= 0·18, $\Delta\rho_{max}/\Delta\rho_{min} = 0{\cdot}6/-0{\cdot}6$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* bond lengths and angles in Fig. 1, the intrastack S···S and S···N contacts in Fig. 2, and the packing arrangement in Fig. 3.

Related literature. The room-temperature structure is reported in Terzis *et al.* (1988).

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* Lists of observed and calculated structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51508 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sakurai, T. & Kobayashi, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–74.

Terzis, A., Psycharis, V., Hountas, A. & Papavassiliou, G. (1988). *Acta Cryst.* C**44**, 128–132.

Acta Cryst. (1989). C**45**, 685–687

Structure of a Sulfonylated Sulfamate: Cyclohexyl *N*-Benzyl-*N*-(*p*-bromophenylsulfonyl)sulfamate

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Abstract. $C_{19}H_{22}BrNO_5S_2$, M_r = 488·4, triclinic, space group $P\bar{1}$, a = 9·184 (3), b = 10·049 (3), c = 12·3825 (4) Å, α = 90·90 (2), β = 91·58 (2), γ = 115·9 (3)°, V = 1027·12 Å^3, Z = 2, D_x = 1·579 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $F(000)$ = 488·0, μ(Mo $K\alpha$) = 21·52 cm^{-1}, T = 291 (1) K, R = 0·053 for 1378 independent reflections with $I_o > 3\sigma(I)$. The most notable feature of the structure is the essentially trigonal planar geometry about the nitrogen, the bond-angle sum being 359·4°. The deviation of nitrogen out of the S_1—S_2—C_1 plane is 0·072 (3) Å. The four S—O bonds: S_1—O_1 [1·434 (6) Å], S_2—O_3 [1·407 (7) Å], S_1—O_2 [1·418 (6) Å] and S_2—O_4 [1·410 (6) Å] are somewhat shorter than similar S—O bonds in related N—S(VI) structures. The two N—S bonds: S_1—N_1 [1·662 (7) Å] and S_2—N_1 [1·672 (7) Å] are also shorter than typical N—S bonds.

Experimental. The title compound was prepared as previously reported (Spillane & Burke, 1986) and crystals were obtained from a saturated *n*-hexane–ethyl acetate (3:1) solution at 273 K. Colorless data crystal 0·30 × 0·40 × 0·50 mm mounted on glass fiber. Intensities were measured with a Hilger & Watts Y290 diffractometer, lattice parameters determined from least-squares data for 12 independent reflections with $2\theta > 20°$. Scan range $2 < \theta < 24°$, standard reflection variation over data collection time was 2%. Data collected in $\theta/2\theta$ mode and $(\sin\theta)/\lambda = 0{\cdot}57$ Å^{-1}, $-10 < h < 10$, $-10 < k < 10$, $0 < l < 13$. 1774 total reflections, 1378 independent reflections with $I > 3\sigma(I)$.

Bromine and sulfur atoms were located by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Structure refinement, using full-matrix least-squares techniques, was carried out using *SHELX*76 (Sheldrick, 1976). Data were corrected for Lorentz and polarization effects but not for absorption. Hydrogen atoms were included in calculated positions. The bromine and sulfur atoms were refined anisotropically. The thermal parameters were terms U_{ij} of $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$. The atomic scattering factors for non-hydrogen and hydrogen atoms, and the anomalous-dispersion correction factors for non-hydrogen atoms

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were taken from the literature (Cromer & Mann, 1968; Cromer & Liberman, 1970; Stewart, Davidson & Simpson, 1965). $R = 0{\cdot}053$, $wR = 0{\cdot}056$, $S = 2{\cdot}8$, $w^{-1} = [(\sigma F_o)^2 - 0{\cdot}0005F_o^2]$. Final $(\Delta/\sigma)_{\rm max} < 0{\cdot}002$, $\Delta\rho_{\rm max} = 0{\cdot}29$ e Å^{-3} and $\Delta\rho_{\rm min} = -0{\cdot}19$ e Å^{-3} on final difference map. 148 parameters refined. All calculations were performed on a VAX 11/785 computer. The *ORTEP*II program was used to obtain the drawings (Johnson, 1971). Table 1 lists the fractional atomic coordinates and thermal parameters.[*] Table 2 gives the bond lengths and angles for the structure shown in Fig. 1.

Table 1. *Fractional atomic coordinates and thermal parameters* (Å^2)

	x	y	z	$U_{\rm iso}$ or $U_{\rm eq}$*
Br1	−0·34422 (15)	0·29213 (14)	0·44332 (9)	0·0852 (9)*
S1	0·0338 (3)	0·1831 (2)	0·8462 (2)	0·041 (1)*
S2	0·3403 (3)	0·2475 (2)	0·7505 (2)	0·046 (1)*
N1	0·1648 (8)	0·1259 (7)	0·7982 (5)	0·041 (2)
O1	−0·0789 (7)	0·0584 (7)	0·9022 (5)	0·059 (2)
O2	0·1226 (7)	0·3201 (6)	0·9029 (5)	0·056 (2)
O3	0·4063 (8)	0·1612 (7)	0·6997 (5)	0·066 (2)
O4	0·3085 (7)	0·3529 (7)	0·6933 (5)	0·061 (2)
O5	0·4480 (7)	0·3341 (6)	0·8494 (4)	0·049 (2)
C1	0·1343 (10)	−0·0331 (9)	0·8131 (7)	0·045 (2)
C2	0·0013 (10)	−0·1368 (9)	0·7358 (6)	0·040 (2)
C3	0·0140 (11)	−0·1245 (10)	0·6248 (7)	0·052 (2)
C4	−0·1141 (12)	−0·2181 (10)	0·5555 (8)	0·062 (3)
C5	−0·2489 (13)	−0·3251 (11)	0·5966 (8)	0·063 (3)
C6	−0·2647 (13)	−0·3424 (12)	0·7045 (8)	0·071 (3)
C7	−0·1351 (12)	−0·2487 (10)	0·7765 (8)	0·060 (3)
C8	−0·0672 (10)	0·2121 (9)	0·7335 (6)	0·038 (2)
C9	−0·0281 (11)	0·3555 (10)	0·7024 (7)	0·050 (2)
C10	−0·1115 (11)	0·3786 (10)	0·6150 (7)	0·054 (2)
C11	−0·2326 (11)	0·2617 (10)	0·5620 (7)	0·055 (3)
C12	−0·2737 (11)	0·1163 (10)	0·5933 (7)	0·056 (3)
C13	−0·1925 (10)	0·0921 (10)	0·6804 (7)	0·049 (2)
C14	0·5182 (10)	0·2650 (10)	0·9254 (7)	0·050 (2)
C15	0·4312 (13)	0·2429 (12)	1·0289 (8)	0·067 (3)
C16	0·5124 (12)	0·1914 (11)	1·1175 (8)	0·070 (3)
C17	0·6908 (11)	0·2963 (10)	1·1339 (7)	0·057 (3)
C18	0·7746 (13)	0·3136 (12)	1·0303 (8)	0·074 (3)
C19	0·6947 (11)	0·3667 (11)	0·9401 (8)	0·060 (3)

* $U_{\rm eq} = (U_{11} + U_{22} + U_{33})/3$.

Table 2. *Bond lengths* (Å) *and angles* (°)

Br1—C11	1·873 (9)	S1—N1	1·662 (7)
S1—O1	1·434 (6)	S1—O2	1·418 (6)
S1—C8	1·752 (8)	S2—N1	1·672 (7)
S2—O3	1·407 (7)	S2—O4	1·410 (6)
S2—O5	1·545 (6)	N1—C1	1·513 (10)
O5—C14	1·470 (10)	C1—C2	1·512 (11)
C2—C3	1·385 (11)	C2—C7	1·382 (12)
C3—C4	1·399 (12)	C4—C5	1·355 (13)
C5—C6	1·353 (13)	C6—C7	1·427 (13)
C8—C9	1·388 (11)	C8—C13	1·390 (11)
C9—C10	1·389 (12)	C10—C11	1·358 (12)
C11—C12	1·404 (12)	C12—C13	1·379 (12)
C14—C15	1·497 (12)	C14—C19	1·495 (12)
C15—C16	1·529 (13)	C16—C17	1·518 (13)
C17—C18	1·488 (13)	C18—C19	1·545 (13)
O1—S1—N1	104·2 (4)	O2—S1—N1	108·2 (3)
O2—S1—O1	119·8 (4)	C8—S1—N1	106·3 (4)
C8—S1—O1	108·6 (4)	C8—S1—O2	108·9 (4)
O3—S2—N1	105·2 (4)	O4—S2—N1	107·3 (4)
O4—S2—O3	121·4 (4)	O5—S2—N1	106·6 (3)
O5—S2—O3	110·4 (4)	O5—S2—O4	105·0 (4)
S2—N1—S1	120·3 (4)	C1—N1—S1	119·0 (5)
C1—N1—S2	120·1 (5)	C14—O5—S2	121·7 (5)
C2—C1—N1	111·2 (6)	C3—C2—C1	121·7 (7)
C7—C2—C1	119·3 (8)	C7—C2—C3	119·0 (8)
C4—C3—C2	120·3 (9)	C5—C4—C3	120 (1)
C6—C5—C4	121 (1)	C7—C6—C5	119 (1)
C6—C7—C2	119·7 (9)	C9—C8—S1	119·6 (6)
C13—C8—S1	119·7 (6)	C13—C8—C9	120·6 (8)
C10—C9—C8	119·6 (8)	C11—C10—C9	120·0 (9)
C10—C11—Br1	120·2 (7)	C12—C11—Br1	119·0 (7)
C12—C11—C10	120·8 (9)	C13—C12—C11	119·6 (9)
C12—C13—C8	119·3 (8)	C15—C14—O5	108·0 (7)
C19—C14—O5	107·2 (7)	C19—C14—C15	112·3 (8)
C16—C15—C14	111·2 (8)	C17—C16—C15	111·7 (8)
C18—C17—C16	110·2 (8)	C19—C18—C17	111·7 (9)
C18—C19—C14	111·0 (8)		

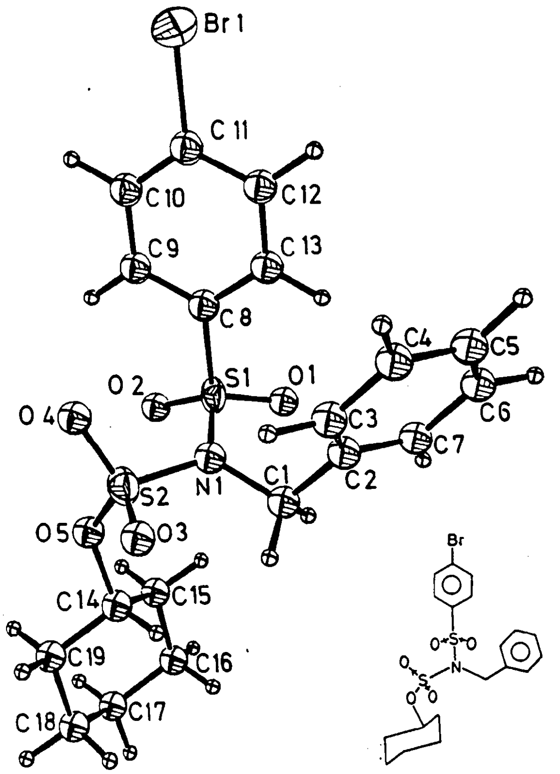

Fig. 1. The structure of cyclohexyl *N*-benzyl-*N*-(*p*-bromophenylsulfonyl)sulfamate.

Related literature. To our knowledge, this is the first X-ray crystallography structural study of a 'sulfonylated sulfamate' *i.e.* $-SO_2N-SO_3-$. Numerous X-ray studies have been reported of sulfamides, $-N-SO_2-N-$, sulfamates, $-N-SO_3-$, and sulfonamides, $-N-SO_2-$. Prominent among the reported

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51517 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

crystal structural studies on (*a*) sulfamides, (*b*) sulfamates and (*c*) sulfonamides are the following: (*a*) Trueblood & Mayer (1956); Jordan, Smith, Lohr & Lipscomb (1963); Atwood, Cowley, Hunter & Mehrotra (1982); (*b*) Morris, Kennard, Hall, Smith & White (1982, 1983); Manickkavachagam & Rajaram (1984); (*c*) Singh, Tiwari & Singh (1985); Rambaud, Maury, Pauvert, Berge, Audran, Lassere & Declercq (1985).

References

Atwood, J. L., Cowley, A. H., Hunter, W. E. & Mehrotra, S. K. (1982). *Inorg. Chem.* **21**, 435–437.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Johnson, C. K. (1971). *ORTEP*II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

Jordan, T., Smith, H. W., Lohr, L. L. & Lipscomb, W. N. (1963). *J. Am. Chem. Soc.* **85**, 846–851.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Manickkavachagam, R. & Rajaram, R. K. (1984). *Z. Kristallogr.* **168**, 179–185.

Morris, A. J., Kennard, C. H. L., Hall, J. R., Smith, G. & White, A. H. (1982). *Inorg. Chim. Acta*, **65**, L153–L155.

Morris, A. J., Kennard, C. H. L., Hall, J. R., Smith, G. & White, A. H. (1983). *Acta Cryst.* C**39**, 1236–1237.

Rambaud, J., Maury, L., Pauvert, B., Berge, G., Audran, M., Lassere, Y. & Declercq, J.-P. (1985). *Acta Cryst.* C**41**, 775–778.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Singh, T. P., Tiwari, R. K. & Singh, M. (1985). *Acta Cryst.* C**41**, 752–755.

Spillane, W. J. & Burke, P. O. (1986). *Synthesis*, pp. 1021–1023.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Trueblood, K. N. & Mayer, S. W. (1956). *Acta Cryst.* **9**, 628–630.

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(3*S*,4*S*)-4-(2-Methoxycarbonylethyl)-3-methoxycarbonylmethyl-3-methyl-2,5-dithioxopyrrolidine

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Abstract. $C_{12}H_{17}NO_4S_2$, $M_r = 303{\cdot}39$, monoclinic, $P2_1$, $a = 7{\cdot}257\,(1)$, $b = 12{\cdot}178\,(2)$, $c = 8{\cdot}650\,(1)$ Å, $\beta = 99{\cdot}70\,(1)°$, $V = 753{\cdot}4$ Å^3, $D_x = 1{\cdot}34$ Mg m^{-3}, $Z = 2$, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 3{\cdot}24$ mm^{-1}, $F(000) = 320$, $T = 293$ K. Final $R = 0{\cdot}036$ for 1054 observed reflections [$F_o > 4\sigma(F_o)$] and 179 parameters. The absolute configuration was established by η refinement, $\eta = 1{\cdot}01\,(11)$. The 2,5-dithioxopyrrolidine ring is crystallographically novel and exhibits approximate C_2 symmetry through N. The C=S bond lengths are identical at 1·630 (4) Å, with C—N at 1·360 (5), 1·367 (5) Å. Molecules are linked by $O(1)\cdots H(1^{i})$—$N(1^{i})$ [(i): $2-x$, $0{\cdot}5+y$, $-z$] hydrogen bonds with $O1\cdots N(1^{i}) = 2{\cdot}899\,(6)$, $O(1)\cdots H(1^{i}) = 2{\cdot}04\,(4)$ Å and $O(1)\cdots H(1^{i})$—$N(1^{i}) = 159\,(3)°$.

Experimental. The dithioimide (I) was prepared from the corresponding imide by treatment with Lawesson's reagent (Scheibye, Pedersen & Lawesson, 1978). A pale yellow plate of size 0·08 × 0·28 × 0·36 mm was selected for the analysis. Data collection on a Nicolet *R*3*mμ* diffractometer with graphite-monochromatized Cu $K\alpha$ radiation and ω–2θ scan range from 0·9° below α_1 to 0·9° above α_2. Cell dimensions obtained by a least-squares procedure based on setting angles for 25 centred reflections with $50 \le 2\theta \le 60°$. Intensities of 1228 reflections were measured within $5 \le 2\theta \le 116°$ (h 0/8, k 0/14, l −10/10). Three check reflections (040, 002, $22\bar{1}$) measured in every 100 showed no significant variation in net intensity. Of 1083 unique reflections ($R_{\rm int} = 0{\cdot}015$) 1056 with $F_o > 4\sigma(F_o)$ were used in the analysis. An empirical absorption correction was applied, based on an ellipsoid model and 400 azimuthal scan data; maximum and minimum transmission factors 0·47 and 0·35. Structure solved by direct methods and refined by blocked-cascade least squares based on F. Non-hydrogen atoms were refined with anisotropic thermal parameters; H atoms riding on C with C—H = 0·96 Å and separate isotropic U's for different H types; H1(N) located on difference Fourier map and x, y, z, U refined freely. Two reflections (121, 020) affected by extinction omitted in final cycles. For 179 parameters $R = 0{\cdot}036$, $wR = 0{\cdot}051$ (observed data), $R = 0{\cdot}038$, $wR = 0{\cdot}052$ (all data), $w^{-1} =$

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