BLAKE, A. J., CRADOCK, S., EBSWORTH, E. A. V., RANKIN, D. W. H. R. & WELCH, A. J. (1984). *J. Chem. Soc. Dalton Trans.* Submitted.
BLAKE, A. J., EBSWORTH, E. A. V. & WELCH, A. J. (1984). *Acta Cryst.* B**40**. To be published.
CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
HASSEL, O. & HOPE, H. (1960). *Acta Chem. Scand.* **14**, 391–397.
HOWELL, J., ROSSI, A., WALLACE, D., HARAKI, K. & HOFFMANN, R. (1977). *ICON*8. Quantum Chemistry Program Exchange.
JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
LIVANT, P., MCKEE, M. L. & WORLEY, S. D. (1983). *Inorg. Chem.* **22**, 895–901.
PANKE, D. (1968). *J. Chem. Phys.* **48**, 2990–2996.
ROBERTS, P. & SHELDRICK, G. M. (1976). *XANADU*. Univ. of Cambridge, England.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The *XRAY*76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
STRØMME, K. O. (1959). *Acta Chem. Scand.* **13**, 268–274.
VAN DER HELM, D., CHILDS, J. D. & CHRISTIAN, S. D. (1969). *J. Chem. Soc. Chem. Commun.* pp. 887–888.
WOLLRAB, J. E. & LAURIE, V. W. (1969). *J. Chem. Phys.* **51**, 1580–1583.

Acta Cryst. (1984). C**40**, 415–416

Structure of *trans*-1,9-Dichloro-1,2,3,4,6,7,8,9-octahydrophenazine 5-Oxide, $C_{12}H_{14}Cl_2N_2O$

BY Z. GAŁDECKI,* P. GROCHULSKI AND Z. WAWRZAK

Institute of General Chemistry and Institute of Physics, Technical University of Łódź, Żwirki 36, 90–924 *Łódź, Poland*

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Abstract. $M_r = 273{\cdot}025$, monoclinic, $P2_1/c$, $a = 6{\cdot}155$ (1), $b = 11{\cdot}390$ (2), $c = 20{\cdot}200$ (4) Å, $\beta = 119{\cdot}85$ (3)°, $V = 1228{\cdot}25$ (1) Å^3, $Z = 4$, $D_m = 1{\cdot}479$, $D_x = 1{\cdot}487$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 3·15 mm^{-1}, $F(000) = 568$, room temperature. Final $R = 0{\cdot}039$ for 1671 reflections. The central ring is almost flat and both unsaturated six-membered rings exist in the crystal in half-chair conformations with both C—Cl bonds lying in axial positions, in the *trans* configuration.

Introduction. 1,9-Dichloro-1,2,3,4,6,7,8,9-octahydrophenazine 5-oxide was first obtained by Fischer & Weitz (1975) as two isomers which were resolved by column chromatography on silica gel. The authors suggested that the compound with m.p. 443 K has the *cis* configuration and that with m.p. 439 K the *trans* configuration of Cl atoms. The compound was obtained again and resolved by thin-layer chromatography by Hahn, Muszkiet, Rybczyński & Skrzypek (1983). One of the isomers has m.p. 447 K and the other decomposes in the temperature range 433–448 K with no clear melting point. This does not allow identification of the isomers and we have therefore investigated the crystal structures of both isomers to identify them. We first solved the structure of the isomer having no clear melting point.

Experimental. Recrystallization from ethanol, D_m by flotation, thin colourless single-crystal fragments ~0·4 × 0·4 × 0·1 mm, Syntex $P2_1$ diffractometer, graphite-monochromatized Cu $K\alpha$ radiation, θ–2θ scan, unit-cell parameters refined from accurately measured 2θ values of 25 high-angle reflections, Lp and empirical absorption corrections (transmission 0·159 to 0·275), $\sin\theta/\lambda \le 0{\cdot}546$ Å^{-1}, 1671 reflections measured ($0 \le h \le 6$, $0 \le k \le 12$, $-22 \le l \le 19$), all unique, 1278 considered observed with $F_o \ge 2\sigma(F_o)$; three standard reflections varied in intensity by <3% throughout data collection. Direct methods [*MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978)] revealed positions of all non-hydrogen atoms; refinement (on F) by full-matrix least squares with anisotropic temperature factors for non-hydrogen atoms, all H atoms located in difference Fourier synthesis and refined isotropically using mixed method, final $R = 0{\cdot}039$, $R_w = 0{\cdot}042$, $S = 1{\cdot}53$ for the observed reflections, $w = 1/\sigma^2$, $(\Delta/\sigma)_{max}$ for non-H atoms = 0·38 [U_{12} for C(7)], $(\Delta/\sigma)_{ave} = 0{\cdot}12$, final $\Delta\rho$ excursions $\le$|0·21| e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974) for neutral atoms, and anomalous-dispersion corrections for non-hydrogen atoms from Cromer & Liberman (1970). All calculations except for *MULTAN* performed using XRAY76 (Stewart, 1976).

* To whom correspondence should be addressed at the Institute of General Chemistry.

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Discussion. A view of the molecule along [001] with the atom numbering is shown in Fig. 1. Positional and equivalent isotropic thermal parameters are given in Table 1. The bond lengths and angles are given in Table 2.* The central pyrazine ring is nearly planar and the maximum deviation from the plane is 0·012 (9) Å. Both unsaturated six-membered rings exist in half-chair conformations with asymmetry parameters (Duax & Norton, 1975) $\Delta C_2^{2,3} = 4{\cdot}9$ (8), $\Delta C_s^1 = 24{\cdot}5$ (10), $|\bar{\psi}| = 29{\cdot}8$ (12)° for the first and $\Delta C_2^{7,8} = 3{\cdot}5$ (9), $\Delta C_s^6 = 25{\cdot}0$ (10), $|\bar{\psi}| = 30{\cdot}2$ (10)° for the other ring. Both C—Cl bonds are in axial positions, in the *trans* configuration. The C—Cl bond lengths [1·826 (4) and 1·832 (5) Å] are longer than in 2,3,7,8-tetrachlorophenazine (Riganti, Locchi, Curti & Bovio, 1965*a*) [1·726 (12) and 1·731 (14) Å], containing all unsaturated rings, and in 1,4,6,9-tetrachlorophenazine (Riganti, Locchi, Curti & Bovio, 1965*b*) [1·708 (17) and 1·727 (17) Å].

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38881 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

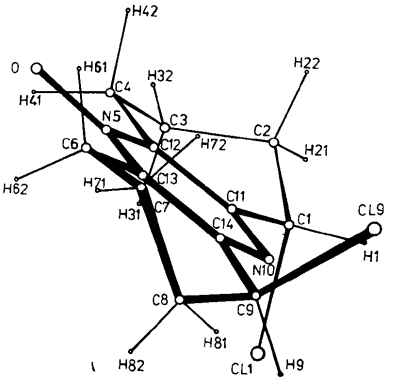

Fig. 1. View of the molecule along [001] showing the atom numbering.

Table 1. *Final atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors for* Cl, O, N *and* C *atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}(Å²)
Cl(1)	−1714 (3)	2228 (2)	275 (1)	3·8 (2)
Cl(9)	1600 (3)	3381 (2)	3726 (1)	4·1 (2)
O	4589 (6)	−827 (3)	2795 (2)	3·3 (4)
N(5)	3315 (7)	147 (4)	2582 (2)	2·3 (4)
N(10)	651 (7)	2211 (4)	2128 (2)	2·4 (4)
C(1)	1452 (8)	2436 (4)	1076 (3)	2·7 (4)
C(2)	3383 (9)	2088 (5)	850 (3)	3·1 (4)
C(3)	3486 (10)	756 (5)	781 (3)	3·4 (4)
C(4)	4217 (10)	144 (5)	1534 (3)	3·2 (4)
C(6)	2778 (10)	−79 (5)	3692 (3)	3·2 (4)
C(7)	1971 (10)	605 (5)	4179 (3)	3·6 (4)
C(8)	−516 (10)	1244 (5)	3686 (3)	3·4 (4)
C(9)	−285 (9)	2133 (4)	3158 (3)	2·8 (4)
C(11)	1696 (8)	1751 (4)	1742 (3)	2·4 (4)
C(12)	3015 (8)	706 (4)	1942 (3)	2·4 (4)
C(13)	2332 (8)	597 (4)	2999 (3)	2·4 (4)
C(14)	976 (8)	1634 (4)	2743 (3)	2·4 (2)

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

Cl(1)–C(1)	1·826 (4)	Cl(1)–C(1)–C(2)	110·4 (3)
Cl(9)–C(9)	1·832 (5)	Cl(1)–C(1)–C(11)	108·3 (4)
O–N(5)	1·302 (5)	C(2)–C(1)–C(11)	112·9 (4)
N(5)–C(12)	1·368 (7)	C(1)–C(2)–C(3)	111·1 (5)
N(5)–C(13)	1·359 (8)	C(2)–C(3)–C(4)	111·6 (5)
N(10)–C(11)	1·340 (8)	C(3)–C(4)–C(12)	111·9 (5)
N(10)–C(14)	1·331 (7)	C(7)–C(6)–C(13)	112·0 (5)
C(1)–C(2)	1·522 (9)	C(6)–C(7)–C(8)	111·4 (4)
C(1)–C(11)	1·498 (8)	C(7)–C(8)–C(9)	111·0 (5)
C(2)–C(3)	1·528 (8)	Cl(9)–C(9)–C(8)	109·1 (3)
C(3)–C(4)	1·524 (8)	Cl(9)–C(9)–C(14)	107·4 (4)
C(4)–C(12)	1·498 (9)	C(8)–C(9)–C(14)	113·0 (5)
C(6)–C(7)	1·519 (10)	N(10)–C(11)–C(1)	117·1 (4)
C(6)–C(13)	1·500 (8)	N(10)–C(11)–C(12)	123·4 (5)
C(7)–C(8)	1·532 (7)	C(1)–C(11)–C(12)	119·5 (6)
C(8)–C(9)	1·528 (8)	N(5)–C(12)–C(4)	116·7 (4)
C(9)–C(14)	1·509 (9)	N(5)–C(12)–C(11)	117·4 (5)
C(11)–C(12)	1·382 (7)	C(4)–C(12)–C(11)	125·9 (5)
C(13)–C(14)	1·389 (7)	N(5)–C(13)–C(6)	117·0 (4)
		N(5)–C(13)–C(14)	117·5 (5)
O–N(5)–C(12)	119·0 (5)	C(6)–C(13)–C(14)	125·4 (6)
O–N(5)–C(13)	119·8 (4)	N(10)–C(14)–C(9)	116·9 (4)
C(12)–N(5)–C(13)	121·1 (4)	N(10)–C(14)–C(13)	123·4 (6)
C(11)–N(10)–C(14)	117·2 (4)	C(9)–C(14)–C(13)	119·6 (5)

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References

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, pp. 16–22. New York: Plenum.

FISCHER, R. H. & WEITZ, H. M. (1975). *Synthesis*, pp. 791–796.

HAHN, W. E., MUSZKIET, B., RYBCZYŃSKI, B. & SKRZYPEK, Z. (1983). *Pol. J. Chem.* In the press.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–147. Birmingham: Kynoch Press.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York. England, and Louvain, Belgium.

RIGANTI, V., LOCCHI, S., CURTI, R. & BOVIO, B. (1965*a*). *J. Heterocycl. Chem.* **2**, 87–90.

RIGANTI, V., LOCCHI, S., CURTI, R. & BOVIO, B. (1965*b*). *J. Heterocycl. Chem.* **2**, 176–180.

STEWART, J. M. (1976). Editor, the XRAY system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.