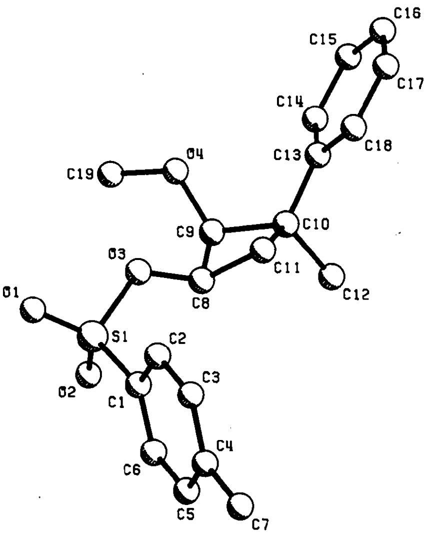

Fig. 1. A diagram of the title molecule.

final cycle $(\Delta/\sigma)_{max} < 0{\cdot}1$ for non-hydrogen atoms, $(\Delta\rho)_{max} = 0{\cdot}24$, $(\Delta\rho)_{min} = -0{\cdot}19$ e Å^{-3}. Neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1* gives the atomic coordinates and Table 2 lists bond lengths and angles. A view of the molecule is shown in Fig. 1.

Related literature. The synthesis has been published (Roskamp & Johnson, 1986). Similar cyclobutanesulfonates for structural comparisons have been published, *e.g.* Decesare, Corbel, Durst & Blount (1981) and Petty, Ikeda, Samuelson, Boriack, Onan, McPhail & Meinwald (1978). A discussion of the stereochemistry of cyclobutane has been given by Moriarty (1974).

Receipt of the material from Professor Carl R. Johnson and Mr Eric J. Roskamp (Wayne State University, Department of Chemistry) is gratefully acknowledged. Thanks also to Dr Kurt L. Loening of Chemical Abstracts Service for his nomenclature advice.

References

Decesare, J. M., Corbel, B., Durst, T. & Blount, J. F. (1981). *Can. J. Chem.* **59**, 1415–1424.

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

Moriarty, R. M. (1974). *Top. Stereochem.* **8**, 271–421.

Petty, R. L., Ikeda, M., Samuelson, G. E., Boriack, C. J., Onan, K. D., McPhail, A. T. & Meinwald, J. (1978). *J. Am. Chem. Soc.* **100**, 2464–2474.

Roskamp, E. J. & Johnson, C. R. (1986). *J. Am. Chem. Soc.* **108**, 6062–6063.

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

Sheldrick, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43967 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1987). C**43**, 1846–1848

Structure of 2-Amino-5-{3-[3-(1-piperidinylmethyl)phenoxy]propylamino}-1,3,4-thiadiazole Hydrochloride (TAS)

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Abstract. $C_{17}H_{26}N_5OS^+.Cl^-$, $M_r = 383{\cdot}96$, monoclinic, $C2/c$, $a = 18{\cdot}050\ (4)$, $b = 22{\cdot}844\ (2)$, $c = 11{\cdot}783\ (2)$ Å, $\beta = 127{\cdot}33\ (1)°$, $V = 3863{\cdot}4\ (12)$ Å^3, $Z = 8$, $D_x = 1{\cdot}32$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}31$ mm^{-1}, $F(000) = 1632$, $T = 293$ K, final $R = 0{\cdot}053$ for 2832 unique observed reflections. The Cl atom is located with equal occupancy (0·5) at two special positions, and protonation occurs at the N atom

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of the piperidine ring by hydrochlorination. The molecules are mainly packed by hydrogen bonding from Cl and N atoms to amine or imine protons.

Table 1. *Atomic positional parameters* (× 10^4) *for* (I) *with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*($Å^2$)
Cl(1)	0	2263 (1)	2500	4·4
Cl(2)	5000	5000	10000	3·8
C(1)	3042 (3)	3740 (2)	7247 (4)	3·2
C(2)	1399 (3)	3528 (2)	5696 (4)	3·0
C(3)	−234 (3)	3585 (2)	4659 (4)	3·6
C(4)	−1133 (3)	3242 (2)	3647 (4)	3·7
C(5)	−1158 (3)	2669 (2)	4255 (4)	3·2
C(6)	−1218 (2)	2348 (2)	6130 (4)	3·1
C(7)	−1195 (2)	1765 (2)	5837 (4)	3·0
C(8)	−1205 (2)	1333 (2)	6657 (4)	2·9
C(9)	−1231 (3)	1485 (2)	7773 (4)	3·6
C(10)	−1265 (3)	2069 (2)	8044 (4)	4·0
C(11)	−1257 (3)	2502 (2)	7237 (4)	3·7
C(12)	−1168 (2)	698 (2)	6358 (4)	3·0
C(13)	−2880 (3)	604 (2)	5089 (4)	3·5
C(14)	−3785 (3)	370 (2)	3772 (4)	3·9
C(15)	−3718 (3)	−281 (2)	3565 (5)	3·7
C(16)	−2899 (3)	−390 (2)	3549 (5)	4·5
C(17)	−2001 (3)	−163 (2)	4889 (5)	3·7
S	2354 (1)	3251 (0)	5820 (1)	3·3
O	−1190 (2)	2807 (1)	5413 (3)	3·9
N(1)	3961 (2)	3784 (2)	7940 (4)	4·6
N(2)	2568 (2)	4050 (1)	7527 (3)	3·3
N(3)	1609 (2)	3934 (1)	6611 (3)	3·4
N(4)	527 (2)	3328 (1)	4719 (3)	3·7
N(5)	−2084 (2)	477 (1)	5049 (3)	2·6

$$^*B_{eq} = \tfrac{4}{3}\sum_i \sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C(1)–S	1·757 (12)	C(1)–(1)	1·336 (12)
C(1)–N(2)	1·298 (7)	C(2)–S	1·758 (6)
C(2)–N(3)	1·292 (6)	C(2)–N(4)	1·347 (14)
C(3)–C(4)	1·524 (14)	C(3)–N(4)	1·455 (7)
C(4)–C(5)	1·507 (6)	C(5)–O	1·435 (7)
C(6)–C(7)	1·383 (6)	C(6)–C(11)	1·393 (8)
C(6)–O	1·367 (6)	C(7)–C(8)	1·389 (6)
C(8)–C(9)	1·389 (7)	C(8)–C(12)	1·502 (5)
C(9)–C(10)	1·382 (6)	C(10)–C(11)	1·378 (7)
C(12)–N(5)	1·509 (17)	C(13)–C(14)	1·513 (17)
C(13)–N(5)	1·494 (7)	C(14)–C(15)	1·522 (6)
C(15)–C(16)	1·511 (9)	C(16)–C(17)	1·511 (18)
C(17)–N(5)	1·492 (5)	N(2)–N(3)	1·402 (14)

S–C(1)–N(1)	121·9 (7)	S–C(1)–N(2)	113·4 (5)
N(1)–C(1)–N(2)	124·7 (4)	S–C(2)–N(3)	114·4 (7)
S–C(2)–N(4)	121·8 (4)	N(3)–C(2)–N(4)	123·8 (6)
C(4)–C(3)–N(4)	110·9 (5)	C(3)–C(4)–C(5)	113·5 (6)
C(4)–C(5)–O	106·9 (3)	C(7)–C(6)–C(11)	120·2 (4)
C(7)–C(6)–O	124·5 (5)	C(11)–C(6)–O	115·3 (4)
C(6)–C(7)–C(8)	119·7 (4)	C(7)–C(8)–C(9)	120·2 (4)
C(7)–C(8)–C(12)	120·1 (4)	C(9)–C(8)–C(12)	119·7 (4)
C(8)–C(9)–C(10)	119·5 (4)	C(9)–C(10)–C(11)	120·9 (5)
C(6)–C(11)–C(10)	119·5 (4)	C(8)–C(12)–N(5)	112·7 (3)
C(14)–C(13)–N(5)	111·0 (5)	C(13)–C(14)–C(15)	111·6 (4)
C(14)–C(15)–C(16)	109·6 (4)	C(15)–C(16)–C(17)	111·6 (6)
C(16)–C(17)–N(5)	109·9 (4)	C(5)–O–C(6)	117·1 (3)
C(1)–N(2)–N(3)	113·5 (3)	C(2)–N(3)–N(2)	112·2 (6)
C(2)–N(4)–C(3)	118·9 (4)	C(12)–N(5)–C(13)	112·4 (5)
C(12)–N(5)–C(17)	109·0 (3)	C(13)–N(5)–C(17)	111·5 (4)

Experimental. Crystals of the title compound grown from isopropyl alcohol–H_2O (1:1) as colorless prisms, m.p. 482–482·5 K. D_m not measured. Single crystal 0·6 × 0·3 × 0·15 mm. Rigaku AFC-5 FOS diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Cell parameters from setting angles of 24 reflections with $9 < \theta < 10°$. No corrections for absorption. 4240 independent reflections measured within $2\theta = 54°$, range of *hkl*: h −23→18, k 0→29, l 0→15, $\omega/2\theta$ scan mode, 2832 with $F > 2\sigma(F)$. Three standard reflections checked every 100 reflections, no significant deviation. Structure solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1970) and refined (on F) by block-diagonal least squares. H atoms from difference map. Refinement with anisotropic temperature factors for non-H atoms and isotropic thermal parameters for H atoms. $R = 0·053$, $wR = 0·053$, $w = 1$, $(\Delta/\sigma)_{max} = 1·18$ (0·47 for non-H atoms), $S = 2·51$, largest peak in final ΔF map 0·14 e $Å^{-3}$. Scattering factors taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters for non-H atoms are listed in Table 1. Bond lengths and angles are given in Table 2.* The molecule is shown in Fig. 1.

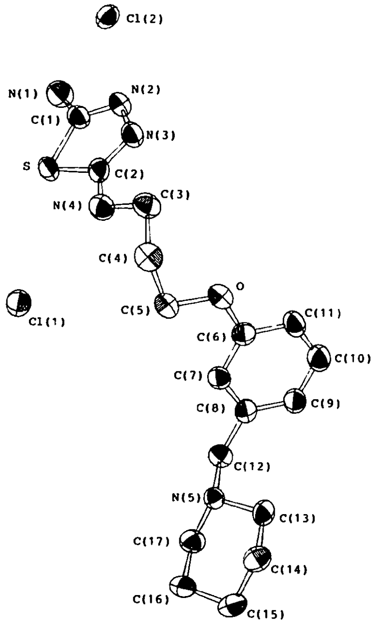

Fig. 1. The molecular structure of (I) with the numbering scheme used.

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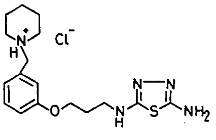
* Lists of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43897 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. This compound (I) was synthesized at our laboratory (Toyofuku, Tsuriya, Kuroda, Aoki & Nagasawa, 1985). It is a new competitive histamine H_2-receptor antagonist, which is eighteen times more potent than ranitidine in inhibiting gastric acid secretion in the pyrolus-ligated rat (6 h) (Tsuriya, Matsukawa, Aoki & Seya, 1984).



(I)

References

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1970). *Acta Cryst.* B**26**, 274–285.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht, The Netherlands.)

TOYOFUKU, H., TSURIYA, Y., KURODA, T., AOKI, H. & NAGASAWA, H. (1985). German Patent No. DE 3,426,533. [*Chem. Abstr.* Vol. 103, P23484*p*.]

TSURIYA, Y., MATSUKAWA, H., AOKI, H. & SEYA, M. (1984). *Jpn. J. Pharmacol.* **36**, Suppl., 90.

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Structure of 2,8-Dihydrodecachlorodihomocubane, or 2,8-Dihydromirex

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Abstract. 1,3,4,5,6,7,9,10,11,12-Decachloropentacyclo[5.3.0.0^{2,6}.0^{3,9}.0^{4,8}]decane, $C_{10}H_2Cl_{10}$, $M_r = 476{\cdot}7$, triclinic, $P\bar{1}$, $a = 8{\cdot}693\ (3)$, $b = 9{\cdot}188\ (4)$, $c = 10{\cdot}396\ (3)$ Å, $\alpha = 88{\cdot}51\ (3)$, $\beta = 74{\cdot}53\ (3)$, $\gamma = 85{\cdot}86\ (3)°$, $V = 798{\cdot}2\ (7)$ Å^3, $Z = 2$, $D_x = 1{\cdot}98$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 17{\cdot}4$ cm^{-1}, $F(000) = 464$, $T = 293$ K, $R = 0{\cdot}030$ for 2245 unique observed reflections. There are two independent molecules in the unit cell, each at a crystallographic inversion center. The C—C—C bond angles vary from 84·7 (2) to 89·9 (2)° for the four-membered rings and from 96·0 (2) to 108·4 (2)° for the five-membered rings. The C—C bond distances range from 1·548 (3) to 1·568 (3) Å for cage C atoms with one *exo* group; the C—C distances to the C atom with the two *exo* Cl atoms are 1·512–1·517 (2) Å. The structure determination establishes the stereochemistry of the dihydro molecule which is one of the first products of chemical and photochemical degradation of the reduction-resistant insecticide mirex.

Experimental. Compound prepared by the literature reaction of dodecachlorodihomocubane (mirex) with phosphine and acetic acid as reported by Kecher, Skibinskaya, Gallai & Zefirov (1974). Crystals of $(CCl)_6(CCl_2)_2(CH)_2$ obtained from hexane solutions. Colorless data crystal 0·22 × 0·28 × 0·46 mm mounted on glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4 to 16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $17 < 2\theta < 23°$. Analytical absorption correction based on crystal shape varied from 0·80 to 1·00. Data collected in $\sin\theta/\lambda$ of 0·60 Å^{-1}, $-10 \le h \le 10$, $-10 \le k \le 10$, $0 \le l \le 12$. Four standard reflections ($2\bar{3}1$, $\bar{4}11$, 214, $\bar{1}\bar{2}4$) lost 1·1% intensity over 25·6 h of data collection; the contribution of each standard to the decay correction was weighted according to the relative distance between the reciprocal-lattice points of the standard reflections and the reflection being corrected. 2978 reflections measured, 2810 unique ($R_{\text{int}} = 0{\cdot}01$), 565 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms refined with isotropic temperature factors, all other atoms refined anisotropically for 189 variables. Three reflections were removed because of extinction. $R = 0{\cdot}030$, $wR = 0{\cdot}039$, $S = 1{\cdot}67$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}009I^2]/4F^2$. Final $(\Delta/\sigma)_{\max} < 0{\cdot}01$, $\Delta\rho_{\max} = 0{\cdot}28\ (3)$ and $\Delta\rho_{\min} = -0{\cdot}51\ (3)$ e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray*

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