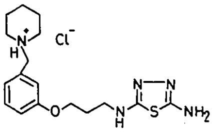
Related literature. This compound (I) was synthesized at our laboratory (Toyofuku, Tsuriya, Kuroda, Aoki & Nagasawa, 1985). It is a new competitive histamine H_2-receptor antagonist, which is eighteen times more potent than ranitidine in inhibiting gastric acid secretion in the pyrolus-ligated rat (6 h) (Tsuriya, Matsukawa, Aoki & Seya, 1984).



(I)

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Structure of 2,8-Dihydrodecachlorodihomocubane, or 2,8-Dihydromirex

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Abstract. 1,3,4,5,6,7,9,10,11,12-Decachloropentacyclo[5.3.0.$0^{2,6}$.$0^{3,9}$.$0^{4,8}$]decane, $C_{10}H_2Cl_{10}$, $M_r = 476{\cdot}7$, triclinic, $P\bar{1}$, $a = 8{\cdot}693$ (3), $b = 9{\cdot}188$ (4), $c = 10{\cdot}396$ (3) Å, $\alpha = 88{\cdot}51$ (3), $\beta = 74{\cdot}53$ (3), $\gamma = 85{\cdot}86$ (3)°, $V = 798{\cdot}2$ (7) Å^3, $Z = 2$, $D_x = 1{\cdot}98$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 17{\cdot}4$ cm^{-1}, $F(000) = 464$, $T = 293$ K, $R = 0{\cdot}030$ for 2245 unique observed reflections. There are two independent molecules in the unit cell, each at a crystallographic inversion center. The C—C—C bond angles vary from 84·7 (2) to 89·9 (2)° for the four-membered rings and from 96·0 (2) to 108·4 (2)° for the five-membered rings. The C—C bond distances range from 1·548 (3) to 1·568 (3) Å for cage C atoms with one *exo* group; the C—C distances to the C atom with the two *exo* Cl atoms are 1·512–1·517 (2) Å. The structure determination establishes the stereochemistry of the dihydro molecule which is one of the first products of chemical and photochemical degradation of the reduction-resistant insecticide mirex.

Experimental. Compound prepared by the literature reaction of dodecachlorodihomocubane (mirex) with phosphine and acetic acid as reported by Kecher, Skibinskaya, Gallai & Zefirov (1974). Crystals of $(CCl)_6(CCl_2)_2(CH)_2$ obtained from hexane solutions. Colorless data crystal 0·22 × 0·28 × 0·46 mm mounted on glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4 to 16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $17 < 2\theta < 23°$. Analytical absorption correction based on crystal shape varied from 0·80 to 1·00. Data collected in $\sin\theta/\lambda$ of 0·60 Å^{-1}, $-10 \le h \le 10$, $-10 \le k \le 10$, $0 \le l \le 12$. Four standard reflections ($2\bar{3}1$, $\bar{4}11$, 214, $\bar{1}\bar{2}4$) lost 1·1% intensity over 25·6 h of data collection; the contribution of each standard to the decay correction was weighted according to the relative distance between the reciprocal-lattice points of the standard reflections and the reflection being corrected. 2978 reflections measured, 2810 unique ($R_{\rm int} = 0{\cdot}01$), 565 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms refined with isotropic temperature factors, all other atoms refined anisotropically for 189 variables. Three reflections were removed because of extinction. $R = 0{\cdot}030$, $wR = 0{\cdot}039$, $S = 1{\cdot}67$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}009I^2]/4F^2$. Final $(\Delta/\sigma)_{\rm max} < 0{\cdot}01$, $\Delta\rho_{\rm max} = 0{\cdot}28$ (3) and $\Delta\rho_{\rm min} = -0{\cdot}51$ (3) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray*

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Crystallography (1974); programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atom coordinates and Table 2 selected bond distances and angles. Fig. 1 shows one molecule with the numbering scheme.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non-*H *atoms*

	x	*y*	*z*	*B*
Cl(1)	0·22517 (9)	−0·74806 (9)	0·60332 (8)	4·52 (2)
Cl(2)	−0·31305 (8)	−0·40775 (8)	0·70266 (7)	3·45 (2)
Cl(3)	−0·01268 (9)	−0·17642 (8)	0·62298 (9)	4·34 (2)
Cl(4)	0·32498 (8)	−0·3964 (1)	0·59320 (9)	4·97 (2)
Cl(5)	0·0534 (1)	−0·4698 (1)	0·81263 (8)	5·04 (2)
Cl(6)	0·5919 (1)	0·17733 (9)	−0·28074 (8)	4·37 (2)
Cl(7)	0·74834 (9)	−0·23824 (8)	0·03533 (8)	4·18 (2)
Cl(8)	0·69856 (9)	0·07408 (9)	0·21177 (7)	4·04 (2)
Cl(9)	0·6377 (1)	0·34080 (8)	−0·00206 (8)	4·31 (2)
Cl(10)	0·88143 (8)	0·1115 (1)	−0·11806 (9)	4·43 (2)
C(1)	0·1162 (3)	−0·5927 (3)	−0·5671 (3)	2·76 (6)
C(2)	−0·0629 (3)	−0·6075 (3)	0·5752 (3)	2·48 (5)
C(3)	−0·1327 (3)	−0·4448 (3)	0·5823 (3)	2·38 (5)
C(4)	0·0179 (3)	−0·3590 (3)	0·5760 (3)	2·61 (5)
C(5)	0·1281 (3)	−0·4543 (3)	0·6384 (3)	3·13 (6)
C(6)	0·5691 (3)	0·1012 (3)	−0·1222 (3)	2·74 (6)
C(7)	0·5720 (3)	−0·0672 (3)	−0·1095 (3)	2·53 (5)
C(8)	0·6033 (3)	−0·0985 (3)	0·0299 (3)	2·61 (5)
C(9)	0·6133 (3)	0·0572 (3)	0·0798 (3)	2·71 (5)
C(10)	0·6750 (3)	0·1534 (3)	−0·0411 (3)	3·06 (6)
H(1)	−0·116 (3)	−0·673 (3)	0·632 (2)	2·5 (6)*
H(2)	0·635 (3)	−0·119 (3)	−0·179 (2)	2·5 (6)*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. *Bond distances* (Å) *and angles* (°)

The second distance or angle given is for the corresponding parameter in independent molecule (2); C(6)—C(10) corresponds to C(1)—C(5), Cl(6)—Cl(10) to Cl(1)—Cl(5), *etc.*

C(1)—Cl(1)	1·745	1·741
C(3)—Cl(2)	1·743	1·744
C(4)—Cl(3)	1·741	1·740
C(5)—Cl(4)	1·767	1·767
C(5)—Cl(5)	1·760	1·777
C(1)—C(2)	1·552	1·549
C(2)—C(3)	1·568	1·560
C(3)—C(4)	1·563	1·550
C(4)—C(5)	1·512	1·517
C(1)—C(5)	1·513	1·512
C(1)—C(3′)	1·552	1·551
C(2)—C(4′)	1·548	1·553
C(2)—H(1)	0·90	0·90

E.s.d.'s	
C—Cl	0·002 Å
C—C	0·003 Å
C—H	0·02 Å
C—C—C	0·2°
C—C—Cl	0·2°
Cl—C—Cl	0·1°
C—C—H	1·°

Cl(1)—C(1)—C(2)	117·2	117·6
Cl(1)—C(1)—C(3′)	117·5	117·4
Cl(1)—C(1)—C(5)	116·5	116·7
Cl(2)—C(3)—C(1′)	121·0	121·1
Cl(2)—C(3)—C(2)	115·7	115·9
Cl(2)—C(3)—C(4)	121·8	121·5
Cl(3)—C(4)—C(2′)	117·5	118·6
Cl(3)—C(4)—C(3)	117·8	117·9
Cl(3)—C(4)—C(5)	116·6	116·1
Cl(4)—C(5)—C(1)	111·7	112·9
Cl(4)—C(5)—C(4)	111·8	112·0
Cl(5)—C(5)—C(1)	112·4	112·3
Cl(5)—C(5)—C(4)	112·4	111·9
C(2)—C(1)—C(3′)	84·9	85·0
C(2)—C(1)—C(5)	107·9	107·5
C(5)—C(1)—C(3′)	108·4	108·0
C(1)—C(2)—C(3)	102·9	103·7
C(1)—C(2)—C(4′)	89·9	89·3
C(3)—C(2)—C(4′)	103·3	103·9
C(2)—C(3)—C(1′)	102·0	102·0
C(4)—C(3)—C(1′)	89·4	89·3
C(2)—C(3)—C(4)	102·3	102·2
C(3)—C(4)—C(2′)	84·7	84·9
C(5)—C(4)—C(2′)	108·0	106·9
C(3)—C(4)—C(5)	107·7	108·1
C(1)—C(5)—C(4)	96·0	96·2
Cl(4)—C(5)—Cl(5)	111·7	110·8
C—C—H	117 to 122	

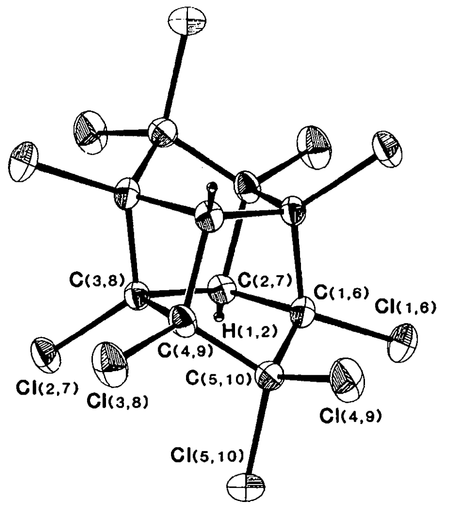

Fig. 1. *ORTEP*II diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii. Each of the two independent molecules is located on a center of symmetry. The first numbers in the parentheses are for one molecule, the second numbers are for the other molecule.

Related literature. Previous identifications of reduction products of mirex have been performed by spectroscopic methods (Kecher, Skibinskaya, Gallai & Zefirov, 1974; Alley, Layton, Minyard & Westerman, 1976; Alley, Layton & Minyard, 1974). The structure of the 5-chlorosulfonate of mirex was reported by Okaya & Bednowitz (1967).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44030 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.