Tableau 3. *Interactions intermoléculaires* (*distances intermoléculaires en* Å)

Liaisons hydrogène			
N(13)···H(24)–C(24)	2,25 (7)	N(15)···H(23)–C(23^{ii})	2,65 (8)
N(15)···H(33)–C(33^{iii})	2,46 (7)	N(16)···H(3)–C(3^{iv})	2,96 (5)
N(16)···H(2)–C(2^{iv})	2,56 (8)	N(14)···H(34)–C(34^{v})	2,82 (9)
Interactions dipôle–dipôle			
N(13)···C(13^{i})	3,271 (7)	C(13)···C(13^{i})	3,481 (8)
N(13)···N(13^{i})	3,451 (8)		
Interactions nucléophile–électrophile			
S(25)···C(13^{i})	3,581 (7)	S(32)···C(16^{i})	3,684 (8)
S(32)···C(12^{i})	3,615 (7)	S(22)···C(2^{i})	3,649 (7)
S(25)···C(9^{i})	3,654 (6)		

Code de symétrie
(i) $1-x, 1-y, 1-z$
(ii) $2-x, 1-y, 1-z$
(iii) $2+x, y, 1+z$
(iv) $2-x, -y, 2-z$
(v) $-x, -y, 1-z$

Le caractère non conducteur de ce complexes est essentiellement du aux nombreuse interactions intra- et intermoléculaires présentes dans cette structure ainsi qu'au mode alterné de l'empilement (Torrance, 1979; Soos, 1978).

Ce travail a été en partie supporté financièrement par contrat du Ministère de la Coopération en soutiênt à la Convention interuniversitaire entre les Universités d'Abidjan et d'Aix-Marseille I. Tous les calculs ont été effectués sur les ordinateurs IBM 4341 de l'Office Central de Mécanographie d'Abidjan.

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Acta Cryst. (1987). C**43**, 1552–1555

Structure of Thiamphenicol, D-*threo*-2,2-Dichloro-*N*-{2-hydroxy-1-(hydroxymethyl)-2-[4-(methylsulfonyl)phenyl]ethyl}acetamide

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(*Received* 4 *July* 1986; *accepted* 28 *January* 1987)

Abstract. $C_{12}H_{15}Cl_2NO_5S$, $M_r = 356{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}781\,(1)$, $b = 15{\cdot}323\,(1)$, $c = 17{\cdot}337\,(2)$ Å, $V = 1535{\cdot}7\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}541$, $D_m = 1{\cdot}550$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 5{\cdot}32$ mm^{-1}, $F(000) = 736$, room temperature, final $R = 0{\cdot}044$ for 1703 observed reflections. All suitable H atoms take part in a hydrogen-bonding network. The packing of the molecules in the crystal is found to be determined by the N—H···O and O—H···O hydrogen bonds.

Introduction. Thiamphenicol belongs to the chloromycetin group of drugs. Structural studies of the broad spectrum antibiotics, chloroamphenicol and a number of related compounds, have not yet led to any definite conclusion about their mode of action at the molecular level. The present compound, thiamphenicol, is less active than chloramphenicol. It has also been established that the introduction of a variety of structural variations in the side chain and at the *para* position of the phenyl ring does not lead to a complete loss of its activity (Hansch, Nakamoto, Gorin, Denisevich, Garrett, Heman-Ackah & Won, 1973). In the title compound the $-NO_2$ group of the chloramphenicol molecule, at the *p* position of the phenyl ring, has been replaced by a methylsulfonyl $-SO_2CH_3$ group. The

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compounds containing the dichloroacetamide group, $-NHCOCHCl_2$, together with the present compound, exhibit significant antifungal and antiamoebic properties (Khaskin, Shomova & Stolper, 1967). The X-ray structural study of this molecule has been carried out in order to study the effects of $-SO_2CH_3$ on the molecular geometry and the change in conformation, if any, over the chloramphenicol (Chatterjee, Dattagupta, Saha, Saenger & Muller, 1979; Ravindra Acharya, Sake Gowda & Post, 1979) molecule.

Experimental. Colourless transparent crystals from aqueous solution (at room temperature), density from benzene–bromoform mixture, symmetry from oscillation and Weissenberg photographs, space group $P2_12_12_1$, with systematic absences ($0k0$: k odd; $00l$: l odd; $h00$: h odd), crystal $0{\cdot}2 \times 0{\cdot}2 \times 0{\cdot}16$ mm was used for intensity data collection. Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Cu $K\alpha$ radiation, with ω–2θ scan technique. Cell parameters refined from setting angles of 25 reflections ($32{\cdot}0 \leq \theta \leq 36{\cdot}0°$), 1845 unique reflections ($0 \leq h \leq 7$, $0 \leq k \leq 19$, $0 \leq l \leq 21$, $1{\cdot}5 < \theta < 75{\cdot}0°$) resulted in 1703 observed reflections with $I \geq 3{\cdot}0\sigma(I)$, corrected for Lp. Three orientation control reflections (400, 095, 2,1,10) monitored after every hour of exposure, intensity variation $< 3\%$. Structure solved by direct methods (*MULTAN*78, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) with E's $> 1{\cdot}50$. Anisotropic full-matrix least-squares refinement based on F, *SHELX*76 (Sheldrick, 1976), H (from ΔF synthesis) refined isotropically, $R = 0{\cdot}044$, $wR = 0{\cdot}047$, $R_G = 0{\cdot}058$, $w = 1/[\sigma^2(|F_o|) - 0{\cdot}00047(|F|)^2]$, max. shift/e.s.d. $< 0{\cdot}30$, residual $\Delta\rho$ excursions $-0{\cdot}25$ to $+0{\cdot}22$ e Å^{-3} in final ΔF synthesis, scattering factors for the non-H atoms from Cromer & Waber (1965), for H atoms from Stewart, Davidson & Simpson (1965).

Discussion. The fractional coordinates of all the atoms with their equivalent isotropic thermal parameters are listed in Table 1.* Fig. 1 corresponds to a perspective diagram of the molecule with its atomic numbering scheme. The intramolecular bond distances, bond angles and torsion angles of the molecule are given in Table 2. The molecular dimensions of the phenyl ring are regular with a slight distortion which can be attributed to the effect of the substituent at the *para* positions of the ring. The S atom is almost coplanar with the phenyl ring as shown by the torsion angles about the C(2)—C(3) and C(2)—C(7) central bonds. The coordination of the S atom is distorted tetrahedral. The maximum distortion from the ideal tetrahedral geometry is in the O(1)—S—O(2) = 117·1 (2) and C(1)—S—C(2) = 104·1 (2)° angles. This type of distortion from the ideal configuration has been attributed by Cotton & Stokley (1970) to non-bonded interactions involving the neighbouring O atoms and has been observed in a number of sulfonamide structures (Kálmán, Czugler & Argay, 1981; Basak, Mazumdar & Chaudhuri, 1982, 1983, 1984; Basak, Chaudhuri & Mazumdar, 1984; Alléaume, Gulko, Herbstein, Kapon & Marsh, 1976, and references cited therein). The apparent shortening of the C(1)—S [1·755 (5) Å] bond over the C(2)—S [1·766 (3) Å] is due to the thermal motion of the terminal methyl carbon, C(1), which is found to be evident as these two distances are found to be nearly equal [$\{C(1)\text{—}S\}_{corr} = 1{\cdot}766$; $\{C(2)\text{—}S\}_{corr} = 1{\cdot}769$ Å] when corrected according to the riding-motion model (Busing & Levy, 1964).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters U_{eq}(Å^2) for the non-hydrogen atoms with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
S	0·3634 (2)	−0·2777 (1)	0·1459 (1)	0·0323 (2)
N	0·0912 (6)	0·1064 (2)	0·0289 (2)	0·0264 (7)
O(1)	0·5940 (6)	−0·2915 (2)	0·1180 (3)	0·0536 (10)
O(2)	0·2994 (6)	−0·3217 (2)	0·2158 (2)	0·0484 (10)
O(3)	0·3854 (6)	0·1634 (2)	0·1553 (2)	0·0380 (8)
O(4)	−0·2504 (6)	0·2411 (2)	0·0540 (2)	0·0434 (9)
O(5)	−0·1069 (6)	−0·0214 (2)	0·0199 (2)	0·0455 (9)
C(1)	0·1670 (9)	−0·3080 (3)	0·0735 (3)	0·0450 (14)
C(2)	0·3173 (7)	−0·1645 (2)	0·1574 (2)	0·0291 (10)
C(3)	0·1207 (7)	−0·1351 (2)	0·1946 (2)	0·0322 (11)
C(4)	0·0841 (7)	−0·0452 (2)	0·1998 (2)	0·0306 (9)
C(5)	0·2403 (7)	0·0136 (2)	0·1676 (2)	0·0282 (10)
C(6)	0·4354 (7)	−0·0177 (2)	0·1306 (2)	0·0342 (10)
C(7)	0·4759 (7)	−0·1073 (2)	0·1252 (2)	0·0329 (10)
C(8)	0·1861 (7)	0·1101 (2)	0·1680 (2)	0·0297 (9)
C(9)	0·0079 (6)	0·1304 (2)	0·1055 (2)	0·0266 (9)
C(10)	−0·0630 (7)	0·2263 (2)	0·1050 (2)	0·0328 (10)
C(11)	0·0226 (6)	0·0338 (2)	−0·0065 (2)	0·0293 (9)
C(12)	0·1138 (8)	0·0189 (3)	−0·0885 (2)	0·0415 (12)
Cl(1)	0·4160 (2)	0·0380 (1)	−0·0956 (1)	0·0504 (4)
Cl(2)	−0·0366 (2)	0·0894 (1)	−0·1537 (1)	0·0688 (4)

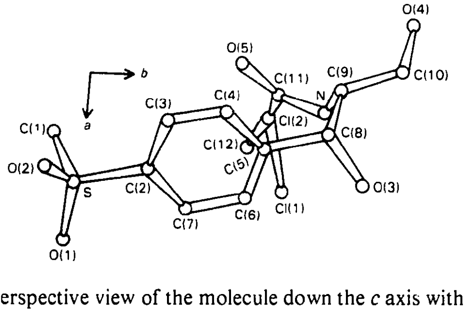

Fig. 1. Perspective view of the molecule down the c axis with atomic numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43761 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The N—C(11) = 1·331 (4) Å distance has partial double-bond character and can be explained by nitrogen lone-pair delocalization towards the carbonyl group, with an sp^2-hybridized character. The two C—Cl bond distances and the Cl—C—Cl angles agree well with the corresponding values observed in chloramphenicol (Chatterjee *et al.*, 1979; Ravindra Acharya *et al.*, 1979) and some other similar structures (Eguchi & Iitaka, 1974; De, Basak, Chaudhuri & Poddar, 1984) except for the Cl(1)—C(12) = 1·716 (6) Å bond in chloramphenicol (Chatterjee *et al.*, 1979) which is shorter. The other differences observed with respect to the chloramphenicol structure are the angles around C(11). The other molecular dimensions are comparable with the values observed in similar structures.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

S—O(1)	1·434 (4)	S—O(2)	1·435 (4)
S—C(1)	1·755 (5)	S—C(2)	1·766 (3)
N—C(9)	1·460 (5)	N—C(11)	1·331 (4)
O(3)—C(8)	1·429 (5)	O(4)—C(10)	1·417 (5)
O(5)—C(11)	1·219 (5)	C(2)—C(3)	1·382 (5)
C(2)—C(7)	1·386 (5)	C(3)—C(4)	1·397 (4)
C(4)—C(5)	1·392 (5)	C(5)—C(6)	1·383 (5)
C(5)—C(8)	1·511 (4)	C(6)—C(7)	1·396 (4)
C(8)—C(9)	1·527 (5)	C(9)—C(10)	1·526 (4)
C(11)—C(12)	1·533 (5)	C(12)—Cl(1)	1·776 (5)
C(12)—Cl(2)	1·789 (4)		

C(1)—S—C(2)	104·1 (2)	O(2)—S—C(2)	109·1 (2)
O(2)—S—C(1)	108·2 (2)	O(1)—S—C(2)	108·9 (2)
O(1)—S—C(1)	108·7 (3)	O(1)—S—O(2)	117·1 (2)
C(9)—N—C(11)	122·1 (3)	S—C(2)—C(7)	118·4 (3)
S—C(2)—C(3)	119·8 (2)	C(3)—C(2)—C(7)	121·7 (3)
C(2)—C(3)—C(4)	118·4 (3)	C(3)—C(4)—C(5)	120·9 (3)
C(4)—C(5)—C(8)	119·8 (3)	C(4)—C(5)—C(6)	119·4 (3)
C(6)—C(5)—C(8)	120·7 (3)	C(5)—C(6)—C(7)	120·6 (3)
C(2)—C(7)—C(6)	118·9 (3)	O(3)—C(8)—C(5)	113·0 (3)
C(5)—C(8)—C(9)	109·6 (3)	O(3)—C(8)—C(9)	108·5 (3)
N—C(9)—C(8)	111·8 (3)	C(8)—C(9)—C(10)	112·4 (3)
N—C(9)—C(10)	109·0 (3)	O(4)—C(10)—C(9)	111·3 (3)
N—C(11)—O(5)	126·1 (3)	O(5)—C(11)—C(12)	117·1 (3)
N—C(11)—C(12)	116·7 (3)	C(11)—C(12)—Cl(2)	109·2 (2)
C(11)—C(12)—Cl(1)	112·2 (2)	Cl(1)—C(12)—Cl(2)	109·6 (2)

Comparison of torsion angles (°)

	Present structure	Chloramphenicol	Bromamphenicol	β-Chloramphenicol palmitate
C(4)—C(5)—C(8)—C(9)	−76·7 (4)	90·6 (7)	82·3	−75·4 (2·9)
C(4)—C(5)—C(8)—O(3)	162·1 (3)	−27·3 (8)	−32·8	166·0 (2·2)
C(6)—C(5)—C(8)—O(3)	−22·7 (5)	155·5 (6)	140·9	−16·2 (3·3)
C(6)—C(5)—C(8)—C(9)	98·5 (4)	−86·5 (7)	−104·0	102·3 (2·7)
C(5)—C(8)—C(9)—N	−59·5 (4)	−54·9 (7)	−30·6	−73·2 (2·5)
C(5)—C(8)—C(9)—C(10)	177·5 (3)	180·0 (5)	−32·0	168·6 (2·2)
O(3)—C(8)—C(9)—N	64·4 (4)	66·1 (6)	80·8	46·8 (2·6)
O(3)—C(8)—C(9)—C(10)	−58·6 (4)	−59·0 (7)	79·3	−71·4 (2·7)
O(5)—C(11)—C(12)—Cl(1)	−134·0 (3)	−52·4 (8)	−65·3	−163·4 (2·1)
O(5)—C(11)—C(12)—Cl(2)	104·3 (4)	69·9 (7)	82·0	69·0 (2·9)
N—C(11)—C(12)—Cl(1)	46·5 (4)	127·2 (5)	118·8	24·7 (3·3)
N—C(11)—C(12)—Cl(2)	−75·1 (4)	−110·5 (5)	−93·9	−102·9 (2·6)
N—C(9)—C(10)—O(4)	63·4 (4)	−53·1 (7)	114·0	−47·2 (2·9)
C(8)—C(9)—C(10)—O(4)	−172·0 (3)	72·2 (7)	−69·3	69·2 (2·8)
C(9)—N—C(11)—O(5)	−2·5 (6)	3·6 (1·0)	1·6	6·3 (4·1)
C(9)—N—C(11)—C(12)	176·9 (3)	−176·0 (5)	176·9	177·9 (2·3)
C(11)—N—C(9)—C(8)	101·8 (4)	124·2 (6)	110·9	154·4 (2·4)
C(11)—N—C(9)—C(10)	−132·9 (3)	−108·8 (7)	114·2	−85·4 (3·0)
O(1)—S—C(2)—C(7)	12·8 (4)			
O(2)—S—C(2)—C(7)	141·7 (3)			
O(1)—S—C(2)—C(3)	−170·2 (3)			
O(2)—S—C(2)—C(3)	−41·4 (4)			
C(1)—S—C(2)—C(3)	73·9 (3)			
C(1)—S—C(2)—C(7)	−103·0 (3)			

The side chain of the molecule is folded back towards the phenyl ring, as is evident from the dihedral angle of 72·8 (1)° between the least-squares plane of the phenyl ring and the mean plane through the atoms N, C(8), C(9), C(10) and O(3).

The conformation of the molecule can be described by the torsion angles about the S—C(2), C(5)—C(8) and C(8)—C(9) central bonds and are compared in Table 2 with the values, calculated from the published coordinates with the numbering scheme adjusted to that of ours, in chloramphenicol (Chatterjee *et al.*, 1979), bromamphenicol (Dunitz, 1952) and the β-form of chloramphenicol palmitate (Eguchi & Iitaka, 1974). The torsion angles about the S—C(2) central bond indicate that the S—O(1) bond is eclipsed to the C(2)—C(7) bond, while the S—O(2) bond is synclinal with respect to the C(2)—C(3) bond. Similar dispositions of the sulfonyl oxygens have also been observed in a number of substituted sulfonamide structures (Kálmán *et al.*, 1981; Basak *et al.*, 1982, 1983, 1984). From the torsion angles about the S—C(2) central bond it is evident that the C(1)—S bond is almost perpendicular to the phenyl ring and the C(8)—O(3) bond is antiperiplanar with respect to the C(4)—C(5) bond of the phenyl ring. The N—C(9)—C(8)—C(5) torsion angle indicates that the N—C(9) bond is synclinal with respect to the C(8)—C(5) bond while the Cl(1)—C(12)—C(11)—N and Cl(2)—C(12)—C(11)—N torsion angles show that both the Cl(1)—C(12) and Cl(2)—C(12) bonds are in the same (synclinal) conformation with respect to the C(11)—N bond. A similar conformational feature has also been observed in chloramphenicol (Chatterjee *et al.*, 1979; Ravindra Acharya *et al.*, 1979) and β-chloramphenicol palmitate (Eguchi & Iitaka, 1974).

The only significant variation in conformation is that in the present molecule both the bonds Cl(1)—C(12)

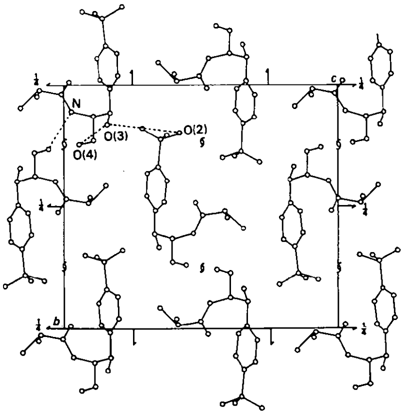

Fig. 2. The packing of the molecules as seen in projection down the *a* axis.

and Cl(2)–C(12) are anticlinal with respect to the O(5)–C(11) bond, while both these bonds are synclinal in chloramphenicol (Chatterjee *et al.*, 1979; Ravindra Acharya *et al.*, 1979) and bromamphenicol (Dunitz, 1952).

A projection of the molecular packing of the thiamphenicol molecule viewed down the *a* axis is shown in Fig. 2. The structure is found to be stabilized by intermolecular hydrogen bonds of the type N–H···O and O–H···O. The proton attached to the amide N takes part in hydrogen bonding with O(4), resulting in an infinite chain extended along the **a** direction. The other two available protons attached to the two hydroxy oxygens, O(3) and O(4), are involved in hydrogen bonding to a sulfonyl oxygen, O(2), of a screw-related molecule and to one of the hydroxy oxygens, O(3), of a translation-related molecule respectively. The geometrical details of the hydrogen bonding with their symmetry code are as follows:

A–H···B	A–H(Å)	A···B(Å)	H···B(Å)	∠A–H···B(°)
O(4)–H(41)···O(3^{i})	0·83 (12)	2·989 (5)	2·17 (12)	170 (12)
N–H(1)···O(4^{ii})	0·86 (6)	2·892 (5)	2·13 (6)	148 (6)
O(3)–H(31)···O(2^{iii})	0·82 (5)	2·892 (5)	2·07 (5)	171 (5)

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

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Acta Cryst. (1987). C**43**, 1555–1557

Structure of Thiamin Perchlorate Monohydrate

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(*Received* 11 *October* 1986; *accepted* 10 *February* 1987)

Abstract. 3-[(4-Amino-2-methyl-5-pyrimidinyl)methyl]-5-(2-hydroxyethyl)-4-methylthiazolium perchlorate monohydrate, $C_{12}H_{17}N_4OS^+.ClO_4^-.H_2O$, $M_r = 382{\cdot}83$, monoclinic, $P2_1/n$, $a = 11{\cdot}968$ (3), $b = 11{\cdot}234$ (2), $c = 12{\cdot}965$ (3) Å, $\beta = 92{\cdot}98$ (2)°, $V = 1740{\cdot}8$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}461$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3{\cdot}8$ cm^{-1}, $F(000) = 800$, $T = 300$ K. Final $R = 0{\cdot}089$ for 2082 reflections with $F_o \geq 3\sigma(F_o)$. The dimensions of the thiamin cation are not significantly different from the average values observed in other thiamin derivatives. The torsion angles in the thiamin cation are $\varphi_T = 2{\cdot}3$ (8), $\varphi_P = 83{\cdot}2$ (7), $\varphi_{5\alpha} = -17{\cdot}8$ (8) and $\varphi_{5\beta} = 54{\cdot}3$ (8)°. The φ_T and φ_P angles are consistent with the F conformation. A comparison with other thiamin derivatives suggests that $\varphi_{5\alpha}$ and $\varphi_{5\beta}$ are dependent on crystal packing and/or hydrogen bonding. The perchlorate anion has high thermal motion as is usually the case.

Introduction. Thiamin pyrophosphate is a coenzyme in a number of important biological processes. The

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0108-2701/87/081555-03$01.50