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(1*R*,2*S*)-2-[(*S*)-(*p*-Chlorophenyl)sulfinyl]-*N*,*N*,3,3-tetramethylcyclopropylamine

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Abstract. $C_{13}H_{18}NOSCl$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}526\,(1)$, $b = 11{\cdot}130\,(1)$, $c = 11{\cdot}993\,(2)$ Å, $Z = 4$, $D_c = 1{\cdot}28$ g cm^{-3}. Positional and thermal parameters for all atoms were refined. The final agreement index R was 0·037 for 1192 reflexions. The O atom of the sulfinyl group and the dimethylamino group are *syn* related.

Introduction. Reactions of some isomeric aminocyclopropyl sulfoxides were studied because the ring openings of the cyclopropanes proceed *via* zwitterionic intermediates and because of the interesting stereochemistry of the reactions (Rynbrandt, Dutton & Chidester, 1976). This crystal structure was determined as part of the study in order to establish the configuration at the S atom relative to the configuration at C(1) and C(2).

The crystals, which were small clear prisms, were supplied by Dr R. H. Rynbrandt. The racemic mixture crystallized from diethyl ether as an equal mixture of (+) and (−) crystals instead of the usual (±) crystals. Face development was not good enough to permit separation into two types under the microscope. One crystal was selected at random. Precession photographs of the crystal, 0·15 × 0·15 × 0·3 mm, showed that the unit cell is orthorhombic; absence of reflections of type $h00$ for h odd, $0k0$ for k odd, and $00l$ for l odd indicated the space group to be $P2_12_12_1$.

Intensity data for the 1192 reflections for which 2θ was less than 120° were measured using graphite-monochromated Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å) on a Syntex $P\bar{1}$ diffractometer controlled by an IBM 1800 computer. The θ–2θ scan technique was used with a scan range of approximately 3·2° and a variable scan

Table 1. *Final atomic parameters, and standard deviations in parentheses*

All values for anisotropic atoms have been multiplied by 10^4. Coordinates of isotropic atoms have been multiplied by 10^3. The form of the anisotropic temperature factors is $\exp(-B_{11}h^2 - B_{22}k^2 - B_{33}l^2 - B_{12}hk - B_{13}hl - B_{23}kl)$.

	X	Y	Z	B11	B22	B33	B12	B13	B23
CL	772 (1)	8636 (1)	4979 (1)	92 (1)	197 (2)	123 (1)	21 (2)	18 (2)	67 (2)
S	5758 (1)	11048 (1)	3164 (1)	96 (1)	82 (1)	87 (1)	28 (2)	2 (1)	-3 (2)
O	6004 (2)	12060 (2)	3952 (2)	137 (3)	83 (2)	139 (2)	8 (5)	15 (4)	-94 (4)
N	8634 (2)	10962 (3)	2439 (2)	91 (2)	79 (3)	74 (2)	-12 (5)	1 (4)	-22 (5)
C(1)	8187 (2)	9927 (3)	3033 (2)	76 (3)	95 (3)	62 (2)	11 (6)	-20 (4)	14 (6)
C(2)	6838 (3)	9866 (3)	3448 (2)	81 (3)	78 (3)	55 (2)	18 (6)	-6 (4)	3 (5)
C(3)	7295 (3)	9058 (3)	2503 (2)	76 (3)	70 (3)	66 (2)	8 (6)	6 (4)	-1 (6)
C(4)	6840 (3)	9278 (3)	1330 (2)	108 (3)	100 (4)	64 (2)	4 (7)	1 (5)	-18 (6)
C(5)	7461 (3)	7746 (3)	2779 (3)	102 (3)	88 (4)	113 (3)	11 (7)	17 (6)	-4 (7)
C(6)	9080 (3)	11887 (3)	3227 (3)	118 (4)	108 (4)	117 (3)	-40 (8)	-11 (6)	-51 (7)
C(7)	9675 (3)	10607 (3)	1699 (3)	115 (4)	113 (4)	105 (3)	-39 (7)	36 (6)	2 (7)
C(8)	4369 (3)	10293 (3)	3684 (2)	85 (3)	77 (3)	66 (2)	34 (7)	-11 (5)	-14 (5)
C(9)	3974 (3)	9199 (3)	3301 (2)	95 (3)	112 (4)	93 (3)	22 (7)	27 (5)	-68 (6)
C(10)	2866 (3)	8680 (3)	3696 (3)	88 (3)	111 (4)	112 (3)	0 (7)	-11 (5)	-34 (7)
C(11)	2179 (3)	9283 (3)	4476 (2)	74 (3)	125 (4)	81 (2)	58 (7)	-16 (5)	34 (6)
C(12)	2542 (3)	10375 (3)	4880 (2)	111 (3)	129 (5)	70 (2)	62 (8)	27 (5)	-11 (6)
C(13)	3642 (3)	10873 (3)	4482 (2)	125 (4)	81 (4)	81 (2)	30 (7)	-3 (5)	-17 (6)

	X	Y	Z	B		X	Y	Z	B
H(1)	874 (2)	962 (3)	350 (2)	4.3 (0.6)	H(6B)	839 (3)	1214 (3)	366 (2)	5.5 (0.7)
H(2)	668 (2)	951 (2)	415 (2)	2.3 (0.5)	H(6C)	984 (3)	1163 (3)	365 (3)	6.8 (0.9)
H(4A)	603 (2)	891 (3)	127 (2)	4.7 (0.6)	H(7A)	938 (3)	994 (3)	118 (2)	5.6 (0.7)
H(4B)	680 (2)	1008 (2)	113 (2)	3.3 (0.5)	H(7B)	985 (3)	1130 (3)	134 (2)	5.7 (0.8)
H(4C)	738 (2)	886 (3)	80 (2)	4.9 (0.6)	H(7C)	1045 (3)	1026 (3)	208 (2)	7.5 (0.8)
H(5A)	783 (2)	775 (3)	353 (2)	5.0 (0.7)	H(9)	438 (2)	886 (2)	277 (2)	3.2 (0.5)
H(5B)	804 (2)	741 (3)	227 (2)	4.9 (0.7)	H(10)	262 (3)	795 (3)	342 (2)	6.9 (0.8)
H(5C)	670 (2)	735 (2)	283 (2)	4.6 (0.7)	H(12)	205 (2)	1080 (3)	544 (2)	5.5 (0.7)
H(6A)	935 (3)	1254 (3)	286 (2)	6.9 (0.8)	H(13)	388 (2)	1159 (2)	467 (2)	4.4 (0.7)

rate (4–24° min^{-1}) depending on the intensity of the reflection being measured. The total time spent counting background – one half counted at each end of the scan – was equal to the time spent scanning. The crystal orientation was determined by the computer before data collection using 10 orienting reflections.

The crystal became opaque during the data collection, but there was no appreciable loss in intensity in 10 reflections which were monitored periodically. Standard deviations of observed intensities were approximated by $\sigma^2(I) = \sigma^2 + (0{\cdot}0079\ I)^2$ where σ was derived from counting statistics, and the coefficient of I in the last term was calculated from those deviations in the monitored reflections not explained by counting statistics. Lorentz and polarization corrections were applied and absorption corrections were made using the method of Busing & Levy (1957) (μ was 35·3 cm^{-1}; transmission ranged from 0·523 to 0·663).

Accurate cell parameters were determined automatically using a least-squares calculation based on very accurately determined $K\alpha_1$ 2θ values for 20 selected high-2θ reflections.

The structure was solved by Patterson analysis. Refinement was by multiple-matrix least squares. The parameters were divided into separate matrices for (1) coordinates of heavier atoms, (2) H atom coordinates, (3) anisotropic temperature factors for Cl, S, and O atoms, the scale factor and the secondary extinction parameter, and (4) isotropic temperature factors of H atoms. The anisotropic temperature factors of the remaining atoms were in individual 6 x 6 matrices. The quantity minimized was $\Sigma\ \omega(F_o^2 - F_c^{*2})^2$, where F_c^* is as defined by Larson (1967). Weights ω were taken equal to the reciprocals of the variances $\sigma^2(F_o^2)$ determined at data reduction and scaled by propagation of error through subsequent corrections.

Although the space group is $P2_12_12_1$, the crystals were from a racemic mixture of the compound. In the final stage of the refinement ($R = 0{\cdot}04$), it was evident that inclusion of the anomalous components in the calculations could have a substantial effect. The crystal, which was chosen at random, could have been either *R,S,S* or *S,R,R*. The enantiomorph selection was made by calculating the R value for both enantiomers using only the 50 reflections found by computer search to be most affected by anomalous dispersion. The R value for the enantiomer used thus far in the refinement was 0·151; for the other enantiomer, assumed to be correct, R was 0·088. The enantiomorph change was made,

Table 2. *Bond lengths* (Å), *and standard deviations in parentheses*

CL	C(11)	1.753(3)	C(1)	H(1)	0.87(2)
S	O	1.492(3)	C(2)	H(2)	0.94(2)
S	C(2)	1.771(3)	C(4)	H(4A)	0.94(3)
S	C(8)	1.798(3)	C(4)	H(4B)	0.92(3)
N	C(1)	1.434(4)	C(4)	H(4C)	0.97(2)
N	C(6)	1.474(4)	C(5)	H(5A)	0.98(3)
N	C(7)	1.464(4)	C(5)	H(5B)	0.93(3)
C(1)	C(2)	1.506(4)	C(5)	H(5C)	0.92(3)
C(1)	C(3)	1.490(4)	C(6)	H(6A)	0.89(3)
C(2)	C(3)	1.524(4)	C(6)	H(6B)	0.93(3)
C(3)	C(4)	1.505(4)	C(6)	H(6C)	0.99(3)
C(3)	C(5)	1.507(5)	C(7)	H(7A)	1.01(3)
C(8)	C(9)	1.366(5)	C(7)	H(7B)	0.89(3)
C(8)	C(13)	1.385(4)	C(7)	H(7C)	1.01(3)
C(9)	C(10)	1.384(4)	C(9)	H(9)	0.85(2)
C(10)	C(11)	1.359(5)	C(10)	H(10)	0.91(3)
C(11)	C(12)	1.363(5)	C(12)	H(12)	0.96(3)
C(12)	C(13)	1.369(5)	C(13)	H(13)	0.86(3)

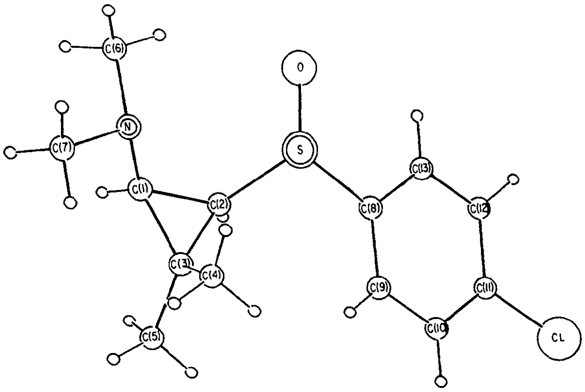

Fig. 1. Numbering, configuration and conformation.

Table 3. *Bond angles and selected torsion angles* (°), *and standard deviations in parentheses*

O	S	C(2)	109.1(1)
O	S	C(8)	105.9(1)
C(2)	S	C(8)	96.2(1)
C(1)	N	C(6)	110.2(2)
C(1)	N	C(7)	109.2(3)
C(6)	N	C(7)	109.7(2)
N	C(1)	C(2)	120.6(3)
N	C(1)	C(3)	121.0(2)
C(2)	C(1)	C(3)	61.1(2)
S	C(2)	C(1)	120.5(2)
S	C(2)	C(3)	119.8(2)
C(1)	C(2)	C(3)	58.9(2)
C(1)	C(3)	C(2)	59.9(2)
C(1)	C(3)	C(4)	119.5(3)
C(1)	C(3)	C(5)	117.4(3)
C(2)	C(3)	C(4)	119.8(3)
C(2)	C(3)	C(5)	116.3(2)
C(4)	C(3)	C(5)	113.6(3)
S	C(8)	C(9)	123.1(2)
S	C(8)	C(13)	118.0(2)
C(9)	C(8)	C(13)	118.7(3)
C(8)	C(9)	C(10)	120.7(3)
C(9)	C(10)	C(11)	118.6(3)
CL	C(11)	C(10)	118.9(3)
CL	C(11)	C(12)	118.7(2)
C(10)	C(11)	C(12)	122.2(3)
C(11)	C(12)	C(13)	118.3(3)
C(8)	C(13)	C(12)	121.2(3)

O	S	C(2)	C(1)	79.0(2)
O	S	C(2)	C(3)	148.3(2)
C(8)	S	C(2)	C(1)	-171.7(2)
C(8)	S	C(2)	C(3)	-102.3(2)
O	S	C(8)	C(9)	168.2(3)
O	S	C(8)	C(13)	-14.1(3)
C(2)	S	C(8)	C(9)	56.3(3)
C(2)	S	C(8)	C(13)	-126.1(2)
C(6)	N	C(1)	C(2)	-86.0(3)
C(6)	N	C(1)	C(3)	-158.6(3)
C(7)	N	C(1)	C(2)	153.2(2)
C(7)	N	C(1)	C(3)	80.6(3)
N	C(1)	C(2)	S	-2.3(4)
N	C(1)	C(2)	C(3)	-110.9(3)
C(3)	C(1)	C(2)	S	108.6(2)
S	C(2)	C(3)	C(4)	-0.8(4)
N	C(1)	C(3)	C(4)	0.8(4)

Table 4. *Selected intermolecular distances*

Distances between nonhydrogen atoms are given which are less than or close to the sum of the van der Waals radii for the individual atoms (Pauling, 1960).

Related atom	Atom	*d* (Å)	Symmetry
C(2)	C(4)	3·846 (4)	$1\frac{1}{2}-x, 2-y, z-\frac{1}{2}$
C(13)	C(7)	3·595 (4)	$1\frac{1}{2}-x, 2-y, z-\frac{1}{2}$
C(6)	C(10)	3·675 (5)	$1-x, y-\frac{1}{2}, \frac{1}{2}-z$
C(8)	C(5)	3·774 (5)	$1-x, y-\frac{1}{2}, \frac{1}{2}-z$
C(13)	C(5)	3·612 (5)	$1-x, y-\frac{1}{2}, \frac{1}{2}-z$
C(7)	C(5)	3·892 (5)	$2-x, y-\frac{1}{2}, \frac{1}{2}-z$
Cl	C(5)	3·571 (3)	$x+\frac{1}{2}, 1\frac{1}{2}-y, 1-z$
C(6)	C(13)	3·739 (5)	$x-\frac{1}{2}, 2\frac{1}{2}-y, 1-z$

and, using the parameters of Cromer & Liberman (1970), anomalous components were included for Cl, S, N, and O atoms.

All calculations were carried out on an IBM 370 computer using the *CRYM* crystallographic system developed by one of the authors (DJD). Atomic form factors are from *International Tables for X-ray Crystallography* (1962), except for H, which was taken from Stewart, Davidson & Simpson (1965). The final value of the secondary extinction parameter *g* was $3{\cdot}8\,(3)\times 10^{-6}$. The final agreement index ($R=\Sigma\,||F_o|-|F_c||/\Sigma F_o$) was 0·037.* The standard deviation of fit $[\Sigma\,\omega(F_o^2-F_c^2)^2/(m-s)]^{1/2}$ was 1·60.

Discussion. Final atomic parameters and their standard deviations are given in Table 1. Fig. 1 shows numbering, configuration and conformation. Bond lengths are listed in Table 2, and bond angles, together with selected torsion angles, in Table 3. Distances and angles about S are consistent with values reported for other sulfoxides (Dahlén, 1974; de la Camp & Hope, 1970).

The torsion angles show that S, N, C(1), and C(2) are approximately in the same plane; the O atom is twisted about 79° out of that plane and is nearly in the plane of the chlorophenyl ring (the perpendicular distance from O to the chlorophenyl plane is 0·23 Å). The reaction mechanism postulated (Rynbrandt *et al.*, 1976) for the stereoselective formation of this compound from a sulfide precursor assumes an N · · · H—O hydrogen bond. The observed intramolecular N—O distance is 3·53 Å; however, rotation about the C(2)—S bond reduces this distance to 2·60 Å, well within hydrogen-bonding range.

Table 4 gives selected intermolecular distances. There are no close aromatic–aromatic contacts, and, for the most part, the methyl groups are also separated. The strongest non-bonded interaction is between Cl and methyl C(5) (distance 3·57 Å). Other interactions are generally between methyls and aromatic carbons.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32010 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.