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Acta Cryst. (1985). C**41**, 1079–1081

2,3,3a,4,5,8-Hexachloro-3a,6,7,7a-tetrahydro-1,6-methano-1*H*-indene (γ-Chlordene), $C_{10}H_6Cl_6$

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Abstract. $M_r = 338{\cdot}8$, monoclinic, $P2_1/n$, $a = 8{\cdot}936\,(1)$, $b = 14{\cdot}372\,(3)$, $c = 9{\cdot}774\,(2)$ Å, $\beta = 100{\cdot}72\,(1)°$, $V = 1233{\cdot}4\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}82$ Mg m^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}35$ mm^{-1}, $F(000) = 672$, $T = 293$ K, $R = 0{\cdot}039$ for 1326 unique observed reflections. The compound used was supplied as an Environmental Protection Agency (US) analytical standard. The structure is similar to the product formed by ozonolysis of γ-chlordene, except for the differences due to the effect of removal of one of the ethylene links. It is a bridged chlorinated cyclodiene but without the basic hexachloronorbornene nucleus common to cyclodiene insecticides.

Introduction. The title compound (1) is one of three bridged structural isomers designated α, β, γ of formula $C_{10}H_6Cl_6$ isolated from technical chlordane insecticide (Sovocool, Lewis, Harless, Wilson & Zehr, 1977). The crystal structure of the α-isomer has been determined (Smith, Kennard & Palm, 1981). There is 9% of the γ-isomer in the technical insecticide (Parlar, Hustert, Gäb & Korte, 1979). It is less toxic to *Musca domestica* than the α-isomer but more toxic to *Spodoptera littoralis* (Gäb, Cochrane, Parlar & Korte, 1975). The structure was determined to verify the assignment based on ^{1}H NMR and gas chromatography–mass spectroscopy evidence (Cochrane, Parlar, Gäb & Korte, 1975; Wilson & Sovocool, 1977).

Experimental. Colourless crystals, m.p. 400–401 K, obtained from isopropyl alcohol. 1955 reflections ($2\theta_{\max} = 50°$, $h\,0$–± 6, $k\,0$–16, $l\,0$–11) collected from one crystal, size $0{\cdot}28 \times 0{\cdot}21 \times 0{\cdot}13$ mm, mounted on CAD-4 diffractometer. 22 non-axial high-angle ($28 < 2\theta < 30°$) reflections used for lattice-parameter determination. Systematic absences $h0l$ ($h + l = 2n + 1$), $0k0$ ($k = 2n + 1$) indicated non-standard space group $P2_1/n$ (standard $P2_1/c$). No absorption correction. Standard reflections 246 [2260 (75)], 382 [1714 (90)], $3\bar{8}2$ [1826 (80)]; reflections measured = 1955, unique = 1726, significant = 1326 [$I > 2{\cdot}5\sigma(I)$]; time of exposure 39 h. Structure solution by multisolution $\sum_2$ sign expansion direct methods of *SHELX*76 (Sheldrick, 1976). Least-squares refinement (F's) with anisotropic thermal parameters on all non-hydrogens gave $R = 0{\cdot}039$, $wR = 0{\cdot}044$. Hydrogens located from

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Table 1. *Atomic coordinates* ($\times 10^4$; *for* H $\times 10^3$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

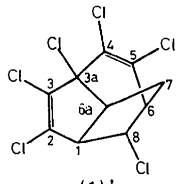
U_{eq} is defined as $(U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}
C(1)	1340 (5)	3033 (3)	889 (4)	28 (1)
C(2)	139 (5)	3689 (3)	1169 (4)	27 (2)
Cl(2)	−1370 (2)	3999·2 (7)	−96 (1)	44·1 (5)
C(3)	394 (6)	4007 (3)	2469 (4)	32 (2)
Cl(3)	−713 (2)	4778·8 (9)	3151 (1)	49·9 (5)
C(3a)	1942 (5)	3688 (3)	3223 (4)	32 (2)
Cl(3a)	1979 (2)	3392·2 (8)	5020 (1)	44·1 (5)
C(4)	3100 (5)	4443 (3)	3137 (4)	32 (2)
Cl(4)	3070 (1)	5409·5 (8)	4174 (1)	46·6 (5)
C(5)	3973 (5)	4392 (3)	2188 (4)	33 (2)
Cl(5)	5136 (2)	5306·3 (9)	1904 (1)	50·1 (5)
C(6)	4034 (6)	3535 (3)	1283 (4)	35 (2)
C(6a)	2217 (6)	2805 (3)	2376 (4)	31 (2)
C(7)	3842 (7)	2684 (4)	2226 (5)	39 (2)
C(8)	2564 (5)	3468 (3)	151 (4)	30 (2)
Cl(8)	2940 (2)	2715·0 (9)	−1223 (1)	50·0 (5)
H(1)	82 (5)	246 (3)	45 (4)	35 (12)*
H(6)	403 (12)	305 (7)	179 (10)	144 (45)*
H(7a)	467 (5)	269 (3)	318 (5)	52 (14)*
H(7b)	400 (5)	210 (3)	162 (4)	32 (12)*
H(6a)	171 (5)	228 (3)	284 (4)	41 (12)*
H(8)	224 (5)	404 (3)	−33 (4)	18 (10)*

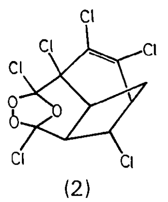
* U_{iso}.

difference-Fourier synthesis, refined isotropically. Max. and av. Δ/σ in final cycle 0·6, 0·2. Max. peak in final difference-Fourier map 0·15 e $Å^{-3}$ above background of 0·10 e $Å^{-3}$; $w = 0.8/[\sigma^2(F_o) + 0.002F_o^2]$. Table 1 lists the atomic parameters.* The computer programs used were for processing CAD-4 intensities (Hursthouse, 1976) and *SHELX*76 (Sheldrick, 1976) for structure determination with scattering factors for C, H and Cl included in program. These were run on PDP-8e and Prime 550 computers, respectively. Molecular geometry was also determined on the Prime 550 (*GEOM*; Owen, 1981).

Discussion. γ-Chlordene (1) was ozonized by Gäb, Nitz, Parlar & Korte (1976), and the structure of the compound formed (2) was determined by Born & Gäb (1978). Two crystallographically independent but structurally similar molecules were found in the asymmetric unit.



(1)†



(2)

Except for the bond changes caused by the reaction with ozone, molecules (2*a*) and (2*b*) are comparable to γ-chlordene (1) (Table 2, Fig. 1). Interatomic distances are quite similar except for C(2)—Cl(2) and C(3)—Cl(3) on either side of the ethene [C(2)=C(3)] link. The observation that the peripheral C(sp^3)—Cl bonds are longer than the peripheral C(sp^2)—Cl bonds has been noted before (Smith, Kennard & Shields, 1977). The angles associated with C(2) and C(3) also differ, *i.e.* C(2)—C(1)—C(6a), C(3)—C(3a)—C(6a), C(1)—C(2)—Cl(2), C(3a)—C(3)—Cl(3). The bridging angle C(1)—C(6a)—C(3a) is quite distorted in γ-chlordene. The torsion angles (Table 3) also reflect this effect. Intramolecular chlorine–chlorine distances are comparable.

Table 2. *Interatomic distances* (Å) *and angles* (°) (*e.s.d.'s in parentheses*) *of γ-chlordene*

C(1)—C(2)	1·491 (6)	C(5)—Cl(5)	1·729 (5)
C(2)—Cl(2)	1·710 (4)	C(5)—C(6)	1·524 (6)
C(2)—C(3)	1·330 (5)	C(6)—C(7)	1·561 (7)
C(3)—Cl(3)	1·702 (4)	C(7)—C(6a)	1·496 (8)
C(3)—C(3a)	1·512 (7)	C(6a)—C(1)	1·553 (6)
C(3a)—Cl(3a)	1·801 (4)	C(6a)—C(3a)	1·559 (5)
C(3a)—C(4)	1·514 (8)	C(6)—C(8)	1·556 (7)
C(4)—Cl(4)	1·723 (4)	C(8)—Cl(8)	1·804 (4)
C(4)—C(5)	1·319 (6)	C(8)—C(1)	1·550 (6)
C(2)—C(1)—C(6a)	102·5 (3)	Cl(4)—C(4)—C(3a)	117·4 (3)
C(2)—C(1)—C(8)	115·1 (3)	C(5)—C(4)—C(3a)	119·9 (4)
C(6)—C(1)—C(8)	104·1 (4)	Cl(5)—C(5)—C(6)	115·6 (3)
C(3)—C(2)—C(1)	113·0 (4)	Cl(5)—C(5)—C(4)	121·5 (3)
Cl(2)—C(2)—C(1)	122·0 (3)	C(6)—C(5)—C(4)	122·9 (4)
Cl(2)—C(2)—C(3)	125·0 (3)	C(5)—C(6)—C(7)	105·7 (3)
Cl(3)—C(3)—C(2)	126·1 (4)	C(5)—C(6)—C(8)	110·3 (4)
C(2)—C(3)—C(3a)	109·9 (4)	C(7)—C(6)—C(8)	101·6 (4)
Cl(3)—C(3)—C(3a)	123·5 (3)	C(3a)—C(6a)—C(1)	102·9 (3)
Cl(3a)—C(3a)—C(4)	109·6 (3)	C(3a)—C(6a)—C(7)	113·5 (4)
Cl(3a)—C(3a)—C(3)	113·3 (3)	C(1)—C(6a)—C(7)	105·3 (4)
Cl(3a)—C(3a)—C(6a)	110·4 (3)	C(6)—C(7)—C(6a)	100·5 (4)
C(4)—C(3a)—C(3)	109·2 (3)	Cl(8)—C(8)—C(6)	108·5 (3)
C(4)—C(3a)—C(6a)	112·1 (3)	Cl(8)—C(8)—C(1)	110·5 (3)
C(3)—C(3a)—C(6a)	102·0 (3)	C(6)—C(8)—C(1)	105·6 (3)
Cl(4)—C(4)—C(5)	122·3 (3)		

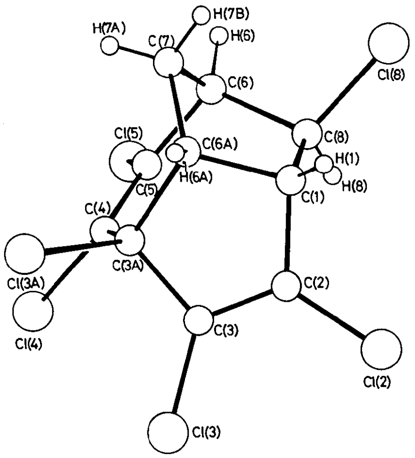


Fig. 1. Molecular configuration and atom numbering scheme for γ-chlordene.

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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 42114 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† The numbering scheme is based on the published structure of α-chlordene (Smith, Kennard & Palm, 1981).

Table 3. *Torsion angles* (°)

C(1)–C(2)–C(3)–C(3a)	+7 (1)
C(2)–C(3)–C(3a)–C(4)	+95 (1)
C(3)–C(3a)–C(4)–C(5)	−98 (1)
C(3a)–C(4)–C(5)–C(6)	−9 (1)
C(4)–C(5)–C(6)–C(7)	−35 (1)
C(5)–C(6)–C(7)–C(6a)	+69 (1)
C(6)–C(7)–C(6a)–C(1)	+45 (1)
C(7)–C(6a)–C(1)–C(2)	−146 (1)
C(6a)–C(1)–C(2)–C(3)	+13 (1)
C(8)–C(1)–C(2)–C(3)	−99 (1)
C(2)–C(3)–C(3a)–C(6a)	−24 (1)
C(3)–C(3a)–C(6a)–C(1)	+30 (1)
C(3a)–C(6a)–C(1)–C(2)	−27 (1)
C(4)–C(5)–C(6)–C(8)	+74 (1)
C(5)–C(6)–C(8)–C(1)	−81 (1)
C(6)–C(8)–C(1)–C(6a)	−4 (1)
C(6)–C(8)–C(1)–C(2)	+107 (1)
C(8)–C(1)–C(6a)–C(3a)	+94 (1)
C(8)–C(1)–C(6a)–C(7)	−26 (1)
C(5)–C(4)–C(3a)–C(6a)	+14 (1)
C(4)–C(3a)–C(6a)–C(7)	+27 (1)
C(3a)–C(6a)–C(7)–C(6)	−67 (1)
C(8)–C(6)–C(7)–C(6a)	−46 (1)
C(7)–C(6)–C(8)–C(1)	+31 (1)
C(4)–C(3a)–C(6a)–C(1)	−86 (1)
C(3)–C(3a)–C(6a)–C(7)	+144 (1)

Fig. 2 shows the packing arrangement for the racemate in the crystal. Short intermolecular distances across centres of symmetry (−*x*, 1 − *y*, −*z*) are Cl(2)–Cl(3) 3·552 (2), Cl(2)–Cl(4) 3·335 (2), Cl(2)–Cl(5) 3·467 (2); and (−*x*, 1 − *y*, 1 − *z*) Cl(3)–Cl(3a) 3·484 (2) Å. These distances are of the same order as for (2).

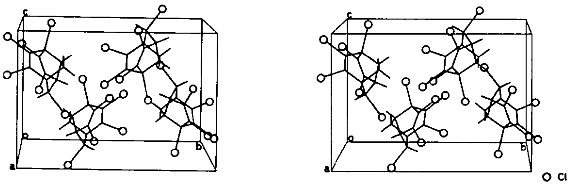

Fig. 2. Stereoview perpendicular to the *bc* plane.

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Structure of 7-Chloro-1-methyl-5-phenyl-1*H*-1,5-benzodiazepine-2,4(3*H*,5*H*)-dione (Clobazam), $C_{16}H_{13}ClN_2O_2$

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Abstract. M_r = 300·74, trigonal, $R\bar{3}$, *a* = 14·656 (5) Å, α = 111·75 (4)°, *V* = 2195·3 Å^3, *Z* = 6, D_x = 1·365 Mg m^{-3}, μ(Mo *K*α, λ = 0·71069 Å) = 0·22 mm^{-1}, *F*(000) = 936, room temperature, *R* = 0·043 for 1588 observed reflections. The seven-membered ring adopts a slightly distorted cycloheptatriene-like boat conformation, the C—N amide bonds of length 1·361 (3) and 1·360 (3) Å acting geometrically as double bonds. The angle between the phenyl ring and the fused benzene moiety is 85·2 (4)°.