   | C27—C22—C23     | 118.3 (3)   |
| C13—C8—C7     | 123.2 (2)   | C27—C22—C21     | 123.5 (2)   |
| C9—C8—C7      | 118.0 (2)   | C23—C22—C21     | 118.1 (3)   |
| C10—C9—C8     | 119.9 (3)   | C24—C23—C22     | 120.2 (3)   |
| C10—C9—H9     | 120.0       | C24—C23—H23     | 119.9       |
| C8—C9—H9      | 120.0       | C22—C23—H23     | 119.9       |
| C11—C10—C9    | 121.8 (3)   | C23—C24—C25     | 122.0 (3)   |
| C11—C10—H10   | 119.1       | C23—C24—H24     | 119.0       |
| C9—C10—H10    | 119.1       | C25—C24—H24     | 119.0       |
| C12—C11—C10   | 117.8 (3)   | C26—C25—C24     | 117.3 (3)   |
| C12—C11—C14   | 121.0 (3)   | C26—C25—C28     | 120.9 (3)   |
| C10—C11—C14   | 121.2 (3)   | C24—C25—C28     | 121.8 (3)   |
| C11—C12—C13   | 121.3 (3)   | C25—C26—C27     | 121.8 (3)   |
| C11—C12—H12   | 119.3       | C25—C26—H26     | 119.1       |
| C13—C12—H12   | 119.3       | C27—C26—H26     | 119.1       |
| C8—C13—C12    | 120.4 (3)   | C26—C27—C22     | 120.5 (3)   |
| C8—C13—H13    | 119.8       | C26—C27—H27     | 119.7       |
| C12—C13—H13   | 119.8       | C22—C27—H27     | 119.7       |
| C11—C14—H14A  | 109.5       | C25—C28—H28A    | 109.5       |
| C11—C14—H14B  | 109.5       | C25—C28—H28B    | 109.5       |
| H14A—C14—H14B | 109.5       | H28A—C28—H28B   | 109.5       |
| C11—C14—H14C  | 109.5       | C25—C28—H28C    | 109.5       |
| H14A—C14—H14C | 109.5       | H28A—C28—H28C   | 109.5       |
| H14B—C14—H14C | 109.5       | H28B—C28—H28C   | 109.5       |
| C7—N1—S1      | 123.28 (19) | C21—N3—S2       | 123.47 (19) |
| C7—N1—H1N     | 121 (2)     | C21—N3—H3N      | 122 (2)     |
| S1—N1—H1N     | 115 (2)     | S2—N3—H3N       | 113.1 (19)  |
| O4—N2—O5      | 124.6 (3)   | O10—N4—O9       | 124.0 (2)   |
| O4—N2—C2      | 118.1 (3)   | O10—N4—C16      | 118.3 (2)   |
| O5—N2—C2      | 117.2 (3)   | O9—N4—C16       | 117.5 (2)   |
| O1—S1—O2      | 119.62 (13) | O7—S2—O6        | 119.71 (14) |
| O1—S1—N1      | 109.89 (13) | O7—S2—N3        | 110.46 (13) |
| O2—S1—N1      | 104.51 (12) | O6—S2—N3        | 104.21 (12) |
| O1—S1—C1      | 106.60 (13) | O7—S2—C15       | 107.13 (13) |
| O2—S1—C1      | 109.45 (13) | O6—S2—C15       | 107.80 (12) |
| N1—S1—C1      | 106.06 (12) | N3—S2—C15       | 106.88 (11) |
| <br>          |             |                 |             |
| C6—C1—C2—C3   | 0.4 (4)     | C20—C15—C16—C17 | 1.0 (4)     |
| S1—C1—C2—C3   | 177.3 (2)   | S2—C15—C16—C17  | -171.2 (2)  |
| C6—C1—C2—N2   | 179.0 (3)   | C20—C15—C16—N4  | -176.5 (2)  |

|                 |             |                 |             |
|-----------------|-------------|-----------------|-------------|
| S1—C1—C2—N2     | -4.1 (4)    | S2—C15—C16—N4   | 11.3 (3)    |
| C1—C2—C3—C4     | 1.5 (4)     | C15—C16—C17—C18 | -1.3 (4)    |
| N2—C2—C3—C4     | -177.2 (3)  | N4—C16—C17—C18  | 176.3 (2)   |
| C2—C3—C4—C5     | -2.1 (5)    | C16—C17—C18—C19 | 0.1 (4)     |
| C3—C4—C5—C6     | 0.9 (5)     | C17—C18—C19—C20 | 1.3 (4)     |
| C2—C1—C6—C5     | -1.6 (4)    | C16—C15—C20—C19 | 0.5 (4)     |
| S1—C1—C6—C5     | -178.7 (2)  | S2—C15—C20—C19  | 173.0 (2)   |
| C4—C5—C6—C1     | 0.9 (5)     | C18—C19—C20—C15 | -1.7 (4)    |
| O3—C7—C8—C13    | 164.3 (3)   | O8—C21—C22—C27  | 157.4 (3)   |
| N1—C7—C8—C13    | -17.4 (4)   | N3—C21—C22—C27  | -21.4 (4)   |
| O3—C7—C8—C9     | -15.1 (4)   | O8—C21—C22—C23  | -18.9 (4)   |
| N1—C7—C8—C9     | 163.2 (3)   | N3—C21—C22—C23  | 162.3 (2)   |
| C13—C8—C9—C10   | 0.1 (4)     | C27—C22—C23—C24 | 0.7 (4)     |
| C7—C8—C9—C10    | 179.5 (3)   | C21—C22—C23—C24 | 177.1 (3)   |
| C8—C9—C10—C11   | -0.4 (5)    | C22—C23—C24—C25 | -1.0 (5)    |
| C9—C10—C11—C12  | 0.7 (5)     | C23—C24—C25—C26 | 0.8 (5)     |
| C9—C10—C11—C14  | -178.8 (3)  | C23—C24—C25—C28 | -177.8 (3)  |
| C10—C11—C12—C13 | -0.8 (4)    | C24—C25—C26—C27 | -0.3 (5)    |
| C14—C11—C12—C13 | 178.8 (3)   | C28—C25—C26—C27 | 178.3 (3)   |
| C9—C8—C13—C12   | -0.1 (4)    | C25—C26—C27—C22 | 0.0 (5)     |
| C7—C8—C13—C12   | -179.5 (2)  | C23—C22—C27—C26 | -0.2 (4)    |
| C11—C12—C13—C8  | 0.5 (4)     | C21—C22—C27—C26 | -176.4 (3)  |
| O3—C7—N1—S1     | -3.1 (4)    | O8—C21—N3—S2    | -12.4 (4)   |
| C8—C7—N1—S1     | 178.58 (18) | C22—C21—N3—S2   | 166.47 (19) |
| C3—C2—N2—O4     | 114.8 (3)   | C17—C16—N4—O10  | 55.5 (3)    |
| C1—C2—N2—O4     | -63.9 (4)   | C15—C16—N4—O10  | -126.9 (3)  |
| C3—C2—N2—O5     | -62.0 (4)   | C17—C16—N4—O9   | -120.9 (3)  |
| C1—C2—N2—O5     | 119.3 (3)   | C15—C16—N4—O9   | 56.8 (3)    |
| C7—N1—S1—O1     | -50.4 (3)   | C21—N3—S2—O7    | 60.8 (3)    |
| C7—N1—S1—O2     | -180.0 (2)  | C21—N3—S2—O6    | -169.4 (2)  |
| C7—N1—S1—C1     | 64.4 (2)    | C21—N3—S2—C15   | -55.4 (2)   |
| C6—C1—S1—O1     | 19.2 (2)    | C20—C15—S2—O7   | -8.7 (2)    |
| C2—C1—S1—O1     | -157.8 (2)  | C16—C15—S2—O7   | 163.4 (2)   |
| C6—C1—S1—O2     | 149.9 (2)   | C20—C15—S2—O6   | -138.8 (2)  |
| C2—C1—S1—O2     | -27.1 (3)   | C16—C15—S2—O6   | 33.3 (2)    |
| C6—C1—S1—N1     | -97.9 (2)   | C20—C15—S2—N3   | 109.7 (2)   |
| C2—C1—S1—N1     | 85.1 (2)    | C16—C15—S2—N3   | -78.2 (2)   |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-------------------------|-------------|---------------|-----------------------|-------------------------|
| N3—H3N...O2             | 0.85 (1)    | 2.30 (1)      | 3.141 (3)             | 168 (3)                 |