Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Monoclinic pseudosymmetry in 2-phenoxybenzenesulfonamide, a triclinic structure having $Z' = 4$, and spontaneous resolution in monoclinic *N*-methyl-2-phenoxybenzenesulfonamide

Christopher Glidewell,[a]* John N. Low,[b] Janet M. S. Skakle[b] and James L. Wardell[c]

[a]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland, [b]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and [c]Instituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, 21945-970 Rio de Janeiro, RJ, Brazil
Correspondence e-mail: cg@st-andrews.ac.uk

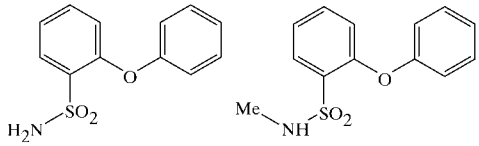
Received 22 March 2004
Accepted 26 March 2004
Online 30 April 2004

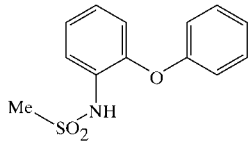
2-Phenoxybenzenesulfonamide, $C_{12}H_{11}NO_3S$, (I), crystallizes in space group $P\overline{1}$ with $Z' = 4$, but the structure closely mimics the monoclinic space group $P2_1/b$ with $Z' = 2$. The molecules of (I) are linked by a combination of N—H···O and C—H···O hydrogen bonds into two independent chains of centrosymmetric edge-fused $R_2^2(18)$ and $R_6^6(34)$ rings. *N*-Methyl-2-phenoxybenzenesulfonamide, $C_{13}H_{13}NO_3S$, (II), crystallizes in space group $P2_1$ with $Z' = 1$, and is an example of spontaneous resolution. The molecules are linked by N—H···O and C—H···O hydrogen bonds into chains of spiro-fused $R_2^2(12)$ rings, and these chains are linked into sheets by a single C—H···π(arene) hydrogen bond.

Comment

We report here the structure of two closely related sulfonamides, namely 2-phenoxybenzenesulfonamide, (I), and its *N*-methyl analogue, (II), which both show interesting crystallization characteristics.

Compound (I) crystallizes in space group $P\overline{1}$ with four independent molecules in the asymmetric unit (Fig. 1). The choice of the asymmetric unit in cases where $Z' > 1$ allows some flexibility, but for (I) the asymmetric unit has been selected such that the molecules labelled *A* and *C* act as hydrogen-bond donors to molecules *B* and *D*, respectively, within the asymmetric unit. The bond lengths and angles present no unusual values, but the orientation of the sulfonamide groups relative to the adjacent arene rings is very similar in all four molecules (Table 1). These orientations are probably controlled in part by the intramolecular N—H···O hydrogen bonds (Table 2), each of which generates an $S(6)$ motif (Bernstein *et al.*, 1995).





The overall molecular conformations, which can be defined in terms of five independent torsion angles for each molecule, indicate the occurrence of pseudosymmetry. The values of these torsion angles (Table 1) show that for the selected asymmetric unit, molecules *A* and *D* form an approximately enantiomorphous pair and molecules *B* and *C* form a second such pair independent of the first. Detailed scrutiny of the atomic coordinates shows that those for molecule *D* are approximately related to those for molecule *A* by the transformation $(x-1, y-\frac{1}{2}, \frac{3}{2}-z)$, while those of molecule *B* are similarly related to those of molecule *C* by the related transformation $(x, y-\frac{1}{2}, \frac{3}{2}-z)$. Overall, therefore, there is a pseudo *b*-glide plane at $z = \frac{3}{4}$. The unit-cell dimensions rule out any symmetry higher than triclinic, and the absence of any additional symmetry was confirmed by examination of the refined structure using *PLATON* (Spek, 2003). However, the structure exhibits a close mimicry of space group $P2_1/b$, an

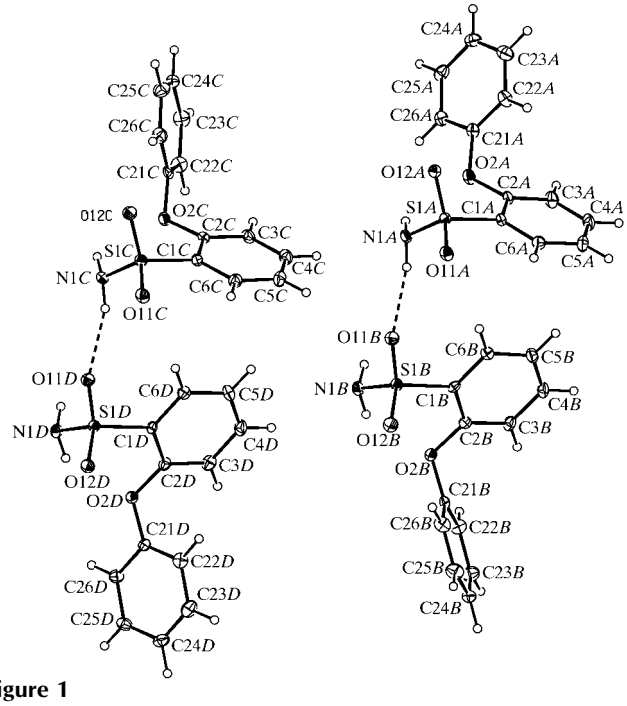


Figure 1
The four independent molecules in (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

  DOI: 10.1107/S010827010400719X

alternative to the conventional setting, $P2_1/c$, of space group No. 14. Consistent with this mimicry, the intensities of the $00l$ reflections are, in general, much weaker when l is odd than when l is even, although there is no obviously consistent pattern amongst the $hk0$ reflections.

The molecules of (I) are linked by a combination of N—H···O and C—H···O hydrogen bonds (Table 2) into two independent but very similar chains of edge-fused rings, one built from molecules of types *A* and *B* only, the other from molecules of types *C* and *D* only. It is necessary to discuss only one of these in any detail. Within the asymmetric unit, atom N1*A* acts as hydrogen-bond donor, *via* atom H11*A*, to atom O11*B*. In a similar manner, atom N1*B* at (x, y, z) acts as donor, *via* atom H11*B*, to atom O11*A* at $(x-1, y, z)$. In this manner, a $C_2^2(8)$ chain built from type *A* and *B* molecules and running parallel to the [100] direction is generated by translation. Within this chain, there is an intermolecular C—H···O contact, which is possibly more an adventitious contact consequent upon the N—H···O hydrogen bonds than a structurally significant hydrogen bond. Two antiparallel chains of *A* and *B* molecules, related to one another by inversion, run through each unit cell and these are linked by a single C—H···O hydrogen bond. Atom C25*B* at (x, y, z) acts as hydrogen-bond donor to atom O12*B* at $(1-x, 1-y, 1-z)$, and propagation of this interaction by translation and inversion generates a chain of centrosymmetric edge-fused rings along $(x, \frac{1}{2}, \frac{1}{2})$, with $R_2^2(18)$ rings centred at $(n+\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ (n = zero or integer) and $R_6^6(34)$ rings centred at $(n, \frac{1}{2}, \frac{1}{2})$ (n = zero or integer) (Fig. 2).

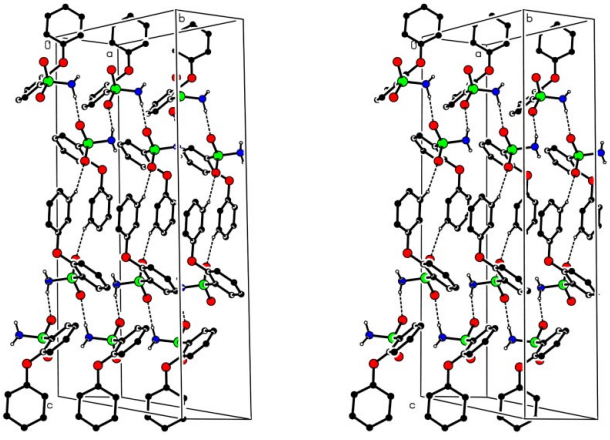


Figure 2
A stereoview of part of the crystal structure of (I), showing the formation of a [100] chain of edge-fused $R_2^2(18)$ and $R_6^6(34)$ rings built from molecules of types *A* and *B* only. For the sake of clarity, H atoms bonded to C atoms but not involved in the hydrogen-bonding motif shown have been omitted

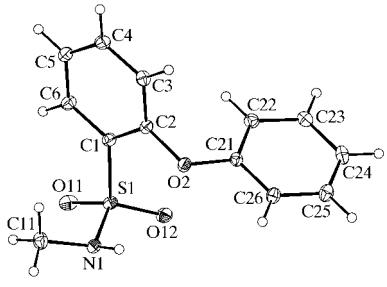


Figure 3
The molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The molecules of types *C* and *D* form an entirely similar chain of edge-fused rings running along the line $(x, 0, 1)$. Hence, in projection down *a*, there is a chain of *A* and *B* type molecules at the cell centre and chains of *C* and *D* type molecules at the cell vertices. However, there are no direction-specific interactions between adjacent chains. Despite the large number of independent aryl groups in the structure of (I), this contains neither C—H···π(arene) hydrogen bonds nor aromatic π–π stacking interactions.

The *N*-methyl analogue, (II) (Fig. 3), of (I) crystallizes in the chiral space group $P2_1$ with $Z' = 1$. The molecular conformation of (II), as judged from the leading torsion angles (Table 1) bears no particularly close resemblance to those in (I), and there are no intramolecular hydrogen bonds in (II). The resulting molecular point group is C_1, so that the molecules are chiral. Accordingly, each crystal of (II) contains only a single enantiomorph, in contrast with the crystals of (I). Since the bulk sample of (II) is racemic, the crystallization represents an example of spontaneous resolution to form a conglomerate, rather than a racemate as in (I).

The molecules of (II) are linked into sheets by a combination of three hydrogen bonds (Table 3) and it is convenient to analyse the sheet formation in terms of its two one-dimensional substructures. Atoms N1 and C26 in the molecule at (x, y, z) act as hydrogen-bond donors to, respectively, atoms O11 and O12, both in the molecule at $(1+x, y, z)$, so generating by translation a $C(4)C(8)[R_2^2(12)]$ chain of rings running parallel to the [100] direction (Fig. 4). In addition, atom C4 at (x, y, z) acts as hydrogen-bond donor to the monosubstituted ring C21–C26 in the molecule at $(x, 1+y, z)$, so generating by

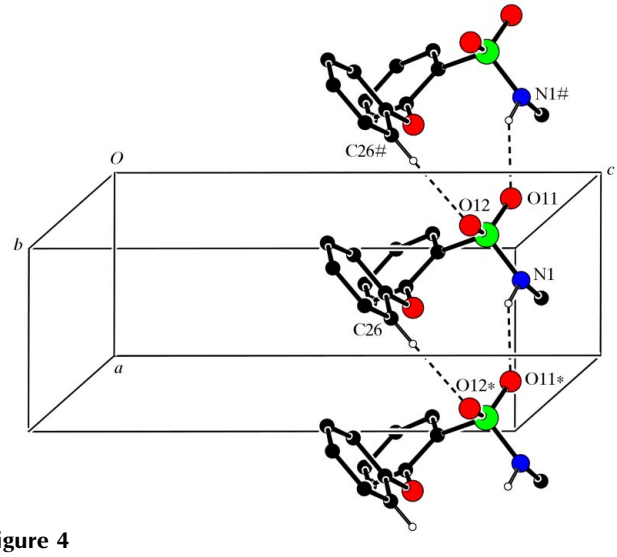


Figure 4
Part of the crystal structure of (II), showing the formation of a [100] chain of spiro-fused $R_2^2(12)$ rings. For the sake of clarity, H atoms bonded to C atoms but not involved in the hydrogen-bonding motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(1+x, y, z)$ and $(x-1, y, z)$, respectively.

translation a chain running parallel to the [010] direction (Fig. 5). The combination of the [100] and [010] chains generates an (001) sheet (Fig. 6). Two such sheets pass through each unit cell, one each in the domains $-0.02 < z < 0.49$ and $0.51 < z < 1.02$, but there are no direction-specific interactions between adjacent sheets.

The $C(4)$ chain motif in (II) is very characteristic of simple sulfonamides (Vorontsova, 1966; Cotton & Stokely, 1970; Klug, 1970; Brink & Mattes, 1986; Lightfoot *et al.*, 1993; Tremayne *et al.*, 1999, 2002; Clark *et al.*, 2003). The related $C_2^2(8)$ motif arises in (I) because two independent molecules participate in the formation of a single chain. On the other hand, the other hydrogen-bond motif most characteristic of sulfonamides, the $R_2^2(8)$ ring (Klug, 1968; Blaschette *et al.*, 1986; Tremayne *et al.*, 1999, 2002; Kelly *et al.*, 2002; Clark *et al.*, 2003), is absent from the structures of both (I) and (II). However, in compound (III) [Cambridge Structural Database (Allen, 2002) refcode SUTYOU; Chandramohan & Ravikumar, 1999], which is an isomer of (II), pairs of N—H···O hydrogen bonds generate centrosymmetric $R_2^2(8)$ rings, as noted in the original report. In addition, however, the resulting dimers are linked into [110] chains by a single aromatic π–π stacking interaction (Fig. 7).

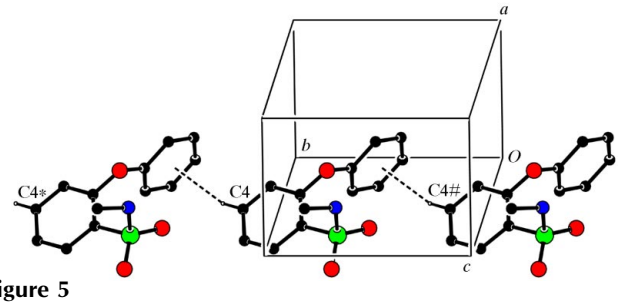


Figure 5
Part of the crystal structure of (II), showing the formation of a C—H···π(arene) chain along [010]. For the sake of clarity, H atoms not involved in the hydrogen-bonding motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, 1 + y, z)$ and $(x, y - 1, z)$, respectively.

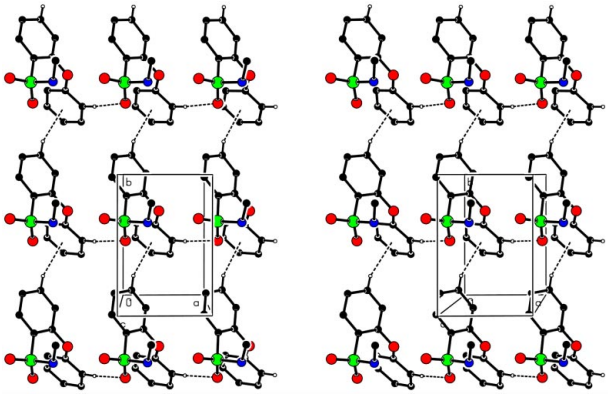


Figure 6
A stereoview of part of the crystal structure of (II), showing the formation of an (001) sheet by combination of the [100] and [010] chains. For the sake of clarity, H atoms not involved in the hydrogen-bonding motifs shown have been omitted.

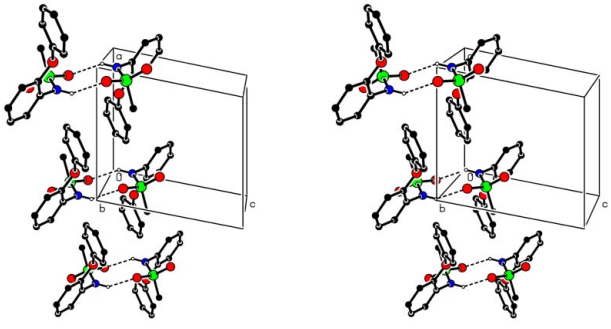


Figure 7
A stereoview of part of the crystal structure of (III) (Chandramohan & Ravikumar, 1999), showing the formation of a π-stacked [110] chain of centrosymmetric hydrogen-bonded dimers. The original atom coordinates have been used. For the sake of clarity, H atoms not involved in the hydrogen-bonding motif shown have been omitted.

Experimental

Samples of (I) and (II) were prepared by the reaction of 2-phenoxybenzenesulfonyl chloride (Neale *et al.*, 1965) with an aqueous ammonia solution for (I) or with an aqueous methylamine solution for (II). Crystals suitable for single-crystal X-ray diffraction were grown from solutions in ethanol. M.p. for (I): 388–389 K [literature value 386–388 K (Abramovitch *et al.*, 1978)]; m.p. for (II): 354–357 K.

Compound (I)

Crystal data

$C_{12}H_{11}NO_3S$
$M_r = 249.28$
Triclinic, $P\overline{1}$
$a = 5.2539\ (2)$ Å
$b = 16.2090\ (8)$ Å
$c = 26.5417\ (9)$ Å
$\alpha = 84.850\ (2)^\circ$
$\beta = 88.951\ (2)^\circ$
$\gamma = 87.607\ (2)^\circ$
$V = 2248.98\ (16)$ Å^3
$Z = 8$
$D_x = 1.472$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9651 reflections
$\theta = 3.1$–27.4°
$\mu = 0.28$ mm^{-1}
$T = 120\ (2)$ K
Block, colourless
0.22 × 0.16 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{min} = 0.926$, $T_{max} = 0.967$
22 883 measured reflections
9651 independent reflections
4912 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 27.4^\circ$
$h = -6 \rightarrow 6$
$k = -20 \rightarrow 20$
$l = -34 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.203$
$S = 1.05$
9651 reflections
614 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0812P)^2 + 1.7227P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ e Å^{-3}

Table 1
Selected torsion angles (°) for (I) and (II).

Parameter	(I, $n = A$)	(I, $n = B$)	(I, $n = C$)	(I, $n = D$)	(II, n = nil)
C21n—O2n—C2n—C1n	132.0 (4)	−130.1 (4)	132.5 (4)	−130.6 (4)	101.8 (3)
C2n—O2n—C21n—C22n	−26.3 (6)	−98.4 (5)	96.5 (5)	23.7 (6)	−6.1 (4)
C2n—C1n—S1n—N1n	51.5 (4)	−50.2 (4)	50.4 (4)	−50.6 (4)	66.5 (3)
C2n—C1n—S1n—O11n	166.2 (3)	−165.4 (3)	165.4 (3)	−165.8 (3)	−179.0 (2)
C2n—C1n—S1n—O12n	−66.1 (4)	66.3 (4)	−67.0 (4)	66.0 (4)	−48.6 (3)
C1—S1—N1—C11					61.3 (2)

Compound (II)

Crystal data

$C_{13}H_{13}NO_3S$
M_r = 263.30
Monoclinic, $P2_1$
a = 5.3804 (2) Å
b = 7.9959 (4) Å
c = 14.4462 (7) Å
β = 95.226 (2)°
V = 618.91 (5) Å^3
Z = 2
D_x = 1.413 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2703 reflections
θ = 2.9–27.5°
μ = 0.26 mm^{-1}
T = 120 (2) K
Block, colourless
0.22 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.951, T_{max} = 0.979
7538 measured reflections
2703 independent reflections
2548 reflections with $I > 2\sigma(I)$
R_{int} = 0.116
θ_{max} = 27.5°
$h = -6 \rightarrow 6$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.145
S = 1.05
2703 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.5323P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Absolute structure: Flack (1983), with 1188 Friedel pairs
Flack parameter = 0.20 (11)

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H12A···O2A	0.88	2.33	2.897 (5)	122
N1B—H12B···O2B	0.88	2.19	2.894 (4)	137
N1C—H12C···O2C	0.88	2.37	2.871 (4)	117
N1D—H12D···O2D	0.88	2.20	2.915 (4)	138
N1A—H11A···O11B	0.88	2.13	2.950 (4)	156
N1B—H11B···O11A^{i}	0.88	2.08	2.945 (4)	165
N1C—H11C···O11D	0.88	2.14	2.947 (4)	153
N1D—H11D···O11C^{i}	0.88	2.07	2.931 (4)	165
C25B—H25B···O12B^{ii}	0.95	2.47	3.371 (6)	157
C25C—H25C···O12C^{iii}	0.95	2.42	3.320 (6)	158
C26A—H26A···O12A^{i}	0.95	2.52	3.410 (5)	156
C26D—H26D···O12D^{i}	0.95	2.46	3.378 (5)	161

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

Table 3
Hydrogen-bonding geometry (Å, °) for (II).

$Cg1$ is the centroid of ring C21–C26.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O11^{i}	0.88	2.21	2.953 (3)	141
C26—H26···O12^{i}	0.95	2.43	3.377 (4)	174
C4—H4···$Cg1^{ii}$	0.95	2.83	3.741 (3)	161

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

Crystals of (I) are triclinic and space group $P\overline{1}$ was selected and confirmed by the subsequent structure analysis. For (II), the systematic absences permitted $P2_1$ and $P2_1/m$ as possible space groups. Consideration of the unit-cell volume led to the selection of $P2_1$, which was confirmed by the successful structure analysis. All H atoms were located in difference maps and then treated as riding atoms, with C—H distances of 0.95 (aromatic) or 0.98 Å (methyl) and N—H distances of 0.88 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}(C)$ for the methyl group. Examination of the refined structure of (I) using the ADDSYM option in *PLATON* (Spek, 2003) revealed no possible additional symmetry, but scrutiny of the reflection data suggested the possibility of twinning about c^*. Following the use of the TWINROTMAT option in *PLATON* to generate a HKLF 5-type reflection file, modified to take into account possible reflection overlap, further refinement led to significant reductions in the R indices, although with only trivial changes to the atomic coordinates and hence to the derived geometric parameters, and indicated a twin fraction of *ca* 8.8 (2)%. The correct absolute configuration of (II) was established by means of the Flack parameter (Flack, 1983).

For both compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement and data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); structure solution: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997); structure refinement: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the authors thank the staff for all their help and advice. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work. JLW thanks CNPq and FAPERJ for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1716). Services for accessing these data are described at the back of the journal.

References

Abramovitch, R. A., Azogu, C. I., McMaster, I. T. M. & Vanderpool, D. P. (1978). *J. Org. Chem.* **43**, 1218–1226.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Blaschette, A., Wieland, E., Schomburg, D. & Adelhelm, M. (1986). *Z. Anorg. Allg. Chem.* **533**, 7–17.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Brink, K. & Mattes, R. (1986). *Acta Cryst.* C**42**, 319–322.
Chandramohan, K. & Ravikumar, K. (1999). *Acta Cryst.* C**55**, IUC9800078.
Clark, J. C., McLaughlin, M. L. & Fronczek, F. R. (2003). *Acta Cryst.* E**59**, o2005–o2006.
Cotton, F. A. & Stokely, P. F. (1970). *J. Am. Chem. Soc.* **92**, 294–302.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Kelly, C. J., Skakle, J. M. S., Wardell, J. L., Wardell, S. M. S. V., Low, J. N. & Glidewell, C. (2002). *Acta Cryst.* B**58**, 94–108.
Klug, H. P. (1968). *Acta Cryst.* B**24**, 792–802.
Klug, H. P. (1970). *Acta Cryst.* B**26**, 1268–1275.
Lightfoot, P., Tremayne, M., Glidewell, C., Harris, K. D. M. & Bruce, P. G. (1993). *J. Chem. Soc. Perkin Trans.* 2, pp. 1625–1630.
McArdle, P. (2003). *OSCAIL for Windows*. Version 10. Crystallography Centre, Chemistry Department, NUI Galway, Ireland.
Neale, A. J., Rawlins, T. J. & McCall, E. B. (1965). *Tetrahedron*, **21**, 1299–1313.
Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tremayne, M., MacLean, E. J., Tang, C. C. & Glidewell, C. (1999). *Acta Cryst.* B**55**, 1068–1074.
Tremayne, M., Seaton, C. C. & Glidewell, C. (2002). *Acta Cryst.* B**58**, 823–834.
Vorontsova, L. G. (1966). *Zh. Strukt. Khim.* **7**, 280–283.

supporting information

Acta Cryst. (2004). C**60**, o364–o367 [doi:10.1107/S010827010400719X]

Monoclinic pseudosymmetry in 2-phenoxybenzenesulfonamide, a triclinic structure having $Z' = 4$, and spontaneous resolution in monoclinic *N*-methyl-2-phenoxybenzenesulfonamide

Christopher Glidewell, John N. Low, Janet M. S. Skakle and James L. Wardell

Computing details

For both compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

(I) 2-Phenoxybenzenesulfonamide

Crystal data

$C_{12}H_{11}NO_3S$
$M_r = 249.28$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.2539$ (2) Å
$b = 16.2090$ (8) Å
$c = 26.5417$ (9) Å
$\alpha = 84.850$ (2)°
$\beta = 88.951$ (2)°
$\gamma = 87.607$ (2)°
$V = 2248.98$ (16) Å^3
$Z = 8$
$F(000) = 1040$
$D_x = 1.472$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9651 reflections
$\theta = 3.1$–$27.4°$
$\mu = 0.28$ mm^{-1}
$T = 120$ K
Block, colourless
0.22 × 0.16 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: rotating anode
Graphite monochromator
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{min} = 0.926$, $T_{max} = 0.967$
22883 measured reflections
9651 independent reflections
4912 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.1°$
$h = -6 \rightarrow 6$
$k = -20 \rightarrow 20$
$l = -34 \rightarrow 33$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.203$
$S = 1.05$
9651 reflections
614 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0812P)^2 + 1.7227P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.33$ e Å^{-3}
	$\Delta\rho_{min} = -0.64$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1A	0.9210 (2)	0.51997 (7)	0.82938 (4)	0.0198 (3)
O11A	1.0634 (6)	0.50443 (18)	0.78422 (11)	0.0251 (7)
O12A	1.0312 (6)	0.49179 (19)	0.87669 (11)	0.0270 (7)
N1A	0.6529 (7)	0.4766 (2)	0.82568 (13)	0.0226 (8)
C1A	0.8649 (8)	0.6284 (3)	0.82745 (15)	0.0191 (9)
C2A	0.6866 (8)	0.6621 (3)	0.85996 (15)	0.0213 (10)
C3A	0.6413 (9)	0.7466 (3)	0.85754 (18)	0.0297 (11)
C4A	0.7776 (9)	0.7977 (3)	0.82392 (18)	0.0308 (12)
C5A	0.9611 (9)	0.7650 (3)	0.79166 (18)	0.0298 (11)
C6A	1.0011 (9)	0.6801 (3)	0.79349 (16)	0.0241 (10)
O2A	0.5518 (6)	0.60737 (19)	0.89177 (11)	0.0283 (7)
C21A	0.5289 (8)	0.6196 (3)	0.94330 (16)	0.0222 (10)
C22A	0.7088 (8)	0.6612 (3)	0.96774 (17)	0.0280 (11)
C23A	0.6791 (10)	0.6672 (3)	1.01920 (18)	0.0363 (12)
C24A	0.4724 (9)	0.6321 (3)	1.04564 (17)	0.0321 (12)
C25A	0.2981 (9)	0.5914 (3)	1.02017 (17)	0.0301 (11)
C26A	0.3255 (8)	0.5852 (3)	0.96859 (16)	0.0250 (10)
S1B	0.4192 (2)	0.47590 (7)	0.67480 (4)	0.0197 (3)
O11B	0.5758 (6)	0.45312 (19)	0.71816 (10)	0.0248 (7)
O12B	0.5083 (6)	0.45230 (19)	0.62674 (11)	0.0269 (7)
N1B	0.1466 (7)	0.4351 (2)	0.68614 (13)	0.0230 (8)
C1B	0.3736 (8)	0.5853 (3)	0.67105 (14)	0.0179 (9)
C2B	0.1844 (8)	0.6248 (3)	0.64027 (15)	0.0200 (10)
C3B	0.1474 (9)	0.7098 (3)	0.63852 (16)	0.0251 (10)
C4B	0.3062 (9)	0.7555 (3)	0.66545 (17)	0.0263 (11)
C5B	0.4992 (9)	0.7165 (3)	0.69449 (17)	0.0275 (11)
C6B	0.5312 (8)	0.6314 (3)	0.69817 (16)	0.0238 (10)
O2B	0.0314 (6)	0.57638 (18)	0.61448 (10)	0.0240 (7)
C21B	0.0086 (8)	0.6011 (3)	0.56234 (16)	0.0229 (10)
C22B	−0.2019 (8)	0.6484 (3)	0.54527 (17)	0.0280 (11)
C23B	−0.2242 (10)	0.6702 (3)	0.49382 (18)	0.0367 (12)
C24B	−0.0384 (9)	0.6447 (3)	0.46043 (17)	0.0318 (12)
C25B	0.1717 (10)	0.5968 (3)	0.47842 (18)	0.0357 (12)
C26B	0.1940 (9)	0.5752 (3)	0.52960 (17)	0.0308 (11)
S1C	0.4039 (2)	0.01826 (7)	0.82718 (4)	0.0211 (3)
O11C	0.5445 (6)	0.00351 (19)	0.78144 (11)	0.0267 (7)
O12C	0.5152 (6)	−0.01135 (19)	0.87439 (11)	0.0281 (7)
N1C	0.1336 (7)	−0.0237 (2)	0.82357 (13)	0.0239 (9)
C1C	0.3535 (8)	0.1270 (3)	0.82664 (15)	0.0201 (10)

C2C	0.1739 (8)	0.1599 (3)	0.85987 (15)	0.0208 (10)
C3C	0.1398 (9)	0.2450 (3)	0.85989 (17)	0.0265 (11)
C4C	0.2879 (9)	0.2962 (3)	0.82792 (18)	0.0286 (11)
C5C	0.4672 (9)	0.2639 (3)	0.79521 (18)	0.0298 (11)
C6C	0.4982 (9)	0.1789 (3)	0.79464 (16)	0.0264 (11)
O2C	0.0296 (6)	0.10505 (19)	0.88953 (10)	0.0250 (7)
C21C	0.0092 (8)	0.1174 (3)	0.94146 (16)	0.0209 (10)
C22C	−0.1974 (8)	0.1603 (3)	0.95901 (18)	0.0298 (11)
C23C	−0.2170 (9)	0.1705 (3)	1.01029 (18)	0.0348 (12)
C24C	−0.0324 (10)	0.1366 (3)	1.04281 (17)	0.0310 (12)
C25C	0.1738 (9)	0.0930 (3)	1.02408 (18)	0.0338 (12)
C26C	0.1970 (9)	0.0835 (3)	0.97331 (18)	0.0308 (11)
S1D	−0.0949 (2)	−0.02297 (7)	0.67248 (4)	0.0189 (3)
O11D	0.0567 (5)	−0.04790 (18)	0.71626 (10)	0.0243 (7)
O12D	−0.0043 (6)	−0.04732 (19)	0.62480 (10)	0.0266 (7)
N1D	−0.3707 (6)	−0.0615 (2)	0.68270 (13)	0.0205 (8)
C1D	−0.1313 (8)	0.0865 (3)	0.66906 (15)	0.0180 (9)
C2D	−0.3151 (8)	0.1286 (3)	0.63740 (16)	0.0217 (10)
C3D	−0.3519 (8)	0.2129 (3)	0.63762 (17)	0.0282 (11)
C4D	−0.1973 (9)	0.2560 (3)	0.66681 (17)	0.0292 (11)
C5D	−0.0105 (8)	0.2159 (3)	0.69662 (17)	0.0258 (11)
C6D	0.0219 (8)	0.1308 (3)	0.69822 (16)	0.0233 (10)
O2D	−0.4625 (6)	0.08193 (19)	0.60998 (11)	0.0260 (7)
C21D	−0.4872 (8)	0.1048 (3)	0.55804 (15)	0.0206 (10)
C22D	−0.3120 (9)	0.1500 (3)	0.53022 (17)	0.0279 (11)
C23D	−0.3449 (9)	0.1664 (3)	0.47859 (18)	0.0341 (12)
C24D	−0.5506 (9)	0.1358 (3)	0.45535 (17)	0.0307 (12)
C25D	−0.7246 (9)	0.0889 (3)	0.48389 (16)	0.0269 (11)
C26D	−0.6950 (8)	0.0738 (3)	0.53546 (16)	0.0250 (10)
H11A	0.5965	0.4820	0.7945	0.027*
H12A	0.5352	0.4894	0.8480	0.027*
H3A	0.5160	0.7696	0.8791	0.036*
H4A	0.7466	0.8560	0.8226	0.037*
H5A	1.0567	0.8007	0.7689	0.036*
H6A	1.1228	0.6569	0.7713	0.029*
H22A	0.8492	0.6849	0.9496	0.034*
H23A	0.8003	0.6954	1.0367	0.044*
H24A	0.4526	0.6363	1.0810	0.039*
H25A	0.1573	0.5673	1.0380	0.036*
H26A	0.2040	0.5572	0.9510	0.030*
H12B	0.0480	0.4591	0.6622	0.028*
H11B	0.0928	0.4540	0.7147	0.028*
H3B	0.0137	0.7369	0.6190	0.030*
H4B	0.2822	0.8141	0.6639	0.032*
H5B	0.6102	0.7485	0.7120	0.033*
H6B	0.6600	0.6044	0.7191	0.029*
H22B	−0.3292	0.6657	0.5684	0.034*
H23B	−0.3679	0.7029	0.4814	0.044*

H24B	−0.0544	0.6599	0.4252	0.038*
H25B	0.2994	0.5790	0.4555	0.043*
H26B	0.3373	0.5425	0.5421	0.037*
H11C	0.0815	−0.0149	0.7922	0.029*
H12C	0.0414	−0.0217	0.8515	0.029*
H3C	0.0159	0.2680	0.8817	0.032*
H4C	0.2663	0.3546	0.8284	0.034*
H5C	0.5674	0.2998	0.7735	0.036*
H6C	0.6193	0.1561	0.7722	0.032*
H22C	−0.3261	0.1829	0.9365	0.036*
H23C	−0.3586	0.2011	1.0230	0.042*
H24C	−0.0469	0.1431	1.0780	0.037*
H25C	0.3013	0.0694	1.0465	0.041*
H26C	0.3402	0.0539	0.9604	0.037*
H12D	−0.4542	−0.0373	0.6566	0.025*
H11D	−0.4246	−0.0460	0.7121	0.025*
H3D	−0.4824	0.2414	0.6179	0.034*
H4D	−0.2207	0.3145	0.6663	0.035*
H5D	0.0956	0.2467	0.7160	0.031*
H6D	0.1485	0.1026	0.7192	0.028*
H22D	−0.1691	0.1700	0.5461	0.033*
H23D	−0.2260	0.1989	0.4590	0.041*
H24D	−0.5722	0.1469	0.4199	0.037*
H25D	−0.8644	0.0672	0.4680	0.032*
H26D	−0.8154	0.0424	0.5553	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0224 (6)	0.0181 (7)	0.0191 (5)	0.0001 (5)	0.0003 (4)	−0.0033 (4)
O11A	0.0259 (16)	0.0231 (19)	0.0272 (16)	0.0007 (13)	0.0060 (14)	−0.0087 (13)
O12A	0.0348 (18)	0.0218 (19)	0.0244 (16)	−0.0009 (14)	−0.0044 (14)	−0.0008 (13)
N1A	0.026 (2)	0.020 (2)	0.0228 (19)	−0.0059 (16)	0.0031 (16)	−0.0054 (15)
C1A	0.020 (2)	0.016 (3)	0.021 (2)	−0.0022 (18)	−0.0015 (18)	−0.0040 (18)
C2A	0.025 (2)	0.017 (3)	0.023 (2)	−0.0045 (19)	0.0037 (19)	−0.0063 (18)
C3A	0.029 (3)	0.021 (3)	0.040 (3)	0.004 (2)	0.004 (2)	−0.009 (2)
C4A	0.037 (3)	0.013 (3)	0.043 (3)	0.002 (2)	−0.007 (2)	−0.006 (2)
C5A	0.037 (3)	0.020 (3)	0.032 (3)	−0.008 (2)	0.002 (2)	−0.001 (2)
C6A	0.025 (2)	0.024 (3)	0.023 (2)	−0.001 (2)	0.0021 (19)	−0.0057 (19)
O2A	0.0341 (18)	0.027 (2)	0.0256 (16)	−0.0082 (15)	0.0125 (14)	−0.0113 (14)
C21A	0.022 (2)	0.018 (3)	0.027 (2)	0.0066 (19)	0.0054 (19)	−0.0072 (18)
C22A	0.018 (2)	0.030 (3)	0.036 (3)	0.000 (2)	0.003 (2)	−0.007 (2)
C23A	0.040 (3)	0.036 (3)	0.034 (3)	0.001 (2)	−0.007 (2)	−0.009 (2)
C24A	0.039 (3)	0.033 (3)	0.024 (2)	0.010 (2)	−0.004 (2)	−0.002 (2)
C25A	0.031 (3)	0.027 (3)	0.031 (3)	0.004 (2)	0.008 (2)	0.000 (2)
C26A	0.022 (2)	0.025 (3)	0.027 (2)	0.0036 (19)	0.0016 (19)	−0.0049 (19)
S1B	0.0210 (6)	0.0169 (7)	0.0214 (6)	0.0013 (5)	0.0015 (5)	−0.0036 (4)
O11B	0.0250 (16)	0.0243 (19)	0.0249 (16)	0.0043 (13)	−0.0016 (14)	−0.0036 (13)

O12B	0.0297 (17)	0.026 (2)	0.0253 (16)	0.0045 (14)	0.0087 (14)	−0.0063 (13)
N1B	0.026 (2)	0.018 (2)	0.0254 (19)	−0.0020 (16)	0.0032 (16)	−0.0035 (15)
C1B	0.019 (2)	0.020 (3)	0.015 (2)	0.0021 (18)	0.0055 (17)	−0.0006 (17)
C2B	0.018 (2)	0.023 (3)	0.019 (2)	−0.0031 (18)	0.0043 (18)	−0.0036 (18)
C3B	0.029 (2)	0.018 (3)	0.027 (2)	0.002 (2)	0.005 (2)	−0.0003 (19)
C4B	0.031 (3)	0.016 (3)	0.032 (2)	−0.003 (2)	0.010 (2)	−0.0048 (19)
C5B	0.029 (3)	0.023 (3)	0.031 (2)	−0.010 (2)	0.004 (2)	−0.005 (2)
C6B	0.021 (2)	0.026 (3)	0.025 (2)	−0.002 (2)	−0.0014 (19)	−0.0056 (19)
O2B	0.0261 (16)	0.0237 (19)	0.0220 (15)	−0.0056 (13)	−0.0020 (13)	0.0015 (12)
C21B	0.023 (2)	0.019 (3)	0.027 (2)	−0.0074 (19)	−0.005 (2)	0.0001 (18)
C22B	0.023 (2)	0.032 (3)	0.029 (2)	0.003 (2)	0.001 (2)	−0.003 (2)
C23B	0.035 (3)	0.042 (3)	0.032 (3)	0.004 (2)	−0.004 (2)	0.001 (2)
C24B	0.041 (3)	0.035 (3)	0.021 (2)	−0.012 (2)	−0.003 (2)	0.001 (2)
C25B	0.034 (3)	0.042 (4)	0.032 (3)	−0.002 (2)	0.008 (2)	−0.012 (2)
C26B	0.022 (2)	0.036 (3)	0.035 (3)	0.002 (2)	0.000 (2)	−0.007 (2)
S1C	0.0225 (6)	0.0202 (7)	0.0209 (5)	0.0017 (5)	−0.0019 (5)	−0.0035 (4)
O11C	0.0292 (17)	0.026 (2)	0.0259 (16)	−0.0003 (14)	0.0059 (14)	−0.0081 (13)
O12C	0.0336 (18)	0.024 (2)	0.0268 (16)	0.0064 (14)	−0.0086 (14)	−0.0016 (13)
N1C	0.030 (2)	0.021 (2)	0.0208 (18)	−0.0054 (17)	−0.0014 (16)	−0.0031 (15)
C1C	0.020 (2)	0.018 (3)	0.022 (2)	−0.0008 (18)	−0.0035 (19)	−0.0024 (18)
C2C	0.022 (2)	0.019 (3)	0.021 (2)	−0.0027 (19)	−0.0030 (19)	−0.0051 (18)
C3C	0.028 (3)	0.024 (3)	0.029 (2)	0.003 (2)	−0.004 (2)	−0.009 (2)
C4C	0.030 (3)	0.019 (3)	0.037 (3)	0.003 (2)	−0.004 (2)	−0.005 (2)
C5C	0.032 (3)	0.024 (3)	0.033 (3)	−0.008 (2)	0.002 (2)	0.000 (2)
C6C	0.027 (2)	0.028 (3)	0.025 (2)	−0.003 (2)	0.001 (2)	−0.003 (2)
O2C	0.0309 (18)	0.024 (2)	0.0217 (15)	−0.0065 (14)	0.0045 (14)	−0.0063 (13)
C21C	0.020 (2)	0.020 (3)	0.023 (2)	−0.0090 (19)	0.0009 (19)	−0.0036 (18)
C22C	0.020 (2)	0.037 (3)	0.033 (3)	0.001 (2)	−0.001 (2)	−0.003 (2)
C23C	0.030 (3)	0.040 (3)	0.035 (3)	0.002 (2)	0.009 (2)	−0.008 (2)
C24C	0.044 (3)	0.027 (3)	0.023 (2)	−0.009 (2)	0.000 (2)	−0.003 (2)
C25C	0.030 (3)	0.040 (3)	0.030 (3)	0.003 (2)	−0.007 (2)	0.001 (2)
C26C	0.031 (3)	0.024 (3)	0.038 (3)	0.003 (2)	0.000 (2)	−0.005 (2)
S1D	0.0193 (5)	0.0182 (7)	0.0191 (5)	0.0000 (4)	0.0006 (4)	−0.0016 (4)
O11D	0.0259 (16)	0.0235 (19)	0.0233 (16)	0.0026 (13)	−0.0040 (13)	−0.0023 (13)
O12D	0.0331 (18)	0.025 (2)	0.0224 (16)	−0.0007 (14)	0.0068 (14)	−0.0064 (13)
N1D	0.0196 (19)	0.018 (2)	0.0242 (18)	−0.0027 (15)	0.0038 (15)	−0.0012 (15)
C1D	0.020 (2)	0.015 (3)	0.019 (2)	0.0003 (17)	0.0007 (18)	−0.0005 (17)
C2D	0.019 (2)	0.021 (3)	0.025 (2)	−0.0022 (19)	−0.0032 (19)	0.0028 (18)
C3D	0.022 (2)	0.027 (3)	0.035 (3)	0.005 (2)	−0.002 (2)	0.003 (2)
C4D	0.038 (3)	0.016 (3)	0.033 (3)	−0.002 (2)	0.009 (2)	−0.003 (2)
C5D	0.022 (2)	0.022 (3)	0.035 (3)	−0.006 (2)	0.001 (2)	−0.009 (2)
C6D	0.018 (2)	0.028 (3)	0.024 (2)	−0.0030 (19)	0.0009 (19)	−0.0018 (19)
O2D	0.0297 (17)	0.0233 (19)	0.0249 (16)	−0.0060 (14)	−0.0084 (14)	0.0021 (13)
C21D	0.023 (2)	0.016 (3)	0.022 (2)	0.0049 (18)	−0.0037 (19)	0.0006 (17)
C22D	0.022 (2)	0.032 (3)	0.030 (2)	−0.001 (2)	−0.002 (2)	−0.001 (2)
C23D	0.027 (3)	0.038 (3)	0.036 (3)	0.002 (2)	0.007 (2)	0.002 (2)
C24D	0.040 (3)	0.033 (3)	0.018 (2)	0.008 (2)	0.002 (2)	0.0006 (19)
C25D	0.026 (2)	0.029 (3)	0.026 (2)	0.006 (2)	−0.008 (2)	−0.0075 (19)

C26D	0.023 (2)	0.025 (3)	0.027 (2)	−0.0001 (19)	−0.0019 (19)	−0.0016 (19)

Geometric parameters (Å, º)

S1A—O12A	1.422 (3)	S1C—O12C	1.428 (3)
S1A—O11A	1.437 (3)	S1C—O11C	1.443 (3)
S1A—N1A	1.609 (4)	S1C—N1C	1.608 (4)
S1A—C1A	1.767 (4)	S1C—C1C	1.770 (5)
N1A—H11A	0.88	N1C—H11C	0.88
N1A—H12A	0.88	N1C—H12C	0.88
C1A—C6A	1.384 (6)	C1C—C6C	1.381 (6)
C1A—C2A	1.390 (6)	C1C—C2C	1.402 (6)
C2A—C3A	1.376 (6)	C2C—O2C	1.376 (5)
C2A—O2A	1.378 (5)	C2C—C3C	1.384 (6)
C3A—C4A	1.374 (7)	C3C—C4C	1.385 (7)
C3A—H3A	0.95	C3C—H3C	0.95
C4A—C5A	1.397 (7)	C4C—C5C	1.388 (7)
C4A—H4A	0.95	C4C—H4C	0.95
C5A—C6A	1.381 (6)	C5C—C6C	1.382 (6)
C5A—H5A	0.95	C5C—H5C	0.95
C6A—H6A	0.95	C6C—H6C	0.95
O2A—C21A	1.402 (5)	O2C—C21C	1.412 (5)
C21A—C26A	1.364 (6)	C21C—C22C	1.362 (6)
C21A—C22A	1.387 (6)	C21C—C26C	1.376 (6)
C22A—C23A	1.384 (7)	C22C—C23C	1.387 (7)
C22A—H22A	0.95	C22C—H22C	0.95
C23A—C24A	1.396 (7)	C23C—C24C	1.375 (7)
C23A—H23A	0.95	C23C—H23C	0.95
C24A—C25A	1.372 (7)	C24C—C25C	1.378 (7)
C24A—H24A	0.95	C24C—H24C	0.95
C25A—C26A	1.386 (6)	C25C—C26C	1.373 (7)
C25A—H25A	0.95	C25C—H25C	0.95
C26A—H26A	0.95	C26C—H26C	0.95
S1B—O12B	1.429 (3)	S1D—O12D	1.427 (3)
S1B—O11B	1.439 (3)	S1D—O11D	1.439 (3)
S1B—N1B	1.614 (4)	S1D—N1D	1.611 (3)
S1B—C1B	1.774 (4)	S1D—C1D	1.770 (4)
N1B—H11B	0.88	N1D—H11D	0.88
N1B—H12B	0.88	N1D—H12D	0.88
C1B—C6B	1.389 (6)	C1D—C6D	1.391 (6)
C1B—C2B	1.396 (6)	C1D—C2D	1.406 (6)
C2B—O2B	1.378 (5)	C2D—O2D	1.367 (5)
C2B—C3B	1.380 (6)	C2D—C3D	1.373 (6)
C3B—C4B	1.387 (6)	C3D—C4D	1.384 (6)
C3B—H3B	0.95	C3D—H3D	0.95
C4B—C5B	1.382 (7)	C4D—C5D	1.376 (6)
C4B—H4B	0.95	C4D—H4D	0.95
C5B—C6B	1.378 (6)	C5D—C6D	1.381 (6)

C5B—H5B	0.95	C5D—H5D	0.95
C6B—H6B	0.95	C6D—H6D	0.95
O2B—C21B	1.412 (5)	O2D—C21D	1.402 (5)
C21B—C26B	1.371 (6)	C21D—C22D	1.365 (6)
C21B—C22B	1.379 (6)	C21D—C26D	1.389 (6)
C22B—C23B	1.386 (7)	C22D—C23D	1.385 (7)
C22B—H22B	0.95	C22D—H22D	0.95
C23B—C24B	1.382 (7)	C23D—C24D	1.386 (7)
C23B—H23B	0.95	C23D—H23D	0.95
C24B—C25B	1.389 (7)	C24D—C25D	1.384 (7)
C24B—H24B	0.95	C24D—H24D	0.95
C25B—C26B	1.378 (7)	C25D—C26D	1.379 (6)
C25B—H25B	0.95	C25D—H25D	0.95
C26B—H26B	0.95	C26D—H26D	0.95

O12A—S1A—O11A	117.79 (19)	O12C—S1C—O11C	117.95 (19)
O12A—S1A—N1A	108.24 (19)	O12C—S1C—N1C	108.2 (2)
O11A—S1A—N1A	106.54 (19)	O11C—S1C—N1C	106.81 (19)
O12A—S1A—C1A	108.49 (19)	O12C—S1C—C1C	107.93 (19)
O11A—S1A—C1A	106.62 (19)	O11C—S1C—C1C	106.6 (2)
N1A—S1A—C1A	108.9 (2)	N1C—S1C—C1C	109.1 (2)
S1A—N1A—H11A	111.3	S1C—N1C—H11C	108.3
S1A—N1A—H12A	115.9	S1C—N1C—H12C	112.5
H11A—N1A—H12A	112.7	H11C—N1C—H12C	127.7
C6A—C1A—C2A	119.9 (4)	C6C—C1C—C2C	120.5 (4)
C6A—C1A—S1A	119.6 (3)	C6C—C1C—S1C	119.6 (4)
C2A—C1A—S1A	120.5 (3)	C2C—C1C—S1C	119.9 (3)
C3A—C2A—O2A	122.8 (4)	O2C—C2C—C3C	122.8 (4)
C3A—C2A—C1A	120.0 (4)	O2C—C2C—C1C	117.7 (4)
O2A—C2A—C1A	117.1 (4)	C3C—C2C—C1C	119.4 (4)
C4A—C3A—C2A	119.9 (4)	C2C—C3C—C4C	119.5 (4)
C4A—C3A—H3A	120.1	C2C—C3C—H3C	120.3
C2A—C3A—H3A	120.1	C4C—C3C—H3C	120.3
C3A—C4A—C5A	120.9 (4)	C3C—C4C—C5C	121.3 (5)
C3A—C4A—H4A	119.6	C3C—C4C—H4C	119.4
C5A—C4A—H4A	119.6	C5C—C4C—H4C	119.4
C6A—C5A—C4A	118.9 (4)	C6C—C5C—C4C	119.2 (4)
C6A—C5A—H5A	120.6	C6C—C5C—H5C	120.4
C4A—C5A—H5A	120.6	C4C—C5C—H5C	120.4
C5A—C6A—C1A	120.4 (4)	C1C—C6C—C5C	120.2 (4)
C5A—C6A—H6A	119.8	C1C—C6C—H6C	119.9
C1A—C6A—H6A	119.8	C5C—C6C—H6C	119.9
C2A—O2A—C21A	119.4 (3)	C2C—O2C—C21C	116.6 (3)
C26A—C21A—C22A	121.8 (4)	C22C—C21C—C26C	121.7 (4)
C26A—C21A—O2A	116.3 (4)	C22C—C21C—O2C	119.6 (4)
C22A—C21A—O2A	121.9 (4)	C26C—C21C—O2C	118.6 (4)
C23A—C22A—C21A	118.4 (5)	C21C—C22C—C23C	118.9 (4)
C23A—C22A—H22A	120.8	C21C—C22C—H22C	120.5

C21A—C22A—H22A	120.8	C23C—C22C—H22C	120.5
C22A—C23A—C24A	120.5 (5)	C24C—C23C—C22C	120.3 (5)
C22A—C23A—H23A	119.8	C24C—C23C—H23C	119.9
C24A—C23A—H23A	119.8	C22C—C23C—H23C	119.9
C25A—C24A—C23A	119.5 (4)	C23C—C24C—C25C	119.6 (4)
C25A—C24A—H24A	120.3	C23C—C24C—H24C	120.2
C23A—C24A—H24A	120.3	C25C—C24C—H24C	120.2
C24A—C25A—C26A	120.6 (5)	C26C—C25C—C24C	120.7 (4)
C24A—C25A—H25A	119.7	C26C—C25C—H25C	119.7
C26A—C25A—H25A	119.7	C24C—C25C—H25C	119.7
C21A—C26A—C25A	119.3 (4)	C25C—C26C—C21C	118.8 (5)
C21A—C26A—H26A	120.4	C25C—C26C—H26C	120.6
C25A—C26A—H26A	120.4	C21C—C26C—H26C	120.6
O12B—S1B—O11B	118.08 (18)	O12D—S1D—O11D	117.78 (19)
O12B—S1B—N1B	107.42 (19)	O12D—S1D—N1D	107.15 (19)
O11B—S1B—N1B	107.34 (19)	O11D—S1D—N1D	107.19 (18)
O12B—S1B—C1B	108.86 (18)	O12D—S1D—C1D	109.24 (19)
O11B—S1B—C1B	106.42 (18)	O11D—S1D—C1D	106.64 (18)
N1B—S1B—C1B	108.40 (19)	N1D—S1D—C1D	108.56 (19)
S1B—N1B—H11B	104.8	S1D—N1D—H11D	106.3
S1B—N1B—H12B	104.2	S1D—N1D—H12D	100.4
H11B—N1B—H12B	106.7	H11D—N1D—H12D	114.3
C6B—C1B—C2B	120.4 (4)	C6D—C1D—C2D	120.0 (4)
C6B—C1B—S1B	119.4 (3)	C6D—C1D—S1D	119.6 (3)
C2B—C1B—S1B	120.1 (3)	C2D—C1D—S1D	120.4 (3)
O2B—C2B—C3B	122.1 (4)	O2D—C2D—C3D	122.7 (4)
O2B—C2B—C1B	118.2 (4)	O2D—C2D—C1D	117.6 (4)
C3B—C2B—C1B	119.6 (4)	C3D—C2D—C1D	119.6 (4)
C2B—C3B—C4B	119.7 (4)	C2D—C3D—C4D	119.5 (4)
C2B—C3B—H3B	120.1	C2D—C3D—H3D	120.2
C4B—C3B—H3B	120.1	C4D—C3D—H3D	120.2
C5B—C4B—C3B	120.5 (4)	C5D—C4D—C3D	121.4 (4)
C5B—C4B—H4B	119.7	C5D—C4D—H4D	119.3
C3B—C4B—H4B	119.7	C3D—C4D—H4D	119.3
C6B—C5B—C4B	120.2 (4)	C4D—C5D—C6D	119.6 (4)
C6B—C5B—H5B	119.9	C4D—C5D—H5D	120.2
C4B—C5B—H5B	119.9	C6D—C5D—H5D	120.2
C5B—C6B—C1B	119.5 (4)	C5D—C6D—C1D	119.8 (4)
C5B—C6B—H6B	120.3	C5D—C6D—H6D	120.1
C1B—C6B—H6B	120.3	C1D—C6D—H6D	120.1
C2B—O2B—C21B	114.7 (3)	C2D—O2D—C21D	118.2 (3)
C26B—C21B—C22B	121.5 (4)	C22D—C21D—C26D	121.3 (4)
C26B—C21B—O2B	118.9 (4)	C22D—C21D—O2D	123.0 (4)
C22B—C21B—O2B	119.6 (4)	C26D—C21D—O2D	115.5 (4)
C21B—C22B—C23B	118.8 (4)	C21D—C22D—C23D	119.2 (4)
C21B—C22B—H22B	120.6	C21D—C22D—H22D	120.4
C23B—C22B—H22B	120.6	C23D—C22D—H22D	120.4
C24B—C23B—C22B	120.3 (4)	C22D—C23D—C24D	120.3 (5)

C24B—C23B—H23B	119.8	C22D—C23D—H23D	119.9
C22B—C23B—H23B	119.8	C24D—C23D—H23D	119.9
C23B—C24B—C25B	120.0 (4)	C25D—C24D—C23D	119.9 (4)
C23B—C24B—H24B	120.0	C25D—C24D—H24D	120.1
C25B—C24B—H24B	120.0	C23D—C24D—H24D	120.1
C26B—C25B—C24B	119.7 (5)	C26D—C25D—C24D	120.0 (4)
C26B—C25B—H25B	120.1	C26D—C25D—H25D	120.0
C24B—C25B—H25B	120.1	C24D—C25D—H25D	120.0
C21B—C26B—C25B	119.7 (4)	C25D—C26D—C21D	119.3 (4)
C21B—C26B—H26B	120.1	C25D—C26D—H26D	120.4
C25B—C26B—H26B	120.1	C21D—C26D—H26D	120.4

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H12*A*···O2*A*	0.88	2.33	2.897 (5)	122
N1*B*—H12*B*···O2*B*	0.88	2.19	2.894 (4)	137
N1*C*—H12*C*···O2*C*	0.88	2.37	2.871 (4)	117
N1*D*—H12*D*···O2*D*	0.88	2.20	2.915 (4)	138
N1*A*—H11*A*···O11*B*	0.88	2.13	2.950 (4)	156
N1*B*—H11*B*···O11*A*^{i}	0.88	2.08	2.945 (4)	165
N1*C*—H11*C*···O11*D*	0.88	2.14	2.947 (4)	153
N1*D*—H11*D*···O11*C*^{i}	0.88	2.07	2.931 (4)	165
C25*B*—H25*B*···O12*B*^{ii}	0.95	2.47	3.371 (6)	157
C25*C*—H25*C*···O12*C*^{iii}	0.95	2.42	3.320 (6)	158
C26*A*—H26*A*···O12*A*^{i}	0.95	2.52	3.410 (5)	156
C26*D*—H26*D*···O12*D*^{i}	0.95	2.46	3.378 (5)	161

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

(II) *N*-methyl-2-phenoxybenzenesulfonamide

Crystal data

$C_{13}H_{13}NO_3S$
$M_r = 263.30$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 5.3804$ (2) Å
$b = 7.9959$ (4) Å
$c = 14.4462$ (7) Å
$\beta = 95.226$ (2)°
$V = 618.91$ (5) Å^3
$Z = 2$
$F(000) = 276$
$D_x = 1.413$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2703 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.26$ mm^{-1}
$T = 120$ K
Block, colourless
0.22 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: rotating anode
Graphite monochromator
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{min} = 0.951$, $T_{max} = 0.979$
7538 measured reflections
2703 independent reflections
2548 reflections with $I > 2\sigma(I)$

R_{int} = 0.116
θ_{max} = 27.5°, θ_{min} = 2.9°
h = −6→6
k = −10→10
l = −18→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.145
S = 1.05
2703 reflections
164 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.5323P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Absolute structure: Flack (1983), 1188 Friedel pairs
Absolute structure parameter: 0.20 (11)

Special details

Experimental. ?.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.06643 (12)	0.66270 (9)	0.88106 (5)	0.0193 (2)
O2	0.4366 (4)	0.7311 (3)	0.74276 (14)	0.0216 (5)
O11	−0.1415 (4)	0.6849 (3)	0.93551 (15)	0.0265 (5)
O12	0.0746 (5)	0.5187 (3)	0.82236 (17)	0.0267 (5)
N1	0.3168 (4)	0.6586 (4)	0.95212 (16)	0.0201 (5)
C1	0.0851 (6)	0.8451 (4)	0.8135 (2)	0.0201 (6)
C2	0.2691 (5)	0.8611 (4)	0.7510 (2)	0.0192 (6)
C3	0.2937 (6)	1.0089 (4)	0.7025 (2)	0.0234 (6)
C4	0.1366 (6)	1.1431 (4)	0.7163 (2)	0.0255 (6)
C5	−0.0479 (6)	1.1285 (4)	0.7776 (2)	0.0257 (7)
C6	−0.0715 (6)	0.9809 (4)	0.8265 (2)	0.0231 (6)
C11	0.3562 (6)	0.7982 (5)	1.0182 (2)	0.0287 (7)
C21	0.3989 (5)	0.6245 (3)	0.6670 (2)	0.0187 (6)
C22	0.1886 (6)	0.6314 (4)	0.6029 (2)	0.0230 (7)
C23	0.1702 (6)	0.5181 (4)	0.5290 (2)	0.0254 (7)
C24	0.3555 (6)	0.4008 (4)	0.5197 (2)	0.0272 (7)
C25	0.5635 (6)	0.3962 (4)	0.5845 (2)	0.0268 (7)
C26	0.5858 (6)	0.5074 (4)	0.6584 (2)	0.0231 (6)
H1	0.4508	0.6401	0.9229	0.024*
H11A	0.3841	0.9015	0.9841	0.043*
H11B	0.5022	0.7749	1.0619	0.043*
H11C	0.2085	0.8112	1.0525	0.043*
H3	0.4174	1.0185	0.6599	0.028*
H4	0.1551	1.2450	0.6839	0.031*
H5	−0.1572	1.2195	0.7858	0.031*
H6	−0.1951	0.9721	0.8692	0.028*

H22	0.0608	0.7114	0.6095	0.028*
H23	0.0288	0.5216	0.4845	0.030*
H24	0.3409	0.3239	0.4694	0.033*
H25	0.6912	0.3162	0.5780	0.032*
H26	0.7275	0.5036	0.7027	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0177 (3)	0.0157 (3)	0.0243 (3)	−0.0019 (3)	0.0015 (2)	0.0018 (3)
O2	0.0201 (10)	0.0217 (10)	0.0226 (10)	0.0034 (8)	−0.0008 (8)	−0.0045 (9)
O11	0.0176 (10)	0.0281 (13)	0.0346 (11)	−0.0010 (9)	0.0073 (8)	0.0084 (10)
O12	0.0340 (13)	0.0166 (11)	0.0287 (12)	−0.0044 (9)	−0.0010 (10)	0.0006 (10)
N1	0.0167 (11)	0.0196 (11)	0.0238 (11)	0.0007 (12)	0.0013 (8)	−0.0008 (12)
C1	0.0208 (15)	0.0179 (14)	0.0214 (14)	0.0003 (12)	0.0003 (11)	0.0002 (11)
C2	0.0178 (14)	0.0177 (13)	0.0220 (14)	0.0008 (11)	0.0007 (11)	−0.0014 (12)
C3	0.0231 (15)	0.0249 (16)	0.0227 (14)	−0.0030 (12)	0.0040 (11)	−0.0007 (13)
C4	0.0331 (16)	0.0184 (15)	0.0249 (13)	−0.0021 (13)	0.0024 (11)	0.0027 (13)
C5	0.0294 (16)	0.0212 (17)	0.0271 (15)	0.0054 (11)	0.0055 (12)	0.0006 (12)
C6	0.0218 (15)	0.0208 (14)	0.0273 (16)	0.0024 (12)	0.0052 (12)	0.0020 (12)
C11	0.0332 (18)	0.0267 (16)	0.0263 (16)	−0.0032 (14)	0.0028 (13)	−0.0058 (14)
C21	0.0202 (14)	0.0186 (16)	0.0179 (13)	−0.0020 (10)	0.0059 (10)	0.0002 (10)
C22	0.0216 (14)	0.0246 (17)	0.0225 (14)	0.0012 (11)	0.0007 (10)	0.0006 (12)
C23	0.0269 (16)	0.0272 (16)	0.0217 (14)	−0.0032 (13)	0.0011 (12)	0.0010 (13)
C24	0.0320 (18)	0.0292 (17)	0.0216 (15)	−0.0076 (13)	0.0085 (13)	−0.0065 (13)
C25	0.0242 (17)	0.0260 (16)	0.0314 (17)	0.0007 (12)	0.0090 (13)	−0.0035 (13)
C26	0.0193 (14)	0.0237 (15)	0.0265 (15)	−0.0001 (11)	0.0035 (11)	−0.0035 (13)

Geometric parameters (Å, º)

S1—O12	1.433 (2)	C4—H4	0.95
S1—O11	1.436 (2)	C5—C6	1.388 (4)
S1—N1	1.618 (2)	C5—H5	0.95
S1—C1	1.763 (3)	C6—H6	0.95
N1—C11	1.471 (4)	O2—C21	1.388 (3)
N1—H1	0.8802	C21—C26	1.387 (4)
C11—H11A	0.98	C21—C22	1.396 (4)
C11—H11B	0.98	C22—C23	1.397 (4)
C11—H11C	0.98	C22—H22	0.95
C1—C6	1.398 (4)	C23—C24	1.384 (5)
C1—C2	1.405 (4)	C23—H23	0.95
C2—C3	1.386 (4)	C24—C25	1.393 (5)
C2—O2	1.388 (4)	C24—H24	0.95
C3—C4	1.392 (5)	C25—C26	1.386 (4)
C3—H3	0.95	C25—H25	0.95
C4—C5	1.393 (4)	C26—H26	0.95
O12—S1—O11	119.40 (15)	C5—C4—H4	119.9

O12—S1—N1	106.87 (15)	C6—C5—C4	119.9 (3)
O11—S1—N1	107.47 (13)	C6—C5—H5	120.1
O12—S1—C1	109.30 (13)	C4—C5—H5	120.1
O11—S1—C1	106.73 (14)	C5—C6—C1	120.5 (3)
N1—S1—C1	106.38 (15)	C5—C6—H6	119.7
C11—N1—S1	117.3 (2)	C1—C6—H6	119.7
C11—N1—H1	111.2	C21—O2—C2	118.6 (2)
S1—N1—H1	111.7	C26—C21—O2	115.5 (3)
N1—C11—H11A	109.5	C26—C21—C22	121.2 (3)
N1—C11—H11B	109.5	O2—C21—C22	123.4 (3)
H11A—C11—H11B	109.5	C21—C22—C23	118.5 (3)
N1—C11—H11C	109.5	C21—C22—H22	120.7
H11A—C11—H11C	109.5	C23—C22—H22	120.7
H11B—C11—H11C	109.5	C24—C23—C22	120.8 (3)
C6—C1—C2	119.0 (3)	C24—C23—H23	119.6
C6—C1—S1	120.2 (2)	C22—C23—H23	119.6
C2—C1—S1	120.7 (2)	C23—C24—C25	119.6 (3)
C3—C2—O2	120.1 (3)	C23—C24—H24	120.2
C3—C2—C1	120.6 (3)	C25—C24—H24	120.2
O2—C2—C1	119.2 (3)	C26—C25—C24	120.6 (3)
C2—C3—C4	119.8 (3)	C26—C25—H25	119.7
C2—C3—H3	120.1	C24—C25—H25	119.7
C4—C3—H3	120.1	C25—C26—C21	119.3 (3)
C3—C4—C5	120.3 (3)	C25—C26—H26	120.4
C3—C4—H4	119.9	C21—C26—H26	120.4

O12—S1—N1—C11	178.0 (2)	C3—C4—C5—C6	1.4 (5)
O11—S1—N1—C11	−52.7 (3)	C4—C5—C6—C1	−1.3 (5)
C1—S1—N1—C11	61.3 (3)	C2—C1—C6—C5	0.8 (5)
O12—S1—C1—C6	136.2 (3)	S1—C1—C6—C5	176.1 (3)
O11—S1—C1—C6	5.8 (3)	C3—C2—O2—C21	−82.3 (3)
N1—S1—C1—C6	−108.7 (3)	C1—C2—O2—C21	101.8 (3)
O12—S1—C1—C2	−48.6 (3)	C2—O2—C21—C26	174.1 (3)
O11—S1—C1—C2	−179.0 (2)	C2—O2—C21—C22	−6.1 (4)
N1—S1—C1—C2	66.5 (3)	C26—C21—C22—C23	−0.4 (4)
C6—C1—C2—C3	−0.5 (4)	O2—C21—C22—C23	179.8 (3)
S1—C1—C2—C3	−175.8 (2)	C21—C22—C23—C24	0.4 (5)
C6—C1—C2—O2	175.4 (3)	C22—C23—C24—C25	−0.4 (5)
S1—C1—C2—O2	0.2 (4)	C23—C24—C25—C26	0.3 (5)
O2—C2—C3—C4	−175.3 (3)	C24—C25—C26—C21	−0.3 (5)
C1—C2—C3—C4	0.6 (5)	O2—C21—C26—C25	−179.8 (3)
C2—C3—C4—C5	−1.1 (5)	C22—C21—C26—C25	0.4 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O11^{i}	0.88	2.21	2.953 (3)	141

C26—H26···O12^{i}	0.95	2.43	3.377 (4)	174
C4—H4···*Cg*1^{ii}	0.95	2.83	3.741 (3)	161

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