

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## Propyl 2-(4-methylbenzenesulfonamido)-benzoate

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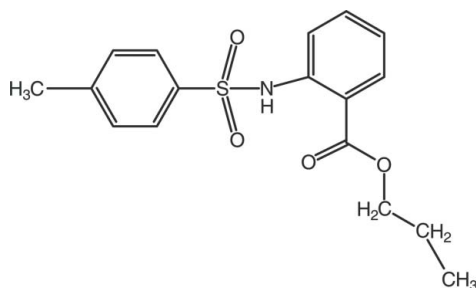
Received 9 April 2012; accepted 10 April 2012

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å; disorder in main residue;  $R$  factor = 0.056;  $wR$  factor = 0.178; data-to-parameter ratio = 19.3.

In the title compound,  $\text{C}_{17}\text{H}_{19}\text{NO}_4\text{S}$ , the terminal ethyl group is disordered over two sets of sites, with refined site occupancies of 0.536 (7) and 0.464 (7). The dihedral angle between the two aromatic rings is  $81.92$  ( $12$ )°. The molecular conformation is stabilized by intramolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, which generate  $S(6)$  motifs. In the crystal, molecules are linked by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, forming chains along the  $b$  axis.

### Related literature

For related structures, see: Mustafa *et al.* (2010, 2011, 2012); Khan *et al.* (2011). For bond-length data, see: Allen *et al.* (1987).



### Experimental

#### Crystal data

$\text{C}_{17}\text{H}_{19}\text{NO}_4\text{S}$   
 $M_r = 333.40$   
Monoclinic,  $P2_1/c$

$a = 16.206$  (5) Å  
 $b = 8.513$  (2) Å  
 $c = 12.021$  (3) Å

$\beta = 92.352$  (2)°  
 $V = 1657.0$  (8) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation

$\mu = 0.22$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.34 \times 0.22 \times 0.21$  mm

#### Data collection

Bruker APEXII CCD  
diffractometer  
15110 measured reflections

4039 independent reflections  
2343 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.071$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.178$   
 $S = 0.95$   
4039 reflections  
209 parameters

4 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.41$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.37$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                             | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1}\cdots\text{O3}$             | 0.86         | 2.11               | 2.643 (3)   | 119                  |
| $\text{C10}-\text{H10}\cdots\text{O1}^{\dagger}$ | 0.93         | 2.49               | 3.391 (3)   | 163                  |
| $\text{C9}-\text{H12}\cdots\text{O2}$            | 0.93         | 2.36               | 3.027 (3)   | 128                  |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors are grateful to the Higher Education Commission (HEC), Pakistan, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5208).

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## supplementary materials

*Acta Cryst.* (2012). E68, o1388 [doi:10.1107/S1600536812015528]

**Propyl 2-(4-methylbenzenesulfonamido)benzoate****Ghulam Mustafa, Tahir Muhmood, Islam Ullah Khan and Mehmet Akkurt****Comment**

As part of our ongoing studies of sulfonamides with potential biological properties (Mustafa *et al.*, 2010, 2011, 2012; Khan *et al.*, 2011), we now describe the title compound, propyl 2-[[4-methylphenyl)sulfonyl]amino}benzoate, (I).

In the title molecule (I), (Fig. 1), the dihedral angle between the two benzene rings (C2—C7) and (C8—C13) is 81.92 (12)°. The C—S—N—C torsion angle is 61.6 (2)°. All the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to those found for similar structures (Mustafa *et al.*, 2010; 2011, 2012; Khan *et al.*, 2011).

The molecular conformation of (I) is stabilized by intramolecular N—H···O and C—H···O hydrogen-bond interactions, generating S(6) motifs (Table 1). The crystal structure is stabilized by C—H···O hydrogen bonds, forming chains along the *b* axis (Table 1, Fig. 2).

**Experimental**

To an aqueous solution of *o*-amino benzoic acid (1.0 g, 7.3 mmol), sodium carbonate (1 N) was added to adjust the pH 8. Then *p*-toluenesulfonyl chloride (1.80 g, 9.48 mmol) was added and the mixture stirred at room temperature keeping the pH of the mixture up to 8.0 with occasional addition of sodium carbonate solution. Progress and completion of the reaction was confirmed by TLC and conversion of suspension into clear solution. After 2 h, whole mixture was poured into a beaker and the pH was adjusted to 2.0 by 1 N HCl. Precipitates were produced which were filtered and washed with distilled water.

The prepared sulfonamide (2-(Toluene-4-sulfonylamino)-benzoic acid) (1.0 g, 3.43 mmol), DMF (10 ml) and *n*-hexane washed sodium hydride (0.25 g, 10.31 mmol) were stirred at room temperature for 40 min followed by the addition of propyl iodide (0.76 g, 4.56 mmol). The whole reaction mixture was stirred till the completion of the reaction and poured into crushed ice in a beaker. The pH of the mixture was adjusted to 4.0 with 1 N HCl. Precipitates were produced, filtered, washed twice with distilled water and crystallized in chloroform.

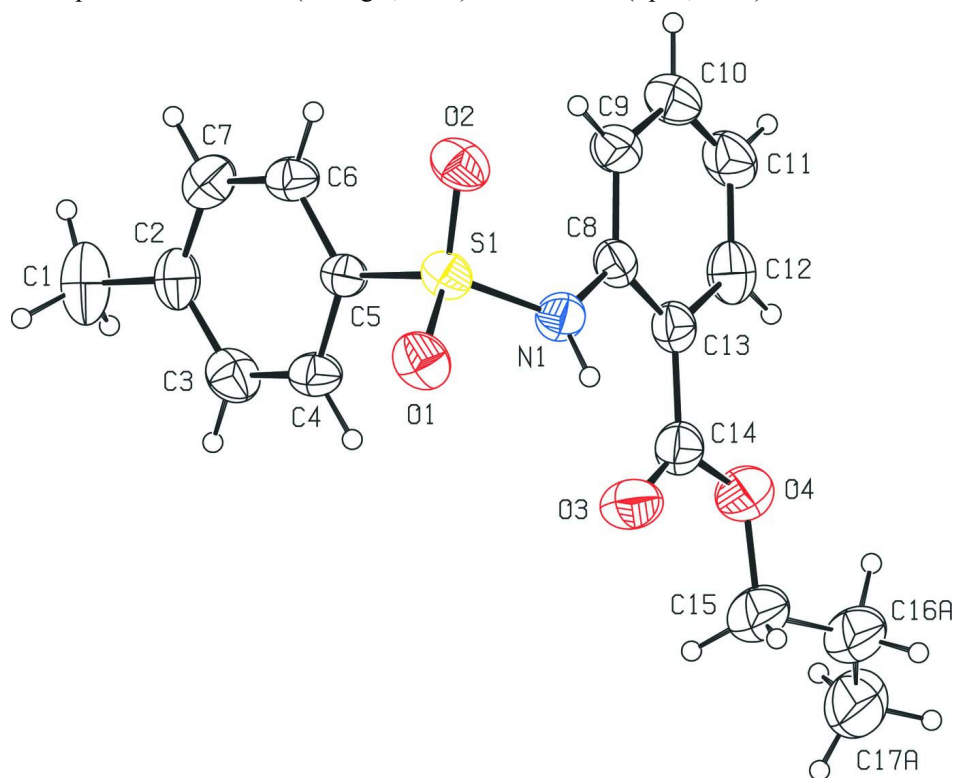
**Refinement**

All H atoms were positioned with idealized geometry and were refined using a riding model with N—H = 0.86 Å, C—H = 0.93, 0.96 or 0.97 Å, and with  $U_{\text{iso}}(\text{H}) = 1.2$  or  $1.5U_{\text{eq}}(\text{C}, \text{N})$ . The atoms of the terminal ethane group are disordered over two sets of sites, with refined site-occupancies of 0.536 (7): 0.464 (7). Five poorly fitted reflections (1 0 0), (-3 2 2), (8 0 2), (-3 6 1) and (-1 1 7) were omitted from the refinement.

**Computing details**

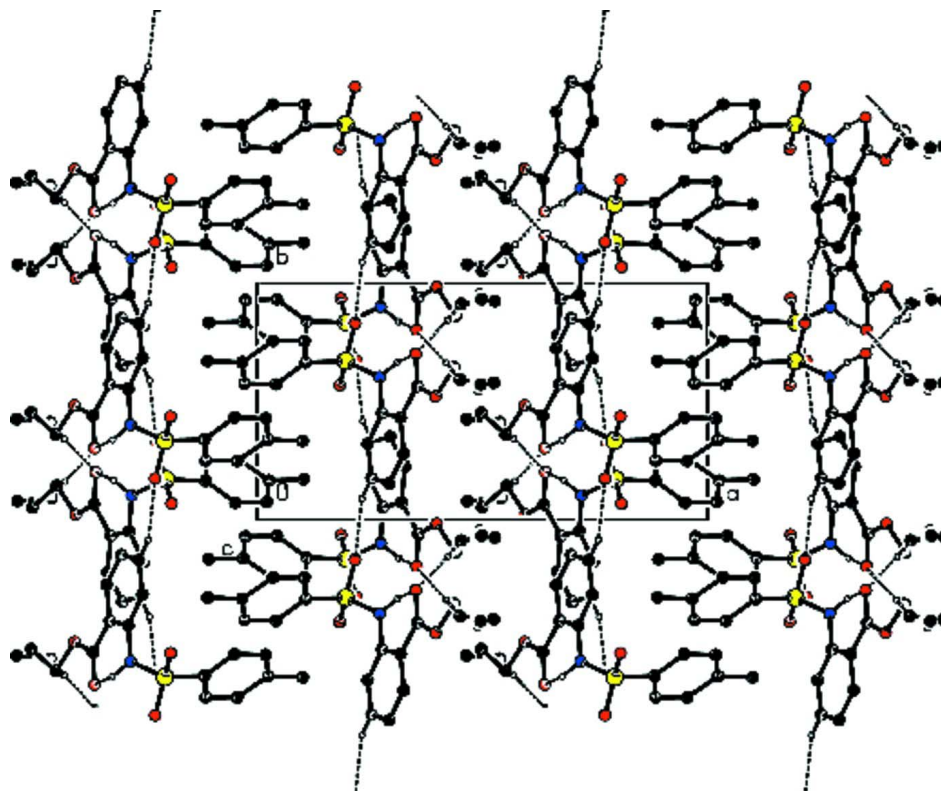
Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used

to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).



**Figure 1**

The molecular structure of (I) with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. Only the atoms of major disorder component are shown.



**Figure 2**

View of the hydrogen-bonding interactions of (I) along the *c* axis in the unit cell. Only the hydrogen atoms involved in hydrogen bonds (dotted lines) are drawn, for clarity.

### Propyl 2-(4-methylbenzenesulfonamido)benzoate

#### Crystal data

$C_{17}H_{19}NO_4S$

$M_r = 333.40$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P 2_1/c$

$a = 16.206 (5) \text{ \AA}$

$b = 8.513 (2) \text{ \AA}$

$c = 12.021 (3) \text{ \AA}$

$\beta = 92.352 (2)^\circ$

$V = 1657.0 (8) \text{ \AA}^3$

$Z = 4$

$F(000) = 704$

$D_x = 1.337 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4265 reflections

$\theta = 2.5\text{--}23.7^\circ$

$\mu = 0.22 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Needle, dark brown

$0.34 \times 0.22 \times 0.21 \text{ mm}$

#### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

15110 measured reflections

4039 independent reflections

2343 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.071$

$\theta_{\text{max}} = 28.3^\circ$ ,  $\theta_{\text{min}} = 2.5^\circ$

$h = -21 \rightarrow 21$

$k = -11 \rightarrow 9$

$l = -16 \rightarrow 16$

Refinement

|  |  |
|--|--|
| Refinement on $F^2$  | Secondary atom site location: difference Fourier map     |
| Least-squares matrix: full                                     | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.056$                                | H-atom parameters constrained                            |
| $wR(F^2) = 0.178$  | $w = 1/[\sigma^2(F_o^2) + (0.1068P)^2]$                  |
| $S = 0.95$   | where $P = (F_o^2 + 2F_c^2)/3$                           |
| 4039 reflections   | $(\Delta/\sigma)_{\max} < 0.001$                         |
| 209 parameters   | $\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$    |
| 4 restraints   | $\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$   |
| Primary atom site location: structure-invariant direct methods |  |

Special details

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

**Refinement.** Refinement on  $F^2$  for ALL reflections except those flagged by the user for potential systematic errors. Weighted  $R$ -factors  $wR$  and all goodnesses 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 observed criterion of  $F^2 > \sigma(F^2)$  is used only for calculating  $-R$ -factor-obs *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 ( $\text{\AA}^2$ )

|      | $x$          | $y$          | $z$           | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|--------------|--------------|---------------|----------------------------------|-----------|
| S1   | 0.80026 (4)  | 0.16805 (7)  | 0.29221 (4)   | 0.0584 (2)                       |           |
| O1   | 0.77925 (11) | 0.32859 (19) | 0.31033 (16)  | 0.0767 (7)                       |           |
| O2   | 0.81365 (11) | 0.06631 (19) | 0.38522 (12)  | 0.0750 (7)                       |           |
| O3   | 0.64329 (12) | 0.1994 (2)   | 0.03046 (17)  | 0.0869 (8)                       |           |
| O4   | 0.59833 (12) | 0.0193 (2)   | -0.09050 (17) | 0.0942 (8)                       |           |
| N1   | 0.72438 (11) | 0.1015 (2)   | 0.21272 (16)  | 0.0630 (7)                       |           |
| C1   | 1.10575 (19) | 0.1688 (4)   | 0.0220 (3)    | 0.1018 (14)                      |           |
| C2   | 1.02933 (15) | 0.1638 (3)   | 0.0893 (2)    | 0.0667 (9)                       |           |
| C3   | 0.96043 (17) | 0.2539 (3)   | 0.0577 (2)    | 0.0722 (10)                      |           |
| C4   | 0.89084 (15) | 0.2530 (3)   | 0.11718 (18)  | 0.0622 (8)                       |           |
| C5   | 0.88847 (13) | 0.1625 (2)   | 0.21260 (16)  | 0.0501 (7)                       |           |
| C6   | 0.95491 (14) | 0.0702 (3)   | 0.24444 (18)  | 0.0606 (8)                       |           |
| C7   | 1.02450 (15) | 0.0731 (3)   | 0.1834 (2)    | 0.0718 (9)                       |           |
| C8   | 0.71875 (13) | -0.0507 (2)  | 0.16566 (19)  | 0.0558 (7)                       |           |
| C9   | 0.75079 (16) | -0.1809 (3)  | 0.2218 (2)    | 0.0696 (9)                       |           |
| C10  | 0.74210 (19) | -0.3271 (3)  | 0.1755 (3)    | 0.0821 (11)                      |           |
| C11  | 0.70270 (19) | -0.3495 (3)  | 0.0752 (3)    | 0.0795 (11)                      |           |
| C12  | 0.66964 (16) | -0.2228 (3)  | 0.0188 (2)    | 0.0763 (10)                      |           |
| C13  | 0.67714 (13) | -0.0703 (3)  | 0.0631 (2)    | 0.0594 (8)                       |           |
| C14  | 0.63951 (15) | 0.0624 (4)   | 0.0015 (2)    | 0.0691 (10)                      |           |
| C15  | 0.5563 (2)   | 0.1421 (5)   | -0.1561 (3)   | 0.1144 (14)                      |           |
| C16A | 0.5014 (6)   | 0.0493 (10)  | -0.2409 (6)   | 0.1144 (14)                      | 0.536 (7) |
| C17A | 0.4972 (7)   | 0.070 (2)    | -0.3449 (9)   | 0.131 (4)                        | 0.536 (7) |
| C16B | 0.5434 (6)   | 0.0897 (12)  | -0.2659 (7)   | 0.1144 (14)                      | 0.464 (7) |
| C17B | 0.4682 (7)   | 0.077 (3)    | -0.3096 (12)  | 0.131 (4)                        | 0.464 (7) |
| H1   | 0.68400      | 0.16460      | 0.19800       | 0.0760*                          |           |

|      |         |          |          |         |           |
|------|---------|----------|----------|---------|-----------|
| H7   | 1.06970 | 0.01200  | 0.20630  | 0.0860* |           |
| H5   | 0.84520 | 0.31260  | 0.09390  | 0.0750* |           |
| H6   | 0.96210 | 0.31630  | -0.00570 | 0.0870* |           |
| H11  | 0.69810 | -0.44980 | 0.04490  | 0.0950* |           |
| H12  | 0.77820 | -0.16880 | 0.29080  | 0.0830* |           |
| H13  | 0.64190 | -0.23790 | -0.04960 | 0.0920* |           |
| H14A | 1.09050 | 0.15610  | -0.05550 | 0.1530* |           |
| H14B | 1.14240 | 0.08550  | 0.04550  | 0.1530* |           |
| H14C | 1.13300 | 0.26800  | 0.03320  | 0.1530* |           |
| H15A | 0.59570 | 0.20750  | -0.19340 | 0.1370* |           |
| H15B | 0.52310 | 0.20800  | -0.10950 | 0.1370* |           |
| H16A | 0.51600 | -0.06020 | -0.22990 | 0.1370* | 0.536 (7) |
| H16B | 0.44540 | 0.06040  | -0.21660 | 0.1370* | 0.536 (7) |
| H17A | 0.44610 | 0.02730  | -0.37500 | 0.1970* | 0.536 (7) |
| H17B | 0.54260 | 0.01780  | -0.37790 | 0.1970* | 0.536 (7) |
| H17C | 0.49940 | 0.18030  | -0.36090 | 0.1970* | 0.536 (7) |
| H8   | 0.95270 | 0.00630  | 0.30700  | 0.0730* |           |
| H10  | 0.76380 | -0.41340 | 0.21400  | 0.0980* |           |
| H16C | 0.57360 | 0.15980  | -0.31310 | 0.1370* | 0.464 (7) |
| H16D | 0.56910 | -0.01290 | -0.27080 | 0.1370* | 0.464 (7) |
| H17D | 0.46750 | 0.00200  | -0.36910 | 0.1970* | 0.464 (7) |
| H17E | 0.45030 | 0.17760  | -0.33810 | 0.1970* | 0.464 (7) |
| H17F | 0.43160 | 0.04330  | -0.25350 | 0.1970* | 0.464 (7) |

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

|      | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| S1   | 0.0714 (4)  | 0.0507 (4)  | 0.0533 (3)  | -0.0016 (3)  | 0.0056 (3)   | -0.0040 (2)  |
| O1   | 0.0920 (13) | 0.0510 (11) | 0.0879 (12) | 0.0020 (8)   | 0.0141 (10)  | -0.0185 (8)  |
| O2   | 0.1016 (13) | 0.0742 (12) | 0.0496 (9)  | -0.0102 (9)  | 0.0074 (9)   | 0.0070 (8)   |
| O3   | 0.0933 (14) | 0.0751 (14) | 0.0913 (13) | 0.0160 (10)  | -0.0083 (11) | 0.0078 (11)  |
| O4   | 0.0951 (14) | 0.1008 (16) | 0.0846 (12) | -0.0095 (11) | -0.0226 (11) | 0.0158 (11)  |
| N1   | 0.0598 (11) | 0.0503 (12) | 0.0785 (13) | 0.0065 (9)   | -0.0010 (9)  | -0.0059 (10) |
| C1   | 0.0773 (19) | 0.131 (3)   | 0.099 (2)   | -0.0341 (17) | 0.0251 (17)  | -0.043 (2)   |
| C2   | 0.0646 (15) | 0.0729 (17) | 0.0628 (14) | -0.0159 (12) | 0.0046 (11)  | -0.0227 (13) |
| C3   | 0.0886 (19) | 0.0786 (18) | 0.0495 (12) | -0.0142 (14) | 0.0033 (12)  | 0.0045 (12)  |
| C4   | 0.0723 (15) | 0.0624 (15) | 0.0515 (12) | 0.0043 (11)  | -0.0038 (11) | 0.0084 (10)  |
| C5   | 0.0618 (12) | 0.0434 (12) | 0.0444 (10) | -0.0025 (9)  | -0.0060 (9)  | -0.0020 (8)  |
| C6   | 0.0729 (15) | 0.0562 (14) | 0.0519 (12) | 0.0057 (11)  | -0.0061 (11) | 0.0017 (10)  |
| C7   | 0.0651 (15) | 0.0727 (18) | 0.0768 (16) | 0.0088 (12)  | -0.0077 (13) | -0.0115 (13) |
| C8   | 0.0532 (12) | 0.0477 (13) | 0.0668 (13) | -0.0045 (10) | 0.0077 (10)  | -0.0013 (10) |
| C9   | 0.0758 (16) | 0.0529 (15) | 0.0792 (16) | -0.0033 (12) | -0.0070 (13) | 0.0034 (12)  |
| C10  | 0.0897 (19) | 0.0503 (16) | 0.106 (2)   | 0.0007 (13)  | 0.0017 (17)  | 0.0060 (14)  |
| C11  | 0.0877 (19) | 0.0483 (16) | 0.103 (2)   | -0.0054 (13) | 0.0099 (16)  | -0.0103 (14) |
| C12  | 0.0714 (17) | 0.0787 (19) | 0.0791 (17) | -0.0157 (14) | 0.0054 (13)  | -0.0180 (15) |
| C13  | 0.0514 (12) | 0.0585 (15) | 0.0687 (14) | -0.0042 (10) | 0.0088 (10)  | -0.0021 (11) |
| C14  | 0.0604 (14) | 0.0755 (19) | 0.0714 (16) | -0.0047 (13) | 0.0026 (12)  | 0.0028 (13)  |
| C15  | 0.092 (2)   | 0.142 (3)   | 0.107 (2)   | -0.0034 (17) | -0.0213 (17) | 0.043 (2)    |
| C16A | 0.092 (2)   | 0.142 (3)   | 0.107 (2)   | -0.0034 (17) | -0.0213 (17) | 0.043 (2)    |

|      |           |           |           |              |              |           |
|------|-----------|-----------|-----------|--------------|--------------|-----------|
| C17A | 0.090 (7) | 0.188 (6) | 0.116 (7) | -0.010 (7)   | 0.012 (4)    | 0.050 (7) |
| C16B | 0.092 (2) | 0.142 (3) | 0.107 (2) | -0.0034 (17) | -0.0213 (17) | 0.043 (2) |
| C17B | 0.090 (7) | 0.188 (6) | 0.116 (7) | -0.010 (7)   | 0.012 (4)    | 0.050 (7) |

*Geometric parameters (Å, °)*

|            |             |               |            |
|------------|-------------|---------------|------------|
| S1—O1      | 1.4273 (18) | C16A—C17A     | 1.262 (13) |
| S1—O2      | 1.4239 (17) | C16B—C17B     | 1.312 (15) |
| S1—N1      | 1.628 (2)   | C1—H14A       | 0.9600     |
| S1—C5      | 1.753 (2)   | C1—H14B       | 0.9600     |
| O3—C14     | 1.218 (4)   | C1—H14C       | 0.9600     |
| O4—C14     | 1.320 (3)   | C3—H6         | 0.9300     |
| O4—C15     | 1.461 (4)   | C4—H5         | 0.9300     |
| N1—C8      | 1.415 (3)   | C6—H8         | 0.9300     |
| N1—H1      | 0.8600      | C7—H7         | 0.9300     |
| C1—C2      | 1.507 (4)   | C9—H12        | 0.9300     |
| C2—C3      | 1.395 (4)   | C10—H10       | 0.9300     |
| C2—C7      | 1.375 (3)   | C11—H11       | 0.9300     |
| C3—C4      | 1.360 (4)   | C12—H13       | 0.9300     |
| C4—C5      | 1.384 (3)   | C15—H15A      | 0.9700     |
| C5—C6      | 1.375 (3)   | C15—H15B      | 0.9700     |
| C6—C7      | 1.371 (3)   | C16A—H16A     | 0.9700     |
| C8—C9      | 1.387 (3)   | C16A—H16B     | 0.9700     |
| C8—C13     | 1.391 (3)   | C16B—H16C     | 0.9700     |
| C9—C10     | 1.368 (4)   | C16B—H16D     | 0.9700     |
| C10—C11    | 1.355 (5)   | C17A—H17C     | 0.9600     |
| C11—C12    | 1.371 (4)   | C17A—H17A     | 0.9600     |
| C12—C13    | 1.407 (4)   | C17A—H17B     | 0.9600     |
| C13—C14    | 1.470 (4)   | C17B—H17D     | 0.9600     |
| C15—C16A   | 1.543 (9)   | C17B—H17E     | 0.9600     |
| C15—C16B   | 1.401 (9)   | C17B—H17F     | 0.9600     |
| O1—S1—O2   | 119.49 (11) | C4—C3—H6      | 119.00     |
| O1—S1—N1   | 104.10 (10) | C3—C4—H5      | 120.00     |
| O1—S1—C5   | 108.30 (10) | C5—C4—H5      | 120.00     |
| O2—S1—N1   | 109.66 (10) | C5—C6—H8      | 120.00     |
| O2—S1—C5   | 108.11 (10) | C7—C6—H8      | 120.00     |
| N1—S1—C5   | 106.47 (10) | C2—C7—H7      | 119.00     |
| C14—O4—C15 | 117.5 (2)   | C6—C7—H7      | 119.00     |
| S1—N1—C8   | 126.02 (15) | C8—C9—H12     | 120.00     |
| C8—N1—H1   | 117.00      | C10—C9—H12    | 120.00     |
| S1—N1—H1   | 117.00      | C9—C10—H10    | 119.00     |
| C1—C2—C7   | 122.2 (2)   | C11—C10—H10   | 119.00     |
| C1—C2—C3   | 120.3 (2)   | C10—C11—H11   | 120.00     |
| C3—C2—C7   | 117.5 (2)   | C12—C11—H11   | 120.00     |
| C2—C3—C4   | 121.7 (2)   | C11—C12—H13   | 120.00     |
| C3—C4—C5   | 119.4 (2)   | C13—C12—H13   | 120.00     |
| C4—C5—C6   | 120.1 (2)   | O4—C15—H15A   | 111.00     |
| S1—C5—C4   | 119.24 (16) | O4—C15—H15B   | 111.00     |
| S1—C5—C6   | 120.69 (15) | C16A—C15—H15A | 111.00     |

|                 |              |                  |              |
|-----------------|--------------|------------------|--------------|
| C5—C6—C7        | 119.5 (2)    | C16A—C15—H15B    | 111.00       |
| C2—C7—C6        | 121.8 (2)    | H15A—C15—H15B    | 109.00       |
| N1—C8—C13       | 119.02 (19)  | C16B—C15—H15A    | 80.00        |
| N1—C8—C9        | 121.4 (2)    | C16B—C15—H15B    | 132.00       |
| C9—C8—C13       | 119.6 (2)    | C15—C16A—H16A    | 106.00       |
| C8—C9—C10       | 119.9 (2)    | C15—C16A—H16B    | 106.00       |
| C9—C10—C11      | 121.8 (3)    | C17A—C16A—H16A   | 106.00       |
| C10—C11—C12     | 119.3 (2)    | C17A—C16A—H16B   | 106.00       |
| C11—C12—C13     | 120.9 (2)    | H16A—C16A—H16B   | 106.00       |
| C12—C13—C14     | 119.4 (2)    | C15—C16B—H16D    | 107.00       |
| C8—C13—C12      | 118.5 (2)    | C17B—C16B—H16C   | 107.00       |
| C8—C13—C14      | 122.0 (2)    | C17B—C16B—H16D   | 107.00       |
| O4—C14—C13      | 113.2 (3)    | H16C—C16B—H16D   | 107.00       |
| O3—C14—O4       | 121.6 (3)    | C15—C16B—H16C    | 107.00       |
| O3—C14—C13      | 125.3 (2)    | H17A—C17A—H17C   | 109.00       |
| O4—C15—C16A     | 103.5 (4)    | H17B—C17A—H17C   | 110.00       |
| O4—C15—C16B     | 109.2 (5)    | C16A—C17A—H17A   | 109.00       |
| C15—C16A—C17A   | 126.1 (9)    | C16A—C17A—H17B   | 110.00       |
| C15—C16B—C17B   | 120.3 (10)   | C16A—C17A—H17C   | 110.00       |
| C2—C1—H14A      | 109.00       | H17A—C17A—H17B   | 109.00       |
| C2—C1—H14B      | 109.00       | C16B—C17B—H17D   | 110.00       |
| C2—C1—H14C      | 109.00       | C16B—C17B—H17E   | 109.00       |
| H14A—C1—H14B    | 110.00       | C16B—C17B—H17F   | 110.00       |
| H14A—C1—H14C    | 109.00       | H17D—C17B—H17E   | 109.00       |
| H14B—C1—H14C    | 109.00       | H17D—C17B—H17F   | 110.00       |
| C2—C3—H6        | 119.00       | H17E—C17B—H17F   | 109.00       |
| O1—S1—N1—C8     | 175.93 (18)  | C3—C4—C5—S1      | 177.35 (18)  |
| O2—S1—N1—C8     | -55.1 (2)    | C4—C5—C6—C7      | 2.5 (3)      |
| C5—S1—N1—C8     | 61.6 (2)     | S1—C5—C6—C7      | -177.21 (18) |
| O1—S1—C5—C4     | -48.9 (2)    | C5—C6—C7—C2      | -1.3 (4)     |
| O2—S1—C5—C4     | -179.66 (17) | N1—C8—C13—C14    | 0.6 (3)      |
| N1—S1—C5—C4     | 62.58 (19)   | C9—C8—C13—C12    | -0.7 (3)     |
| O1—S1—C5—C6     | 130.81 (18)  | C9—C8—C13—C14    | 177.9 (2)    |
| O2—S1—C5—C6     | 0.0 (2)      | C13—C8—C9—C10    | 0.7 (4)      |
| N1—S1—C5—C6     | -117.76 (18) | N1—C8—C13—C12    | -178.0 (2)   |
| C15—O4—C14—O3   | -0.8 (4)     | N1—C8—C9—C10     | 178.0 (2)    |
| C15—O4—C14—C13  | 178.1 (2)    | C8—C9—C10—C11    | 0.0 (4)      |
| C14—O4—C15—C16A | -168.8 (4)   | C9—C10—C11—C12   | -0.8 (5)     |
| S1—N1—C8—C9     | 34.1 (3)     | C10—C11—C12—C13  | 0.9 (4)      |
| S1—N1—C8—C13    | -148.64 (18) | C11—C12—C13—C14  | -178.8 (2)   |
| C3—C2—C7—C6     | 0.1 (4)      | C11—C12—C13—C8   | -0.1 (4)     |
| C1—C2—C3—C4     | -179.4 (3)   | C8—C13—C14—O3    | 2.9 (4)      |
| C1—C2—C7—C6     | 179.6 (3)    | C8—C13—C14—O4    | -175.9 (2)   |
| C7—C2—C3—C4     | 0.1 (4)      | C12—C13—C14—O3   | -178.5 (2)   |
| C2—C3—C4—C5     | 1.1 (4)      | C12—C13—C14—O4   | 2.7 (3)      |
| C3—C4—C5—C6     | -2.3 (3)     | O4—C15—C16A—C17A | -129.7 (11)  |



*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> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1···O3                | 0.86        | 2.11          | 2.643 (3)             | 119                     |
| C6—H8···O2                | 0.93        | 2.53          | 2.902 (3)             | 104                     |
| C10—H10···O1 <sup>i</sup> | 0.93        | 2.49          | 3.391 (3)             | 163                     |
| C9—H12···O2               | 0.93        | 2.36          | 3.027 (3)             | 128                     |
| C12—H13···O4              | 0.93        | 2.35          | 2.681 (3)             | 101                     |

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