this work on average agree with those reported by H77 to within 0·005 Å for the D(donor)—A(acceptor) distance, 0·06 Å for the H—A distances, and 5° for the D—H—A angle.

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The Absolute Configuration of (2*R*)-2-(Benzyloxycarbonylamino)-3-hydroxypropyl Chloromethyl (*R*)-Sulphoxide

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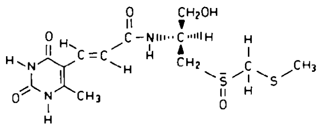
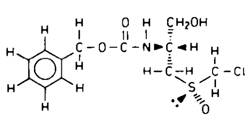
Abstract. $C_{12}H_{16}ClNO_4S$, monoclinic, $P2_1$, a = 4·829 (1), b = 10·402 (2), c = 14·174 (4) Å, β = 97·95 (2)°, Z = 2, V = 705 Å^3, D_c = 1·44 Mg m^{-3}. The structure was solved by routine direct methods and refined to R = 0·033 for 2901 observed reflections. Determination of the absolute configuration, based upon the anomalous scattering of S and Cl, using Mo $K\alpha$ and Cu $K\alpha$ radiations, showed R chirality at the S atom and the asymmetric C atom.

Introduction. The structure of the antibiotic sparsomycin (I) was suggested mainly on the basis of spectroscopic and degradation studies (Wiley & MacKellar, 1976). The chiral C atom has the S configuration as depicted. The configuration of the sulphoxide S atom is unknown. Recently, a total synthesis of the enantiomer and a diastereomer of sparsomycin, both having R chirality at the C atom, was reported (Ottenheijm, Liskamp & Tijhuis, 1979). One of the intermediates in the synthesis of the diastereomeric compound is (II). The chirality of the C atom is R, whereas that of the S atom is unknown, but is identical with that of the S atom in the natural product (I). To determine the stereochemistry of the sulphoxide S atom in (II), we subjected (II) to an X-ray study.



(I)



(II)

A crystal (0·1 × 0·1 × 0·5 mm) was used for data collection on a Nonius CAD-4 diffractometer with a graphite monochromator (Mo $K\alpha$, λ = 0·71069 Å). Systematic absences (0k0, k odd) are consistent with space group $P2_1$. Unit-cell parameters were obtained by least-squares treatment of 25 reflections with 38° < 2θ < 42°. Intensities were collected in the ω–2θ scan mode with a scan width of $\Delta\theta$ = (0·95 + 0·35 tan θ)° 4090 independent reflections with 2θ < 60° were measured of which 2901 with I > $3\sigma(I)$ were considered observed. The intensities of three standard reflections, monitored every 100 reflections, showed no

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significant variations. The data were corrected for Lorentz and polarization effects, but not for extinction and absorption [μ(Mo $K\alpha$) = 0·425 mm^{-1}, maximum effect in I less than 10%].

The structure was solved by direct methods with *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977). An E map, based on 190 phased reflections with $|E| > 1{\cdot}58$ clearly showed seven C atoms, four O, one N, one S and one Cl.

A subsequent difference Fourier synthesis revealed five C atoms. 14 H atoms were assigned calculated positions ($d_{\rm C-H}$ = 1·08 Å). Two H atoms could be located from further difference syntheses. Anisotropic temperature factors for non-hydrogen atoms and positional parameters for all atoms were refined. The isotropic temperature factor for each H atom was equal to that of the parent atom. The refinement was based upon F_o, the function minimized being $\sum w(|F_o| - |F_c|)^2$ with $w^{-1} = [\sigma^2(F_o) + (0{\cdot}011\,F_o)^2]$. The final disagreement factor $R = \sum(|F_o| - |F_c|)/\sum|F_o|$ was 0·033 and $R_w = [\sum(w|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$ = 0·034 for 2901 observed reflections. Scattering factors were taken from Cromer & Mann (1968) for C, N, O, S, Cl and from Stewart, Davidson & Simpson (1965) for H. Anomalous-dispersion corrections for S and Cl were taken from *International Tables for X-ray Crystallography* (1974).

Least-squares refinement of the enantiomer resulted in $R = 0{\cdot}035$ ($R_w = 0{\cdot}036$). According to the Hamilton (1965) test, this is a significant difference. As a proof for the correctness of the suggested absolute configuration, the observed Bijvoet differences, $\Delta F_o = |F_{o,H}| - |F_{o,\bar{H}}|$, were compared with the calculated Bijvoet differences, $\Delta F_c = |F_{c,H}| - |F_{c,\bar{H}}|$. Summation of $\Delta F_o \times \Delta F_c$ over all pairs gave values of $19{\cdot}8 \pm 3{\cdot}1$ for the model with $R = 0{\cdot}033$ and $-19{\cdot}9 \pm 3{\cdot}1$ for the enantiomer [e.s.d. based on $\sigma(F_o)$], indicating the absolute configuration as that of the model with R = 0·033. As a second check, 22 selected Bijvoet pairs were measured with Cu $K\alpha$ radiation. Comparison of observed intensities of reflections H and $\bar{H}$ with calculated values based on the model with R = 0·033, gave consistent answers for all pairs. Final coordinates are given in Table 1.*

Table 1. *Fractional atomic coordinates* (× 10^4 *for non-hydrogen atoms*; × 10^3 *for hydrogen atoms*)

E.s.d.'s (in parentheses) refer to the last decimal digit.

	x	y	z
C(1)	2623 (6)	1803 (3)	3874 (2)
C(2)	3832 (5)	2631 (2)	5703 (2)
C(3)	3675 (4)	2482 (2)	6767 (2)
C(4)	5030 (5)	1262 (3)	7195 (2)
C(5)	3275 (4)	4506 (2)	7598 (1)
C(6)	3326 (5)	6383 (3)	8552 (2)
C(7)	5536 (4)	7175 (2)	9133 (2)
C(8)	6607 (6)	6819 (3)	10047 (2)
C(9)	8701 (6)	7524 (3)	10568 (2)
C(10)	9747 (6)	8602 (3)	10171 (2)
C(11)	8680 (7)	8970 (3)	9267 (2)
C(12)	6575 (6)	8256 (3)	8750 (2)
O(1)	−661 (3)	1206 (3)	5165 (1)
O(2)	7915 (3)	1187 (2)	7114 (1)
O(3)	726 (3)	4533 (2)	7451 (1)
O(4)	4844 (3)	5398 (2)	8103 (1)
S	2364 (1)	1239 (1)	5056 (1)
Cl	662 (3)	3208 (1)	3578 (1)
N	4889 (4)	3604 (2)	7275 (1)
H(C1_1)	437 (7)	199 (3)	384 (2)
H(C1_2)	194 (7)	109 (3)	342 (2)
H(C2_1)	285 (5)	331 (3)	544 (2)
H(C2_2)	563 (6)	268 (3)	559 (2)
H(C3)	177 (5)	248 (3)	689 (2)
H(C4_1)	487 (6)	125 (3)	786 (2)
H(C4_2)	399 (6)	50 (3)	693 (2)
H(C6_1)	197 (6)	597 (3)	893 (2)
H(C6_2)	225 (6)	688 (3)	802 (2)
H(C8)	587 (6)	606 (4)	1028 (2)
H(C9)	933 (7)	726 (4)	1124 (2)
H(C10)	1115 (7)	908 (3)	1055 (2)
H(C11)	943 (7)	970 (4)	896 (3)
H(C12)	597 (7)	859 (3)	818 (2)
H(O2)	795 (7)	105 (3)	652 (2)
H(N)	658 (6)	363 (3)	740 (2)

Table 2. *Bond distances* (Å) *and angles* (°) *involving the non-hydrogen atoms*

E.s.d.'s are in parentheses.

Cl–C(1)	1·760 (5)	C(5)–O(3)	1·220 (5)
S–C(1)	1·795 (4)	C(5)–O(4)	1·341 (8)
S–O(1)	1·490 (3)	C(6) O(4)	1·457 (7)
S–C(2)	1·806 (7)	C(6)–C(7)	1·501 (12)
C(2)–C(3)	1·528 (4)	C(7)–C(8)	1·378 (10)
C(3)–C(4)	1·516 (6)	C(8)–C(9)	1·378 (11)
C(4)–O(2)	1·416 (4)	C(9)–C(10)	1·382 (6)
C(3)–N	1·453 (6)	C(10)–C(11)	1·367 (10)
N–C(5)	1·340 (6)	C(11)–C(12)	1·384 (11)
Cl–C(1)–S	112·9 (2)	O(3)–C(5)–O(4)	123·9 (2)
C(1)–S–O(1)	107·5 (2)	C(5)–O(4)–C(6)	116·0 (2)
C(1)–S–C(2)	98·0 (1)	O(4)–C(6)–C(7)	105·2 (2)
O(1)–S–C(2)	106·7 (1)	C(6)–C(7)–C(8)	121·0 (2)
S–C(2)–C(3)	110·5 (2)	C(7)–C(8)–C(9)	120·9 (2)
C(2)–C(3)–C(4)	113·7 (2)	C(8)–C(9)–C(10)	119·7 (2)
C(2)–C(3)–N	109·6 (2)	C(9)–C(10)–C(11)	119·8 (3)
C(3)–C(4)–O(2)	112·9 (2)	C(10)–C(11)–C(12)	120·0 (3)
C(3)–N–C(5)	121·2 (2)	C(11)–C(12)–C(7)	120·8 (2)
N–C(5)–O(3)	125·4 (2)	C(12)–C(7)–C(8)	118·8 (2)
N–C(5)–O(4)	110·7 (2)	C(12)–C(7)–C(6)	120·3 (2)

Discussion. Bond distances and angles are given in Table 2.

The absolute configuration is as depicted in Fig. 1, which also shows the atomic labelling. The S atom of the sulphoxide group and the chiral C atom have R chirality. This result establishes the chirality of the S

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34656 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

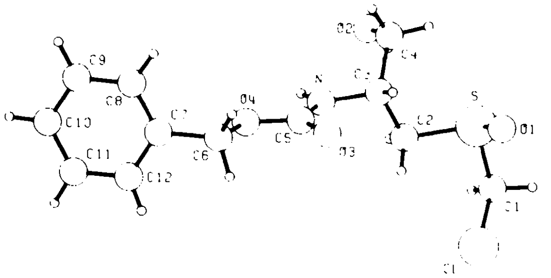

Fig. 1. Molecular conformation and atomic numbering.

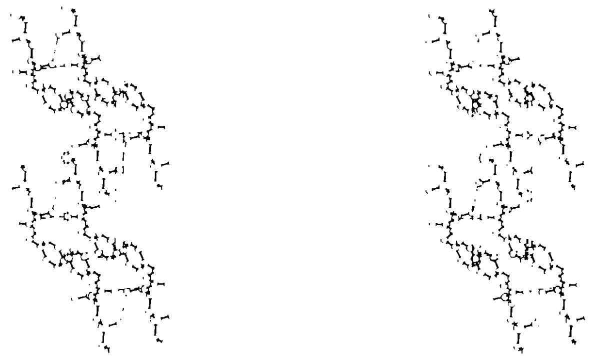

Fig. 2. A stereoscopic view of the molecular packing down the *b* axis. Hydrogen bonds are denoted as dashed lines.

atom in sparsomycin as *R*. This result will serve as a check for future measurements, based upon circular dichroism using the Cotton effect, performed by the Organic Chemistry Department at our University.

Bond distances and angles show no significant variations from the expected values. The molecular packing is determined by two hydrogen bonds: N–O(3) 2·957 Å [H(N)···O(3) 2·020 Å] and O(1)–O(2) 2·937 Å [H(O2)···O(1) 2·132 Å]. Both acceptor atoms are related to the asymmetric unit by an *a* translation, constituting a simple packing pattern of two chains in the *a* direction, related by the 2_1 axis along *b*. No other distances shorter than van der Waals contacts (Bondi, 1964) were noticed. The molecular packing is shown in Fig. 2.

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Structure de *S*-[Méthyl-1 (méthyl-3 phénylamino)-4 pyridinio-3] Isopropylcarbamoylsulfamoylate

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Abstract. $C_{17}H_{22}N_4O_3S$, $M_r = 362{\cdot}45$, orthorhombic, $P2_1ab$, $a = 13{\cdot}334\ (4)$, $b = 32{\cdot}984\ (6)$, $c = 8{\cdot}172\ (3)$ Å, $Z = 8$, $D_c = 1{\cdot}34$ Mg m^{-3}, $V = 3594{\cdot}1$ Å^3. The structure was determined by direct methods and refined by least squares using a block-diagonal-matrix approximation to a final *R* of 0·054. Two different conformations of the isopropylcarbamoylsulfamoylate group are observed, which are among the three conformations previously described for the diuretic torasemide and its derivatives. In the crystal, the only

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