The final positional and thermal parameters for the atoms are given in Table 1.* Bond lengths and bond angles are shown in Fig. 1 along with the atomic numbering scheme for the 2-hydroxyquinoxaline molecule.

Related literature. The quinoxaline moiety, present in peptide antibiotics such as echinomycin and triostin A (Ughetto, Wang, Quigley, van der Marel, van Boom & Rich, 1985) and TANDEM (Viswamitra *et al.*, 1981), is known to intercalate bifunctionally into DNA. Hence it is of interest to obtain accurate structural parameters of the quinoxaline moiety and its chemical modifications.

* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44127 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

We thank the Departments of Science and Technology and Biotechnology, Government of India, for financial support. We thank Dr N. Borthakur, Regional Research Laboratory, Jorhat, Assam, India, for supplying us with the crystals.

References

Enraf–Nonius (1979). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. D. (1968). *Acta Cryst.* A**24**, 351–359.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

STĘPIEŃ, A., GRABOWSKI, M. J., CYGLER, M. & WAJSMAN, E. (1976). *Acta Cryst.* B**32**, 2048–2050.

UGHETTO, G., WANG, A. H. J., QUIGLEY, G. J., VAN DER MAREL, G. A., VAN BOOM, J. H. & RICH, A. (1985). *Nucleic Acids Res.* **13**, 2305–2323.

VISWAMITRA, M. A., KENNARD, O., CRUSE, W. B. T., EGERT, E., SHELDRICK, G. M., JONES, P. G., WARING, M. J., WAKELIN, L. P. G. & OLSEN, R. K. (1981). *Nature (London)*, **289**, 817–819.

Acta Cryst. (1987). C**43**, 2240–2241

Structure of the Product Formed by Reaction of (±)-Synthanecine A with Thionyl Chloride

BY ANDREW A. FREER* AND DAVID J. ROBINS

Department of Chemistry, University of Glasgow, Glasgow G12 8QQ, Scotland

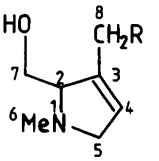
(*Received* 26 *May* 1987; *accepted* 24 *June* 1987)

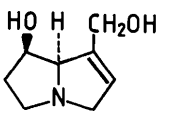
Abstract. (1): 3-Chloromethyl-2,5-dihydro-1-methyl-1*H*-pyrrole-2-methanol hydrochloride, $C_7H_{13}ClNO^+.Cl^-$, $M_r = 198{\cdot}1$, monoclinic, $P2_1/c$, $a = 7{\cdot}005$ (2), $b = 8{\cdot}685$ (2), $c = 15{\cdot}904$ (3) Å, $\beta = 91{\cdot}85$ (2)°, $V = 967{\cdot}0$ Å^3, $Z = 4$, $D_x = 1{\cdot}36$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 6{\cdot}24$ cm^{-1}, $F(000) = 416$, $T = 291$ K, final $R = 0{\cdot}068$ for 1201 observed reflections. The X-ray structure analysis of the title compound has established that treatment of synthanecine A (2) with thionyl chloride produced an allylic chloride (1) apparently by reaction of the less nucleophilic hydroxy group in (2). The Cl^- anion is chelated *via* hydrogen bonds to N and O atoms; Cl(1)···N = 3·056 (5), Cl(1)···O = 3·106 (6) Å with respective H-bond angles of 174 (4) and 172 (5)°. The stabilization due to chelation may help to account for the formation of (1) in preference to the alternative 7-chloro compound.

* To whom all correspondence should be addressed.

0108-2701/87/112240-02$01.50

Experimental. Treatment of synthanecine A (2) with thionyl chloride (Barbour & Robins, 1987) gave a chloro compound (1) isolated as the hydrochloride.





(3)

(1) R = Cl, HCl salt
(2) R = OH

Colourless, cube-shaped crystals were grown by slow evaporation from an ethanol–acetone mixture, crystal *ca* 0·4 × 0·4 × 0·3 mm used in data collection, CAD-4 diffractometer. Systematic absences from Weissenberg photographs indicated the crystals to be monoclinic $P2_1/c$. 1897 independent intensities, θ limit 26°, $\omega/2\theta$ scan. Although crystal colour changed from colourless

Table 1. *Final positional parameters and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}
Cl(1)	0·7641 (3)	0·3418 (3)	0·5897 (1)	0·060
Cl(2)	0·5214 (3)	0·3329 (3)	0·8275 (1)	0·076
O(1)	1·0535 (7)	0·5878 (7)	0·7958 (3)	0·065
N(1)	0·8020 (7)	0·8018 (5)	0·9054 (3)	0·034
C(2)	0·7446 (9)	0·6686 (7)	0·8490 (4)	0·039
C(3)	0·7452 (9)	0·5372 (7)	0·9114 (5)	0·044
C(4)	0·7511 (10)	0·5850 (9)	0·9870 (4)	0·048
C(5)	0·7500 (10)	0·7553 (8)	0·9941 (4)	0·048
C(6)	0·7283 (11)	0·9531 (7)	0·8795 (5)	0·051
C(7)	0·8742 (10)	0·6519 (9)	0·7757 (4)	0·052
C(8)	0·7413 (12)	0·3737 (8)	0·8819 (6)	0·063

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

Cl(2)–C(8)	1·779 (9)	O(1)–C(7)	1·401 (9)
N(1)–C(2)	1·510 (8)	N(1)–C(5)	1·524 (9)
N(1)–C(6)	1·466 (9)	C(2)–C(3)	1·512 (10)
C(2)–C(7)	1·508 (10)	C(3)–C(4)	1·272 (11)
C(3)–C(8)	1·494 (10)	C(4)–C(5)	1·483 (11)
C(2)–N(1)–C(5)	106·3 (5)	C(2)–N(1)–C(6)	115·8 (5)
C(5)–N(1)–C(6)	113·9 (6)	N(1)–C(2)–C(3)	101·2 (5)
N(1)–C(2)–C(7)	112·2 (6)	C(3)–C(2)–C(7)	116·4 (6)
C(2)–C(3)–C(4)	111·9 (7)	C(2)–C(3)–C(8)	120·8 (7)
C(4)–C(3)–C(8)	127·3 (8)	C(3)–C(4)–C(5)	113·5 (7)
N(1)–C(5)–C(4)	101·0 (6)	O(1)–C(7)–C(2)	114·8 (6)
Cl(2)–C(8)–C(3)	110·4 (6)		

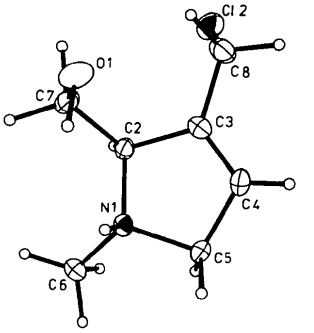


Fig. 1. *ORTEP*II (Johnson, 1976) diagram showing the numbering scheme with thermal ellipsoids at 50% probability.

to brown during data collection the two standard intensities, used to monitor variations in intensity data, recorded a mean deviation of <3% for the observed standard intensities. Least-squares techniques based on 16 reflections, $\theta > 12°$, used to refine lattice parameters. h 0 to 8, k 0 to 10, l −19 to 19. Structure solution by direct phasing techniques using *MITHRIL* (Gilmore, 1984). Full-matrix least-squares refinement on F of coordinates and anisotropic thermal parameters for non-H atoms converged to R and wR of 0·079 and 0·12 with $w = 1/\sigma^2(F_o)$. Empirical absorption correction (Walker & Stuart, 1983) was applied in which $T_{min}/T_{max} = 0·62/1·19$. Introduction of unit weights improved $w\Delta^2F$ with a corresponding improvement in e.s.d.'s and resulted in a final R and wR of 0·068 and 0·075. H-atom coordinates, located from difference Fourier maps, were included, but not refined in the final cycles of least squares. 1201 reflections, $I \geq 3·0\sigma_I$, used. $\Delta_{max}/\sigma = 0·18$, max. and min. heights in final difference Fourier synthesis of 0·29 and −0·39 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a Gould SEL 32/27 computer using Glasgow *GX* package (Mallinson & Muir, 1985). Final positional and equivalent isotropic thermal parameters are given in Table 1* while bond lengths and angles with their standard deviations are given in Table 2. An *ORTEP*II (Johnson, 1976) diagram, Fig. 1, illustrates the numbering scheme used in the analysis.

Related literature. Pyrrolizidine alkaloids are widespread and many are hepatotoxic (Robins, 1982; Mattocks, 1986). Synthanecine A (2) is a monocyclic analogue of the pyrrolizidine base retronecine (3). Macrocyclic diesters of (2) and (3) undergo similar metabolism in animals and show similar toxicity (Mattocks, Driver, Barbour & Robins, 1986). A good route to macrocyclic diesters of (3) is by conversion of the more nucleophilic primary allylic alcohol of (3) by treatment with thionyl chloride into the corresponding allylic chloride followed by reaction with an anhydride in the presence of a base (Burton & Robins, 1986). This method was recently extended to prepare macrocyclic diesters of synthanecine A (2) (Barbour & Robins, 1987). The formation of the allylic chloride (1) by treatment of (2) with thionyl chloride may proceed *via* a cyclic sulfite ester.

* Lists of structure factors, anisotropic thermal parameters, bond angles and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44185 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BARBOUR, R. H. & ROBINS, D. J. (1987). *J. Chem. Soc. Perkin Trans.* 1. Submitted.

BURTON, M. & ROBINS, D. J. (1986). *J. Chem. Soc. Perkin Trans.* 1, pp. 585–589.

GILMORE, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

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

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

MALLINSON, P. R. & MUIR, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

MATTOCKS, A. R. (1986). *Chemistry and Toxicology of Pyrrolizidine Alkaloids*. London: Academic Press.

MATTOCKS, A. R., DRIVER, H. E., BARBOUR, R. H. & ROBINS, D. J. (1986). *Chem. Biol. Interact.* **58**, 95–108.

ROBINS, D. J. (1982). *Fortschr. Chem. Org. Naturst.* **41**, 115–203.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.