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N-(3,4-Dimethylphenyl)benzene-sulfonamide

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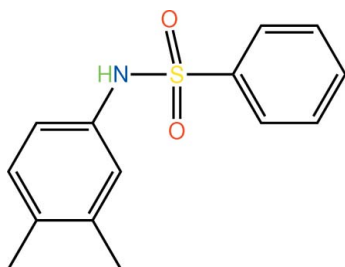
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.055; wR factor = 0.196; data-to-parameter ratio = 17.0.

The structure of the title compound, $\text{C}_{14}\text{H}_{15}\text{NO}_2\text{S}$, shows the sulfonamide N atom to be approximately perpendicular to the plane through the S-bound benzene ring [the N—S—C—C torsion angle is -87.4 (3°)] and to lie to the opposite side of this ring to the two sulfonamide O atoms. The N-bound benzene ring is splayed out with respect to the rest of the molecule so that overall, the molecule adopts a twisted conformation. The dihedral angle between the two benzene rings is 64.5 (3°). In the crystal, supramolecular chains aligned along the b axis are formed *via* N—H \cdots O hydrogen bonds.

Related literature

For background to the pharmacological uses of sulfonamides, see: Korolkovas (1988); Mandell & Sande (1992). For the structure of the *S*-tosyl derivative, see: Gowda *et al.* (2009). For related structures, see: Khan *et al.* (2010); Sharif *et al.* (2010).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{15}\text{NO}_2\text{S}$
 $M_r = 261.33$

 Monoclinic, $C2/c$
 $a = 24.2129$ (12) Å

 $b = 9.2616$ (5) Å
 $c = 16.5584$ (16) Å
 $\beta = 132.014$ (2°)
 $V = 2758.9$ (3) Å³
 $Z = 8$

 Mo $K\alpha$ radiation
 $\mu = 0.23$ mm⁻¹
 $T = 293$ K
 $0.31 \times 0.08 \times 0.07$ mm

Data collection

 Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.795$, $T_{\max} = 0.947$

 11486 measured reflections
 2864 independent reflections
 2112 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.196$
 $S = 1.09$
 2864 reflections
 168 parameters
 37 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.56$ e Å⁻³
 $\Delta\rho_{\min} = -0.65$ 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
N1—H1 \cdots O2 ⁱ	0.85 (3)	2.07 (2)	2.921 (3)	177 (5)

 Symmetry code: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg, 2006); software used to prepare material for publication: publCIF (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5533).

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supporting information

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***N*-(3,4-Dimethylphenyl)benzenesulfonamide**

Peter John, Shahzad Sharif, Islam Ullah Khan, Saima Khizar and Edward R. T. Tiekink

S1. Comment

In continuation of on-going structural studies of sulfonamides (Khan *et al.*, 2010; Sharif *et al.*, 2010), of interest owing to their putative anti-microbial activity (Korolkovas, 1988; Mandell & Sande, 1992), the crystal and molecular structure of the title compound, (I), was investigated.

In (I), both sulfonamido-O atoms lie to one side of the S-bound benzene ring [the O1–S1–C1–C6 and O2–S1–C1–C2 torsion angles are 19.3 (4) and -28.0 (3) °, respectively] and are on the opposite side of this ring to the sulfonamido-N1 atom, Fig. 1. The latter is approximately perpendicular to the benzene ring [N1–S1–C1–C2 is -87.4 (3) °] with the N-bound benzene ring clearly displaced to one side of the molecule; the dihedral angle formed between the two benzene rings is 64.5 (3) °. Although not isomorphous, the overall molecular conformation found in the closely related derivative, with an S-bound tosyl group, is almost identical (Gowda *et al.* 2009).

The presence of N1–H···O2 hydrogen bonding, Table 1, leads to the formation of supramolecular chains along the *b* axis, Fig. 2.

S2. Experimental

To 3,4-dimethyl aniline (484 mg, 4 mmol) in distilled water (10 ml) was added benzene sulfonyl chloride (510 ml, 4 mmol) with stirring at room temperature while maintaining the pH of the reaction mixture at 8 using 3% sodium carbonate. The progress of the reaction was monitored by TLC. The precipitate formed was washed with water, dried and crystallized from a methanol/ethyl acetate mixture (50:50 V/V) to yield colourless prisms of (I); m. pt 414 K.

S3. Refinement

The C-bound H atoms were geometrically placed (C–H = 0.93–0.96 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$. The N-bound H atom was refined with the distance restraint N–H = 0.86±0.01 Å, and with $U_{iso}(H) = 1.2U_{eq}(N)$. High thermal motion was noted for several atoms in the S-bound benzene ring but multiple positions were not resolved. The anisotropic displacement parameters for this ring were refined with the ISOR command to constrain these to be approximately isotropic.

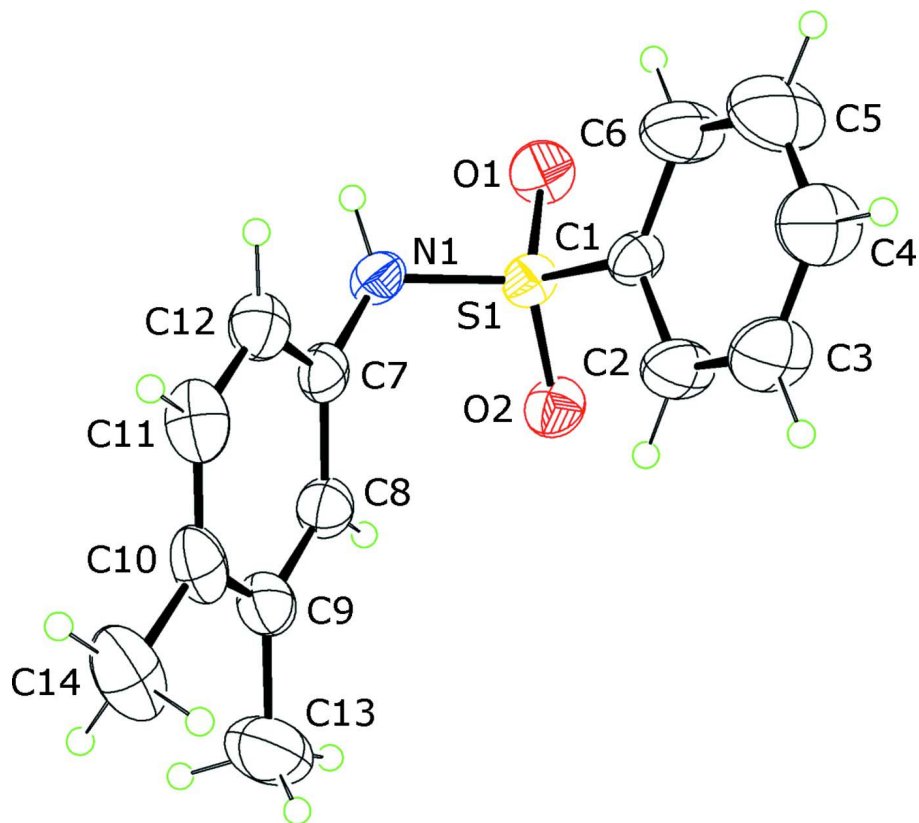


Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 35% probability level.

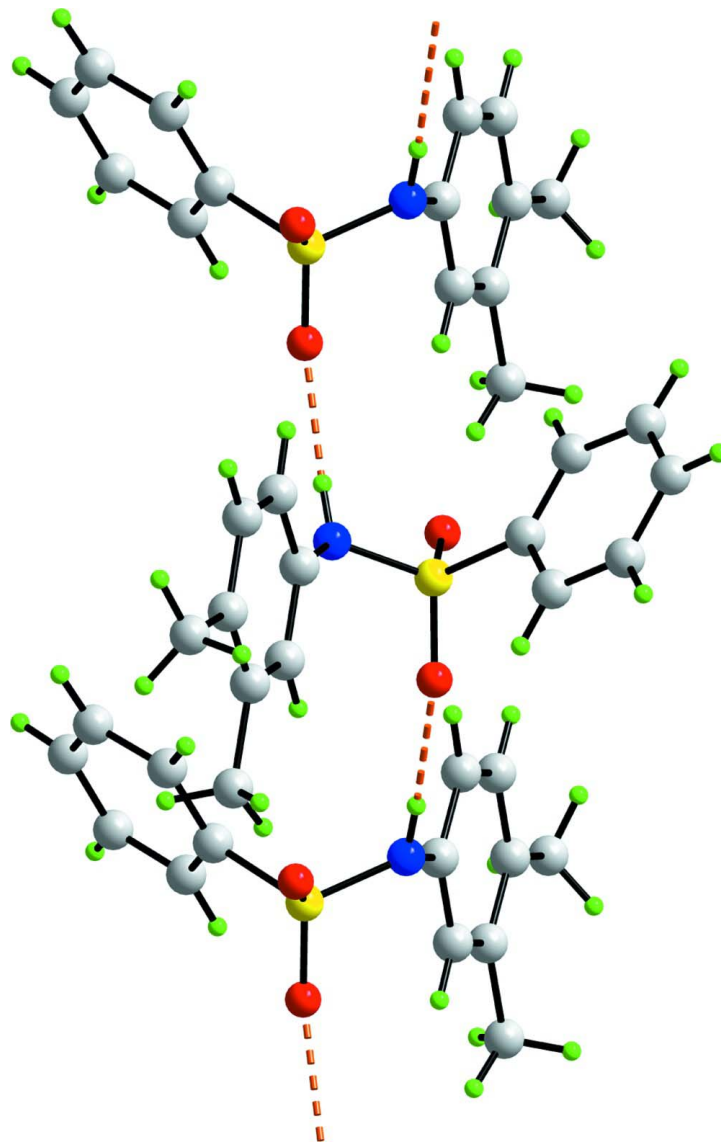


Figure 2

A view of the supramolecular chain mediated by N–H···O hydrogen bonding (orange dashed lines) in (I). Colour code: S, yellow; O, red; N, blue; C, grey; and H, green.

***N*-(3,4-Dimethylphenyl)benzenesulfonamide**

Crystal data

$C_{14}H_{15}NO_2S$

$M_r = 261.33$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 24.2129 (12) \text{ \AA}$

$b = 9.2616 (5) \text{ \AA}$

$c = 16.5584 (16) \text{ \AA}$

$\beta = 132.014 (2)^\circ$

$V = 2758.9 (3) \text{ \AA}^3$

$Z = 8$

$F(000) = 1104$

$D_x = 1.258 \text{ Mg m}^{-3}$

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

Cell parameters from 2975 reflections

$\theta = 2.5\text{--}24.2^\circ$

$\mu = 0.23 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prism, colourless

$0.31 \times 0.08 \times 0.07 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.795$, $T_{\max} = 0.947$

11486 measured reflections
2864 independent reflections
2112 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$
 $\theta_{\max} = 26.5^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -30 \rightarrow 30$
 $k = -11 \rightarrow 11$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.196$
 $S = 1.09$
2864 reflections
168 parameters
37 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
 $w = 1/[\sigma^2(F_o^2) + (0.1221P)^2 + 0.6451P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.56 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.21969 (4)	0.08206 (8)	0.26474 (6)	0.0437 (3)
O1	0.16507 (13)	0.0323 (3)	0.15598 (16)	0.0610 (6)
O2	0.22892 (12)	0.2337 (2)	0.28664 (18)	0.0567 (6)
N1	0.29812 (14)	0.0196 (3)	0.3083 (2)	0.0492 (6)
H1n	0.291 (2)	-0.063 (2)	0.280 (3)	0.059*
C1	0.20212 (16)	0.0052 (3)	0.3427 (2)	0.0448 (7)
C2	0.2297 (2)	0.0696 (4)	0.4375 (3)	0.0690 (10)
H2	0.2586	0.1526	0.4613	0.083*
C3	0.2149 (3)	0.0117 (5)	0.4979 (3)	0.0875 (13)
H3	0.2337	0.0557	0.5625	0.105*
C4	0.1730 (3)	-0.1089 (5)	0.4635 (4)	0.0935 (14)
H4	0.1643	-0.1497	0.5054	0.112*
C5	0.1437 (4)	-0.1704 (6)	0.3671 (5)	0.1114 (18)
H5	0.1129	-0.2507	0.3418	0.134*
C6	0.1591 (3)	-0.1147 (5)	0.3065 (4)	0.0851 (13)

H6	0.1403	-0.1588	0.2420	0.102*
C7	0.36691 (17)	0.0393 (3)	0.4183 (2)	0.0470 (7)
C8	0.39442 (19)	0.1756 (3)	0.4603 (3)	0.0537 (8)
H8	0.3673	0.2562	0.4178	0.064*
C9	0.46209 (19)	0.1938 (4)	0.5652 (3)	0.0607 (9)
C10	0.50388 (19)	0.0737 (4)	0.6287 (3)	0.0638 (10)
C11	0.4750 (2)	-0.0623 (4)	0.5834 (3)	0.0683 (10)
H11	0.5022	-0.1436	0.6246	0.082*
C12	0.4080 (2)	-0.0805 (4)	0.4804 (3)	0.0581 (8)
H12	0.3902	-0.1728	0.4523	0.070*
C13	0.4905 (3)	0.3462 (5)	0.6065 (4)	0.1030 (16)
H13A	0.5075	0.3559	0.6778	0.154*
H13B	0.4511	0.4139	0.5581	0.154*
H13C	0.5309	0.3653	0.6096	0.154*
C14	0.5778 (2)	0.0900 (6)	0.7430 (3)	0.0921 (15)
H14A	0.6053	0.1672	0.7454	0.138*
H14B	0.6054	0.0017	0.7654	0.138*
H14C	0.5697	0.1115	0.7912	0.138*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0524 (5)	0.0421 (5)	0.0424 (4)	0.0023 (3)	0.0340 (4)	0.0028 (3)
O1	0.0623 (14)	0.0769 (16)	0.0388 (12)	-0.0017 (12)	0.0317 (11)	-0.0011 (10)
O2	0.0678 (14)	0.0410 (12)	0.0680 (14)	0.0076 (10)	0.0483 (13)	0.0091 (10)
N1	0.0572 (15)	0.0462 (14)	0.0533 (15)	-0.0004 (12)	0.0407 (13)	-0.0075 (12)
C1	0.0560 (16)	0.0416 (15)	0.0466 (15)	-0.0004 (12)	0.0384 (14)	-0.0012 (12)
C2	0.089 (2)	0.072 (2)	0.0622 (19)	-0.0237 (18)	0.0574 (19)	-0.0184 (16)
C3	0.118 (3)	0.102 (3)	0.071 (2)	-0.024 (2)	0.075 (2)	-0.019 (2)
C4	0.132 (3)	0.098 (3)	0.091 (3)	-0.025 (3)	0.092 (3)	-0.005 (2)
C5	0.155 (4)	0.101 (3)	0.118 (3)	-0.054 (3)	0.108 (3)	-0.021 (2)
C6	0.121 (3)	0.079 (2)	0.084 (2)	-0.038 (2)	0.080 (2)	-0.026 (2)
C7	0.0513 (16)	0.0521 (17)	0.0538 (17)	0.0004 (13)	0.0418 (15)	0.0004 (14)
C8	0.0551 (18)	0.0536 (19)	0.0588 (19)	0.0010 (14)	0.0408 (17)	-0.0001 (14)
C9	0.0553 (19)	0.071 (2)	0.064 (2)	-0.0095 (17)	0.0436 (18)	-0.0090 (18)
C10	0.0481 (19)	0.094 (3)	0.058 (2)	-0.0018 (18)	0.0389 (17)	0.0061 (19)
C11	0.062 (2)	0.075 (3)	0.074 (2)	0.0142 (18)	0.048 (2)	0.0238 (19)
C12	0.061 (2)	0.0554 (19)	0.067 (2)	0.0035 (15)	0.0462 (18)	0.0068 (16)
C13	0.078 (3)	0.098 (4)	0.091 (3)	-0.020 (3)	0.039 (3)	-0.022 (3)
C14	0.058 (2)	0.142 (4)	0.068 (3)	-0.008 (2)	0.038 (2)	0.011 (3)

Geometric parameters (Å, °)

S1—O1	1.420 (2)	C7—C8	1.379 (4)
S1—O2	1.430 (2)	C7—C12	1.381 (4)
S1—N1	1.621 (3)	C8—C9	1.386 (5)
S1—C1	1.758 (3)	C8—H8	0.9300
N1—C7	1.436 (4)	C9—C10	1.394 (5)

N1—H1n	0.85 (3)	C9—C13	1.518 (6)
C1—C6	1.356 (5)	C10—C11	1.392 (5)
C1—C2	1.364 (4)	C10—C14	1.508 (5)
C2—C3	1.379 (5)	C11—C12	1.365 (5)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.351 (6)	C12—H12	0.9300
C3—H3	0.9300	C13—H13A	0.9600
C4—C5	1.363 (6)	C13—H13B	0.9600
C4—H4	0.9300	C13—H13C	0.9600
C5—C6	1.384 (6)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C6—H6	0.9300	C14—H14C	0.9600
O1—S1—O2	119.67 (15)	C12—C7—N1	119.2 (3)
O1—S1—N1	105.54 (14)	C7—C8—C9	120.7 (3)
O2—S1—N1	107.46 (13)	C7—C8—H8	119.7
O1—S1—C1	108.80 (14)	C9—C8—H8	119.7
O2—S1—C1	106.57 (13)	C8—C9—C10	120.1 (3)
N1—S1—C1	108.39 (14)	C8—C9—C13	118.5 (4)
C7—N1—S1	122.54 (19)	C10—C9—C13	121.4 (4)
C7—N1—H1N	117 (2)	C11—C10—C9	117.8 (3)
S1—N1—H1N	109 (2)	C11—C10—C14	120.9 (4)
C6—C1—C2	120.5 (3)	C9—C10—C14	121.3 (4)
C6—C1—S1	119.9 (2)	C12—C11—C10	122.2 (3)
C2—C1—S1	119.6 (2)	C12—C11—H11	118.9
C1—C2—C3	120.0 (4)	C10—C11—H11	118.9
C1—C2—H2	120.0	C11—C12—C7	119.5 (3)
C3—C2—H2	120.0	C11—C12—H12	120.2
C4—C3—C2	120.1 (4)	C7—C12—H12	120.2
C4—C3—H3	120.0	C9—C13—H13A	109.5
C2—C3—H3	120.0	C9—C13—H13B	109.5
C3—C4—C5	119.7 (4)	H13A—C13—H13B	109.5
C3—C4—H4	120.1	C9—C13—H13C	109.5
C5—C4—H4	120.1	H13A—C13—H13C	109.5
C4—C5—C6	120.7 (4)	H13B—C13—H13C	109.5
C4—C5—H5	119.7	C10—C14—H14A	109.5
C6—C5—H5	119.7	C10—C14—H14B	109.5
C1—C6—C5	119.0 (4)	H14A—C14—H14B	109.5
C1—C6—H6	120.5	C10—C14—H14C	109.5
C5—C6—H6	120.5	H14A—C14—H14C	109.5
C8—C7—C12	119.7 (3)	H14B—C14—H14C	109.5
C8—C7—N1	121.0 (3)		
O1—S1—N1—C7	-176.1 (2)	C4—C5—C6—C1	-2.1 (9)
O2—S1—N1—C7	55.1 (3)	S1—N1—C7—C8	-60.8 (4)
C1—S1—N1—C7	-59.7 (3)	S1—N1—C7—C12	123.0 (3)
O1—S1—C1—C6	19.3 (4)	C12—C7—C8—C9	-1.6 (4)
O2—S1—C1—C6	149.6 (3)	N1—C7—C8—C9	-177.7 (3)

N1—S1—C1—C6	-95.0 (4)	C7—C8—C9—C10	1.4 (5)
O1—S1—C1—C2	-158.3 (3)	C7—C8—C9—C13	178.7 (3)
O2—S1—C1—C2	-28.0 (3)	C8—C9—C10—C11	-0.5 (5)
N1—S1—C1—C2	87.4 (3)	C13—C9—C10—C11	-177.8 (4)
C6—C1—C2—C3	0.9 (6)	C8—C9—C10—C14	179.9 (3)
S1—C1—C2—C3	178.6 (3)	C13—C9—C10—C14	2.6 (5)
C1—C2—C3—C4	0.1 (7)	C9—C10—C11—C12	-0.2 (5)
C2—C3—C4—C5	-2.1 (9)	C14—C10—C11—C12	179.4 (3)
C3—C4—C5—C6	3.1 (10)	C10—C11—C12—C7	-0.1 (5)
C2—C1—C6—C5	0.1 (7)	C8—C7—C12—C11	0.9 (5)
S1—C1—C6—C5	-177.6 (4)	N1—C7—C12—C11	177.1 (3)

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 n ...O2 ⁱ	0.85 (3)	2.07 (2)	2.921 (3)	177 (5)

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