Monoclinic
$P2_1/n$
$a = 11.056\,(4)$ Å
$b = 8.340\,(2)$ Å
$c = 15.5461\,(12)$ Å
$\beta = 104.829\,(13)^\circ$
$V = 1385.6\,(6)$ Å^3
$Z = 4$
$D_x = 1.372$ Mg m^{-3}
D_m not measured

Cell parameters from 16 reflections
$\theta = 10.9\text{–}18.8^\circ$
$\mu = 0.098$ mm^{-1}
$T = 296$ K
Block
$0.37 \times 0.35 \times 0.20$ mm
Amber

Data collection

Rigaku AFC-5*R* diffractometer
$\omega\text{–}2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.947$, $T_{\max} = 1.000$
2575 measured reflections
2442 independent reflections

1771 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.011$
$\theta_{\max} = 25.06^\circ$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 9$
$l = -18 \rightarrow 17$
3 standard reflections every 150 reflections intensity variation: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.147$
$S = 1.045$
2441 reflections
244 parameters
H atoms treated by a mixture of constrained and independent refinement

$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.476P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

The structure was solved by direct methods (*SIR*92; Altomare *et al.*, 1994) and expanded using Fourier techniques (*DIRDIF*94; Beurskens *et al.*, 1994). H atoms were found by difference Fourier techniques. Some were refined isotropically and, for the the remainder, coordinates were refined and the isotropic displacement parameters were fixed.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1995*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995*b*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

AD is an EPSRC-funded postdoctoral assistant. We thank the EPSRC for their support for our work related to Moco and the SERC for funds for the purchase of the Rigaku AFC-5*R* diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1143). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* 27, 435.

Armarego, W. L. F. & Milloy, B. A. (1977). *Aust. J. Chem.* **30**, 2023–2028.

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., de Gelder, R., Israel, R. & Smits, J. M. M. (1994). *The DIRDIF94 Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Brown, D. J. & Jacobsen, N. W. (1961). *J. Chem. Soc.* pp. 4413–4420.

Chan, M. K., Mukund, S., Kletzin, A., Adams, M. W. W. & Rees, D. C. (1995). *Science*, **267**, 1463–1469.

Collison, D., Garner, C. D. & Joule, J. A. (1996). *Chem. Soc. Rev.* pp. 25–32.

Dinsmore, A., Birks, J. H., Garner, C. D. & Joule, J. A. (1997). *J. Chem. Soc. Perkin Trans.* 1, pp. 801–807.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Johnson, J. L., Wuebbens, M. M. & Rajagopalan, K. V. (1989). *J. Biol. Chem.* **264**, 13440–13447.

Molecular Structure Corporation (1995*a*). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1995*b*). *TEXSAN. Single Crystal Structure Analysis Software*. Version 1.7-1. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Romão, M. J., Archer, M., Moura, I., Moura, J. J. G., LeGall, J., Engh, E., Schneider, M., Hof, P. & Huber, R. (1995). *Science*, **270**, 1170–1176.

Schindelin, H., Kisker, C., Hilton, J., Rajagopalan, K. V. & Rees, D. C. (1996). *Science*, **272**, 1615–1621.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1997). C**53**, 1669–1671

Polysulfonylamines. LXXXIX.† *N*,*N*-Bis(methylsulfonyl)benzylamine

Dagmar Henschel, Armand Blaschette and Peter G. Jones*

Institut für Anorganische und Analytische Chemie,
Technische Universität Braunschweig, Postfach 3329,
38023 Braunschweig, Germany. E-mail: jones@xray36.anchem.nat.tu-bs.de

(*Received 7 May 1997; accepted 19 June 1997*)

Abstract

The two independent molecules of the title compound, $C_9H_{13}NO_4S_2$, are inverted with respect to each other but otherwise closely similar; unusually, a racemate thus crystallizes in a chiral space group ($P2_12_12_1$ with $Z = 8$). As is normal for compounds of type R—N$(SO_2CH_3)_2$, the C—N bonds [both 1.493 (3) Å] are lengthened with respect to standard values for acyclic

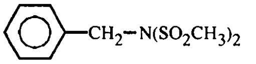
---

† Part LXXXVIII: Lange, Moers, Blaschette & Jones (1997).

amides. The independent molecules are linked into two independent chains by C—H···O hydrogen bonding.

Comment

N-Alkyl disulfonylamines, R—N(SO_2R')$_2$ (R = alkyl, R' = alkyl or aryl), can be readily obtained from primary amines by twofold sulfonylation and have been observed to undergo facile and synthetically useful deamination (Baumgarten & Curtis, 1982). Recent results from this laboratory (Jones, Hamann, Schaper, Lange & Blaschette, 1995) have revealed that the N atom in such molecules usually displays a trigonal-planar geometry and that the electron-withdrawing effect of the two sulfonyl groups causes the C—N distances to be significantly longer than the average C_{sp^3}—N_{sp^2} value for acyclic amides, 1.454 Å (Allen *et al.*, 1987). In this context we now report the structure of the title compound, (1).



(1)

The asymmetric unit of (1) consists of two independent molecules *A* and *A'* (Fig. 1). The molecules are closely similar except that they are inverted with respect to each other; a corresponding least-squares fit indicated a mean deviation of 0.08 Å for all non-H atoms. The frozen conformation imparts chirality to the molecules and the structure is thus a rare example of a racemate crystallizing in a chiral space group (excluding cases of spontaneous resolution, where both forms do not exist in the same crystal). Similar cases have been discussed by Görbitz, Kazmeier & Grandel (1997).

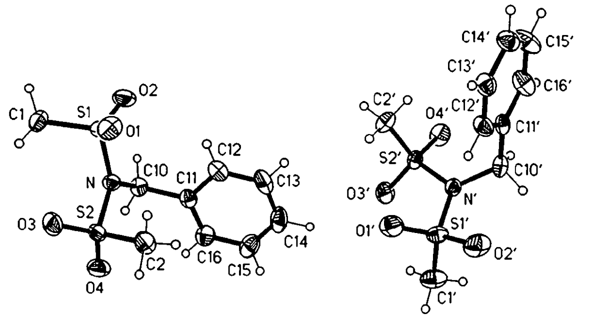


Fig. 1. The structure of the title compound in the crystal. Ellipsoids represent 50% probability levels. H-atom radii are arbitrary.

The C—N distance, 1.493 (3) Å in both molecules, is well above the upper quartile value of 1.461 Å for C_{sp^3}—N_{sp^2} bonds in acyclic amides (Allen *et al.*, 1987). For comparison, the corresponding C—N bond lengths observed in related molecules R—N(SO_2CH_3)$_2$, where R = methyl, cyclobutyl, cyclopropylmethyl or 1-adamantyl, are 1.484 (3), 1.491 (2), 1.498 (3) and 1.556 (2) Å, respectively (Jones *et al.*, 1995, and unpublished results from this laboratory). In (1), the angles at N lie in the range 118.27 (14)–120.79 (14)° (*cf.* Table 1), N and N' being 0.064 (2) and 0.079 (2) Å out of the planes defined by the atoms to which they are bonded. Bond lengths and angles within the N(SO_2CH_3)$_2$ moiety are normal for this class of compound, which generally exhibit appreciably shortened S—N bonds [here: average 1.672 (2) Å]; see Jones *et al.* (1995) and other previous papers in this series. The CH_2—C_{ar} distances, 1.511 (3) Å in *A* and 1.516 (3) Å in *A'*, do not differ significantly from the standard value of 1.510 Å for RCH_2—C_{ar} bonds (Allen *et al.*, 1987).

As quantified by the torsion angles in Table 1, the N(SO_2C)$_2$ frameworks of both molecules adopt local pseudo-C_2 symmetry, while the tilts of the phenyl groups are described by the dihedral angles between their least-squares planes and the corresponding C11, C10, N plane; 56.4 (2)° in *A* and 49.7 (2)° in *A'*.

The molecular packing involves C—H···O interactions that may be classed as hydrogen bonds between the O4 and benzylic H atoms (Table 2; Desiraju, 1996). Through these, the molecules are linked by 2_1 operators into chains; molecules *A* parallel to the *y* axis (Fig. 2) and molecules *A'* parallel to the *x* axis.

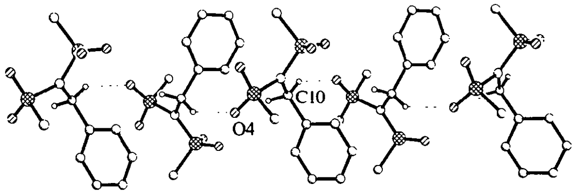


Fig. 2. Chain of molecules *A* (see text). Hydrogen bonds are indicated by broken lines. Radii are arbitrary. H atoms other than those of the methylene group have been omitted.

Experimental

Compound (1) (m.p. 390 K) was prepared as formerly described (Dalluhn, Pröhl, Henschel, Blaschette & Jones, 1996) and crystallized from CH_2Cl_2–petroleum ether at 243 K.

Crystal data

$C_9H_{13}NO_4S_2$
M_r = 263.32
Orthorhombic
$P2_12_12_1$
a = 8.3369 (10) Å
b = 9.5060 (12) Å
c = 29.565 (3) Å
V = 2343.0 (5) Å^3
Z = 8
D_x = 1.493 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 41 reflections
θ = 5.2–12.2°
μ = 0.452 mm^{-1}
T = 173 (2) K
Prism
0.6 × 0.6 × 0.5 mm
Colourless

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
5774 measured reflections
5372 independent reflections
4720 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.020$

$\theta_{\max} = 27.51°$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 0$
$l = 0 \rightarrow 38$
3 standard reflections every 247 reflections intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.077$
$S = 1.046$
5372 reflections
293 parameters
H atoms treated by a mixture of constrained and independent refinement
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.237$ e Å^{-3}
$\Delta\rho_{\min} = -0.283$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = −0.06 (6), 2306 Friedel pairs

Table 1. *Selected geometric parameters (Å, °)*

N—S1	1.6651 (18)	N′—S1′	1.6705 (18)
N—S2	1.6777 (18)	N′—S2′	1.6723 (17)
S1—O1	1.4306 (18)	S1′—O1′	1.4197 (19)
S1—O2	1.4319 (18)	S1′—O2′	1.4249 (18)
S1—C1	1.753 (2)	S1′—C1′	1.749 (3)
N—C10	1.493 (3)	N′—C10′	1.493 (3)
C10—C11	1.511 (3)	C10′—C11′	1.516 (3)
S1—N—S2	120.48 (11)	S1′—N′—S2′	120.48 (11)
C10—N—S1	120.79 (14)	C10′—N′—S1′	119.94 (14)
C10—N—S2	118.27 (14)	C10′—N′—S2′	118.86 (15)
O1—S1—N	108.38 (10)	O1′—S1′—N′	108.05 (10)
O2—S1—N	105.73 (10)	O2′—S1′—N′	105.83 (11)
O1—S1—C1	109.06 (12)	O1′—S1′—C1′	109.31 (13)
O2—S1—C1	107.97 (12)	O2′—S1′—C1′	107.98 (13)
N—S1—C1	105.62 (11)	N′—S1′—C1′	105.74 (12)
O1—S1—O2	119.25 (12)	O1′—S1′—O2′	119.13 (12)
N—C10—C11	113.72 (17)	N′—C10′—C11′	113.51 (18)
S2—N—S1—O1	39.91 (16)	S2′—N′—S1′—O1′	−39.00 (15)
S2—N—S1—O2	168.85 (13)	S2′—N′—S1′—O2′	−167.65 (12)
S2—N—S1—C1	−76.85 (15)	S2′—N′—S1′—C1′	77.95 (16)
S1—N—S2—O3	34.18 (16)	S1′—N′—S2′—O3′	−34.41 (14)
S1—N—S2—O4	162.81 (12)	S1′—N′—S2′—O4′	−162.51 (12)
S1—N—S2—C2	−81.89 (15)	S1′—N′—S2′—C2′	82.26 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···O4$^{\text{i}}$	0.99	2.58	3.435 (3)	144
C10′—H10D···O4′$^{\text{ii}}$	0.99	2.36	3.172 (3)	139

Symmetry codes: (i) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$.

Methyl H atoms were refined as rigid groups allowed to rotate but not tip; other H atoms riding.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

We thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauf for technical assistance. Mr O. Moers assisted in preparing the figures.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1348). Services for accessing these data are described at the back of the journal.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Baumgarten, R. J. & Curtis, V. A. (1982). *The Chemistry of Functional Groups*, Suppl. F, edited by S. Patai, pp. 929–997. Chichester: John Wiley & Sons.
Dalluhn, J., Pröhl, H.-H., Henschel, D., Blaschette, A. & Jones, P. G. (1996). *Phosphorus Sulfur Silicon*, **114**, 149–160.
Desiraju, G. R. (1996). *Acc. Chem. Res.* **29**, 441–449.
Fait, J. (1991). *XSCANS Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Görbitz, C. H., Kazmeier, U. & Grandel, R. (1997). *Acta Cryst.* C**53**, 1302–1305.
Jones, P. G., Hamann, T., Schaper, W., Lange, I. & Blaschette, A. (1995). *Phosphorus Sulfur Silicon*, **106**, 91–104.
Lange, I., Moers, O., Blaschette, A. & Jones, P. G. (1997). *Z. Anorg. Allg. Chem.* In the press.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1994). *XP. Molecular Graphics Program*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1997). C**53**, 1671-1673

trans-(±)-*N*,*N*′-Bis(salicylidene)-1,2-cyclohexanediamine†

Qiancai Liu,‡ Mengxian Ding, Yonghua Lin and Yan Xing

Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, 159 Renmin Street, Changchun 130022, People's Republic of China. E-mail: yangliu@public.cc.jl.cn

(*Received 21 January 1997; accepted 10 June 1997*)

Abstract

The title compound, $C_{20}H_{22}N_2O_2$, is C_2 symmetric with the two N atoms bonded to salicylidene groups which are *trans* with respect to the cyclohexane ring.

---

† Alternative name: 2,2′-[1,2-cyclohexanediylbis(iminomethyl)]diphenol.

‡ Present address: Institute of Applied Chemistry, East China University of Science and Technology, 130 Meilong Road, Shanghai 200237, People's Republic of China.

Acta Crystallographica Section C
ISSN 0108-2701 © 1997