organic compounds

N-(4-Methoxybenzoyl)benzenesulfon-amide

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In the title compound, $C_{14}H_{13}NO_4S$, the dihedral angle between the aromatic rings is 69.81 (1)°; the dihedral angle between the planes defined by the S—N—C=O segment of the central chain and the sulfonyl benzene ring is 74.91 (1)°. In the crystal, the molecules are linked by weak N—H···O hydrogen bonds into $C(4)$ chains running along [100]. The molecules in adjacent chains are linked by weak C—H···O interactions, generating $R_2^2(16)$ dimeric pairs. Weak C—H···π interactions connect the double chains into (001) sheets.

Related literature

For similar structures, see: Gowda et al. (2009); Suchetan et al. (2009, 2010); Sreenivasa et al. (2013).

Experimental

Crystal data

$C_{14}H_{13}NO_4S$	$b = 10.6343$ (10) Å
$M_r = 291.31$	$c = 11.9139$ (11) Å
Triclinic, $P\bar{1}$	$\alpha = 89.792$ (3)°
$a = 5.3059$ (5) Å	$\beta = 87.392$ (3)°
$\gamma = 83.944$ (3)°	$\mu = 2.28$ mm^{-1}
$V = 667.79$ (11) Å^3	$T = 293$ K
$Z = 2$	$0.37 \times 0.26 \times 0.20$ mm
Cu $K\alpha$ radiation	

Data collection

Bruker APEXII diffractometer	8561 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2009)	2168 independent reflections
$T_{min} = 0.515$, $T_{max} = 0.633$	2076 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.068$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.158$	
$S = 1.13$	
2168 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
186 parameters	$\Delta\rho_{min} = -0.84$ e Å^{-3}
1 restraint	

Table 1
Hydrogen-bond geometry (Å, °).

Cg is the centroid of the methoxybenzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H$N1$···O1^i	0.83 (3)	2.41 (3)	3.1662 (3)	153
C12—H12···O2^{ii}	0.93	2.58	3.286 (3)	133
C4—H4···Cg^{iii}	0.93	2.90	3.7396	150

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

Data collection: APEX2 (Bruker, 2009); cell refinement: APEX2 and SAINT-Plus (Bruker, 2009); data reduction: SAINT-Plus and XPREP (Bruker, 2009); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae et al., 2008); software used to prepare material for publication: SHELXL97.

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N-(4-Methoxybenzoyl)benzenesulfonamide

S. Sreenivasa, M. S. Nanjundaswamy, S. Madankumar, N. K. Lokanath, E. Suresha and P. A. Suchetan

S1. Introduction

As a part of our continued efforts to study the crystal structures of N-(aroyl)-arylsulfonamides (Suchetan *et al.*, 2009, 2010; Sreenivasa *et al.*, 2013; Gowda *et al.*, 2009), we report here the crystal structure of the title compound (I) (Fig 1).

S2. Experimental

S2.1. Synthesis and crystallization

The title compound (I) was prepared by refluxing a mixture of 4-methoxybenzoic acid, benzenesulfonamide and phosphorous oxychloride (POCl$_3$) for 2 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid obtained was filtered and washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. The compound obtained was filtered and later dried (Melting point: 399 K).

Colorless prisms of (I) were obtained from a slow evaporation of its aqueous methanolic solution at room temperature.

S2.2. Refinement

The H atom of the NH group was located in a difference map and later restained to N—H = 0.86 (4) Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93-0.96 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2-1.5 times of the U eq of the parent atom).

S3. Results and discussion

In (I), the dihedral angle between the aromatic rings is 69.81 (1)°. Compared to this, the dihedral angle is 80.3 (1)° in N-(benzoyl)-benzenesulfonamide (II) (Gowda *et al.*, 2009), 68.6 (1)° in N-(4-chlorobenzoyl)-benzenesulfonamide (III) (Suchetan *et al.*, 2009), 71.9 (1)° in N-(4-methylbenzoyl)-benzenesulfonamide (IV) (Suchetan *et al.*, 2010) and 78.62 (16)° in N-(4-methoxybenzoyl)-4-methylbenzenesulfonamide (V) (Sreenivasa *et al.*, 2013). This shows that introducing a substituent into the *para* position of the benzoyl ring correlates with a decrease of the dihedral angle between the aromatic rings. Further, the molecule is twisted at the S atom, the dihedral angle between the planes defined by the S—N—C=O segment in the central chain and the sulfonyl benzene ring being 74.91 (1)°.

In the crystal, both strong classical N—H⋯O hydrogen bonds and weaker C—H⋯O and C—H⋯Cg interactions are observed. The molecules are linked to one another through strong N1—HN1⋯O1 hydrogen bonds into C(4) chains running along [100]. The molecules in the adjacent chains are linked through weak C12—H12⋯O2 interactions, generating R$_2^2$(16) dimeric pairs. Combination of the N1—HN1⋯O1 linked chains and C12—H12⋯O2 linked dimers form columns along *a* axis (Figure 2 & 3). The molecules are further connected into C(10) chains running along [010] through C4—H4⋯Cg interactions (Figure 4) resulting in a two dimensional architecture.
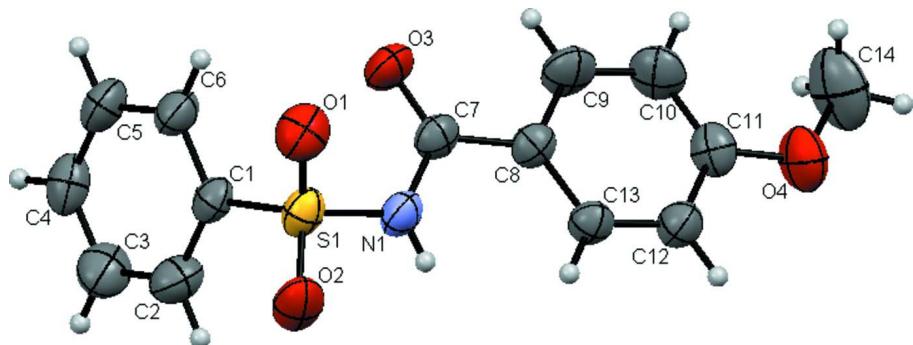
Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.
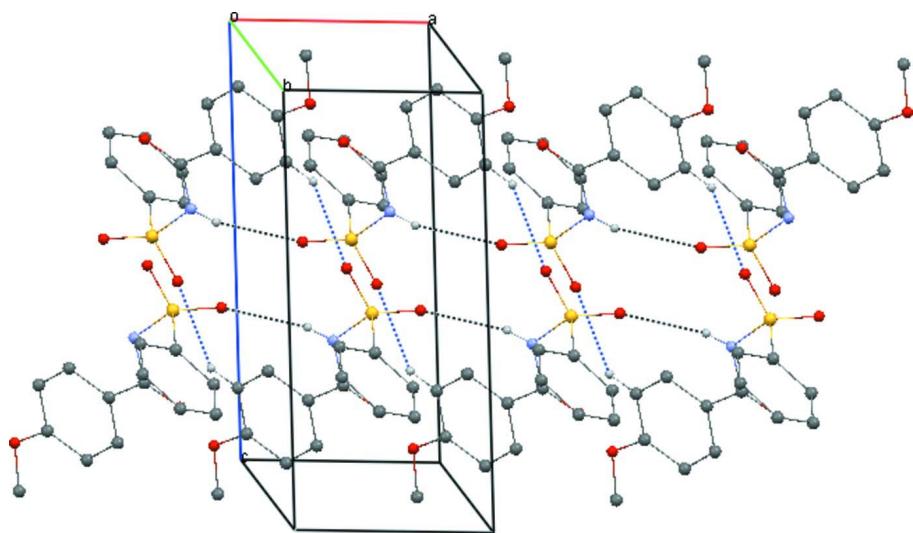
Figure 2

Columns generated from the combination of N1—HN1···O1 linked chains and C12—H12···O2 linked dimers in (I) when viewed along a *axis*. H-atoms not involved in H-bonds are omitted for clarity purpose.
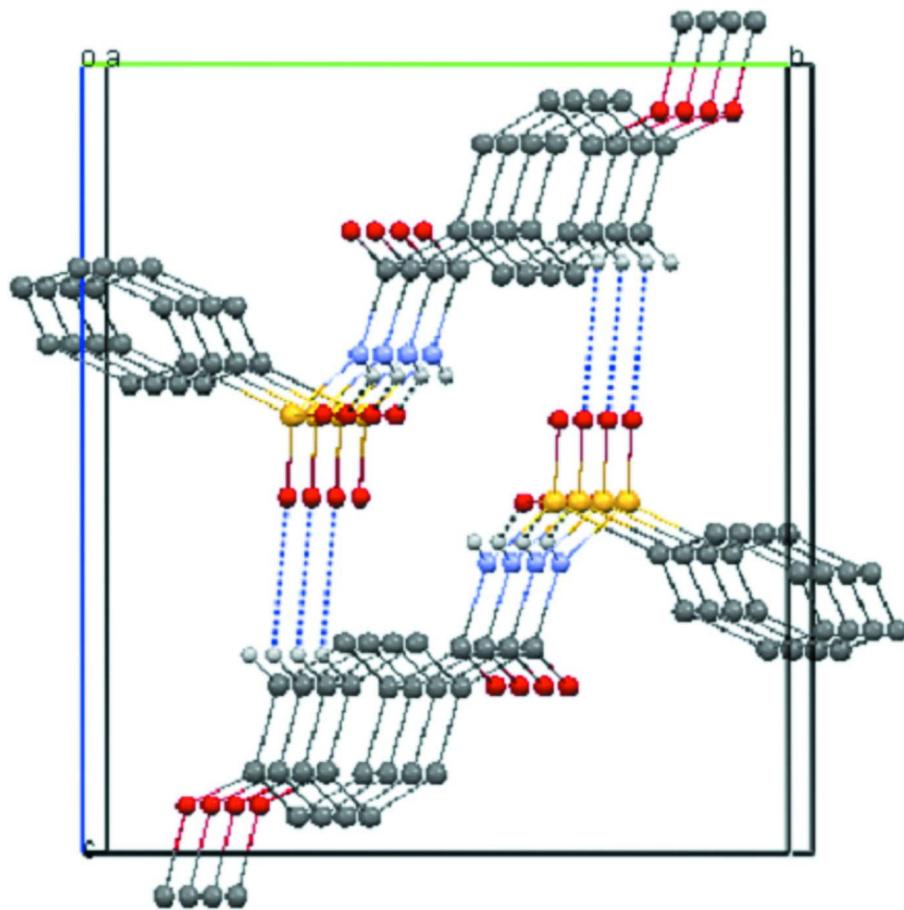
Figure 3

Columns generated from the combination of N1—HN1···O1 linked chains and C12—H12···O2 linked dimers in (I) when viewed down a *axis*. H-atoms not involved in H-bonds are omitted for clarity purpose.
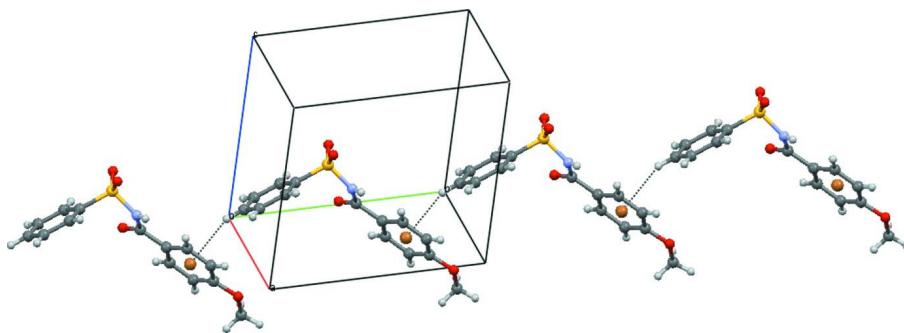
Figure 4

C4—H4···*Cg* interaction observed in the structure of (I). *Cg* is the centroid of the methoxyphenyl ring.

N-(4-Methoxybenzoyl)benzenesulfonamide

Crystal data

$C_{14}H_{13}NO_4S$	Hall symbol: -P 1
$M_r = 291.31$	$a = 5.3059$ (5) Å
Triclinic, $P\bar{1}$	$b = 10.6343$ (10) Å

$c = 11.9139\ (11)$ Å
$\alpha = 89.792\ (3)°$
$\beta = 87.392\ (3)°$
$\gamma = 83.944\ (3)°$
$V = 667.79\ (11)$ Å^3
$Z = 2$
$F(000) = 304$
Prism
$D_x = 1.449$ Mg m^{-3}

Melting point: 399 K
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 3.7–64.8°$
$\mu = 2.28$ mm^{-1}
$T = 293$ K
Prism, colourless
$0.37 \times 0.26 \times 0.20$ mm

Data collection

Bruker APEXII
   diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan
   (*SADABS*; Bruker, 2009)
$T_{min} = 0.515$, $T_{max} = 0.633$
8561 measured reflections

2168 independent reflections
2076 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 64.8°$, $\theta_{min} = 3.7°$
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$
3 standard reflections every 120 min
   intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.158$
$S = 1.13$
2168 reflections
186 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.1013P)^2 + 0.1754P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.84$ e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.2427 (3)	0.36646 (17)	0.44504 (16)	0.0596 (5)
N1	0.6766 (4)	0.40318 (19)	0.36803 (17)	0.0478 (5)
O3	0.4534 (3)	0.39943 (16)	0.21164 (15)	0.0559 (5)
O4	1.1816 (4)	0.81223 (19)	0.06023 (17)	0.0695 (6)
C13	0.9563 (4)	0.5966 (2)	0.26610 (18)	0.0421 (5)
H13	0.9898	0.5737	0.3398	0.051*

C7	0.6197 (4)	0.4435 (2)	0.25993 (19)	0.0424 (5)
O2	0.6259 (4)	0.30297 (18)	0.54898 (15)	0.0624 (5)
C9	0.7269 (6)	0.5777 (3)	0.1010 (2)	0.0609 (7)
H9	0.6030	0.5417	0.0631	0.073*
C1	0.5310 (4)	0.1655 (2)	0.37956 (18)	0.0405 (5)
C12	1.0878 (4)	0.6857 (2)	0.2130 (2)	0.0475 (6)
H12	1.2101	0.7227	0.2512	0.057*
C8	0.7732 (4)	0.5405 (2)	0.21008 (18)	0.0402 (5)
C11	1.0410 (5)	0.7213 (2)	0.1032 (2)	0.0472 (6)
C5	0.3772 (5)	0.0164 (2)	0.2584 (2)	0.0564 (6)
H5	0.2579	−0.0048	0.2089	0.068*
C3	0.7531 (5)	−0.0405 (3)	0.3552 (3)	0.0649 (8)
H3	0.8890	−0.0996	0.3709	0.078*
C4	0.5776 (5)	−0.0707 (2)	0.2829 (2)	0.0547 (6)
H4	0.5930	−0.1504	0.2500	0.066*
C2	0.7307 (5)	0.0778 (3)	0.4055 (2)	0.0574 (6)
H2	0.8487	0.0979	0.4562	0.069*
C14	1.1590 (10)	0.8451 (4)	−0.0546 (3)	0.0986 (13)
H14A	1.2092	0.7720	−0.1005	0.148*
H14B	1.2665	0.9100	−0.0734	0.148*
H14C	0.9860	0.8757	−0.0676	0.148*
C6	0.3516 (4)	0.1360 (2)	0.3071 (2)	0.0498 (6)
H6	0.2157	0.1948	0.2910	0.060*
C10	0.8596 (6)	0.6669 (3)	0.0467 (2)	0.0635 (7)
H10	0.8267	0.6899	−0.0270	0.076*
S1	0.50162 (10)	0.31426 (5)	0.44550 (4)	0.0452 (3)
HN1	0.808 (6)	0.419 (4)	0.397 (3)	0.091 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0540 (10)	0.0592 (10)	0.0642 (11)	−0.0033 (8)	0.0092 (8)	−0.0138 (8)
N1	0.0543 (12)	0.0501 (11)	0.0432 (11)	−0.0222 (9)	−0.0093 (9)	0.0000 (8)
O3	0.0563 (10)	0.0581 (10)	0.0573 (10)	−0.0197 (8)	−0.0149 (8)	−0.0071 (8)
O4	0.0829 (14)	0.0677 (12)	0.0582 (12)	−0.0183 (10)	0.0158 (10)	0.0140 (9)
C13	0.0460 (12)	0.0463 (11)	0.0348 (11)	−0.0079 (9)	−0.0035 (9)	−0.0026 (9)
C7	0.0460 (12)	0.0400 (11)	0.0419 (12)	−0.0065 (9)	−0.0039 (9)	−0.0105 (9)
O2	0.0851 (13)	0.0689 (11)	0.0385 (9)	−0.0294 (10)	−0.0111 (9)	−0.0024 (8)
C9	0.0753 (18)	0.0729 (17)	0.0393 (13)	−0.0241 (13)	−0.0155 (12)	−0.0048 (11)
C1	0.0383 (11)	0.0445 (11)	0.0403 (11)	−0.0142 (8)	0.0036 (8)	−0.0022 (8)
C12	0.0477 (12)	0.0519 (13)	0.0445 (12)	−0.0126 (10)	−0.0019 (9)	−0.0006 (10)
C8	0.0427 (11)	0.0417 (11)	0.0361 (11)	−0.0047 (9)	0.0001 (9)	−0.0081 (8)
C11	0.0523 (13)	0.0460 (12)	0.0418 (12)	−0.0035 (10)	0.0118 (10)	−0.0010 (9)
C5	0.0568 (14)	0.0537 (14)	0.0624 (16)	−0.0215 (11)	−0.0058 (11)	−0.0110 (11)
C3	0.0542 (15)	0.0558 (15)	0.083 (2)	0.0029 (12)	−0.0004 (14)	−0.0073 (13)
C4	0.0572 (14)	0.0445 (12)	0.0628 (16)	−0.0153 (10)	0.0159 (12)	−0.0095 (10)
C2	0.0450 (13)	0.0652 (15)	0.0628 (15)	−0.0071 (11)	−0.0084 (11)	−0.0083 (12)
C14	0.147 (4)	0.091 (2)	0.0561 (19)	−0.018 (2)	0.032 (2)	0.0156 (17)

C6	0.0449 (12)	0.0469 (12)	0.0598 (14)	−0.0128 (9)	−0.0081 (10)	−0.0050 (10)
C10	0.084 (2)	0.0740 (17)	0.0338 (12)	−0.0134 (14)	−0.0043 (12)	0.0032 (11)
S1	0.0516 (4)	0.0468 (4)	0.0393 (4)	−0.0156 (3)	0.0011 (3)	−0.0074 (2)

Geometric parameters (Å, º)

O1—S1	1.4264 (19)	C1—S1	1.758 (2)
N1—C7	1.392 (3)	C12—C11	1.387 (3)
N1—S1	1.646 (2)	C12—H12	0.9300
N1—HN1	0.82 (3)	C11—C10	1.376 (4)
O3—C7	1.211 (3)	C5—C4	1.376 (4)
O4—C11	1.367 (3)	C5—C6	1.390 (4)
O4—C14	1.418 (4)	C5—H5	0.9300
C13—C12	1.371 (3)	C3—C4	1.360 (4)
C13—C8	1.389 (3)	C3—C2	1.388 (4)
C13—H13	0.9300	C3—H3	0.9300
C7—C8	1.486 (3)	C4—H4	0.9300
O2—S1	1.4232 (19)	C2—H2	0.9300
C9—C10	1.381 (4)	C14—H14A	0.9600
C9—C8	1.382 (3)	C14—H14B	0.9600
C9—H9	0.9300	C14—H14C	0.9600
C1—C6	1.375 (3)	C6—H6	0.9300
C1—C2	1.381 (4)	C10—H10	0.9300
C7—N1—S1	123.91 (17)	C4—C3—C2	120.4 (2)
C7—N1—HN1	122 (3)	C4—C3—H3	119.8
S1—N1—HN1	114 (3)	C2—C3—H3	119.8
C11—O4—C14	118.2 (3)	C3—C4—C5	120.2 (2)
C12—C13—C8	120.1 (2)	C3—C4—H4	119.9
C12—C13—H13	119.9	C5—C4—H4	119.9
C8—C13—H13	119.9	C1—C2—C3	119.2 (2)
O3—C7—N1	120.0 (2)	C1—C2—H2	120.4
O3—C7—C8	123.6 (2)	C3—C2—H2	120.4
N1—C7—C8	116.42 (19)	O4—C14—H14A	109.5
C10—C9—C8	121.9 (2)	O4—C14—H14B	109.5
C10—C9—H9	119.1	H14A—C14—H14B	109.5
C8—C9—H9	119.1	O4—C14—H14C	109.5
C6—C1—C2	121.0 (2)	H14A—C14—H14C	109.5
C6—C1—S1	120.16 (18)	H14B—C14—H14C	109.5
C2—C1—S1	118.81 (18)	C1—C6—C5	118.7 (2)
C13—C12—C11	120.9 (2)	C1—C6—H6	120.6
C13—C12—H12	119.5	C5—C6—H6	120.6
C11—C12—H12	119.5	C11—C10—C9	119.1 (2)
C9—C8—C13	118.3 (2)	C11—C10—H10	120.4
C9—C8—C7	117.3 (2)	C9—C10—H10	120.4
C13—C8—C7	124.4 (2)	O2—S1—O1	119.44 (12)
O4—C11—C10	125.0 (2)	O2—S1—N1	103.74 (11)
O4—C11—C12	115.3 (2)	O1—S1—N1	109.21 (11)

C10—C11—C12	119.6 (2)	O2—S1—C1	108.88 (11)
C4—C5—C6	120.5 (2)	O1—S1—C1	108.65 (10)
C4—C5—H5	119.7	N1—S1—C1	106.11 (10)
C6—C5—H5	119.7		
S1—N1—C7—O3	−10.3 (3)	S1—C1—C2—C3	179.0 (2)
S1—N1—C7—C8	169.94 (15)	C4—C3—C2—C1	−1.4 (4)
C8—C13—C12—C11	0.0 (3)	C2—C1—C6—C5	−1.2 (4)
C10—C9—C8—C13	1.0 (4)	S1—C1—C6—C5	−178.49 (18)
C10—C9—C8—C7	−179.8 (2)	C4—C5—C6—C1	0.4 (4)
C12—C13—C8—C9	−0.6 (3)	O4—C11—C10—C9	−178.6 (2)
C12—C13—C8—C7	−179.8 (2)	C12—C11—C10—C9	0.1 (4)
O3—C7—C8—C9	−2.4 (3)	C8—C9—C10—C11	−0.7 (4)
N1—C7—C8—C9	177.3 (2)	C7—N1—S1—O2	−177.30 (19)
O3—C7—C8—C13	176.8 (2)	C7—N1—S1—O1	−48.9 (2)
N1—C7—C8—C13	−3.5 (3)	C7—N1—S1—C1	68.0 (2)
C14—O4—C11—C10	−6.6 (4)	C6—C1—S1—O2	153.0 (2)
C14—O4—C11—C12	174.7 (3)	C2—C1—S1—O2	−24.3 (2)
C13—C12—C11—O4	179.0 (2)	C6—C1—S1—O1	21.4 (2)
C13—C12—C11—C10	0.2 (4)	C2—C1—S1—O1	−155.9 (2)
C2—C3—C4—C5	0.6 (4)	C6—C1—S1—N1	−95.9 (2)
C6—C5—C4—C3	−0.1 (4)	C2—C1—S1—N1	86.8 (2)
C6—C1—C2—C3	1.7 (4)		

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the methoxybenzene ring.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H*N*1⋯O1[i]	0.83 (3)	2.41 (3)	3.1662 (3)	153
C12—H12⋯O2[ii]	0.93	2.58	3.286 (3)	133
C4—H4⋯*Cg*[iii]	0.93	2.90	3.7396	150

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) *x*, *y*−1, *z*.