

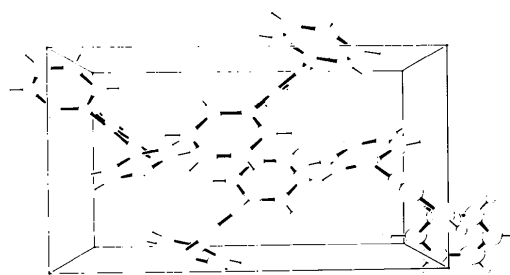
Table 3. Bond angles ( $^{\circ}$ ) with standard deviations in parentheses

N(8)—N(1)—C(2)	101.7 (3)	N(1)—N(8)—C(7)	128.3 (3)
N(1)—C(2)—N(3)	117.3 (4)	N(3)—C(3a)—N(4)	127.7 (4)
C(2)—N(3)—C(3a)	101.8 (3)	N(5)—C(6)—C(9)	114.1 (3)
N(3)—C(3a)—N(8)	110.0 (3)	C(7)—C(6)—C(9)	122.8 (3)
C(3a)—N(8)—N(1)	109.2 (3)	C(6)—C(7)—C(15)	126.5 (3)
N(4)—C(3a)—N(8)	122.2 (3)	N(8)—C(7)—C(15)	120.2 (3)
C(3a)—N(4)—N(5)	117.0 (3)	C(6)—C(9)—C(10)	120.6 (2)
N(4)—N(5)—C(6)	122.0 (3)	C(6)—C(9)—C(14)	119.4 (2)
N(5)—C(6)—C(7)	123.1 (3)	C(7)—C(15)—C(16)	121.8 (2)
C(6)—C(7)—N(8)	113.1 (3)	C(7)—C(15)—C(20)	118.2 (2)
C(7)—N(8)—C(3a)	122.4 (3)		

itself (III) (Goldstein, Ladell & Abowitz, 1969). However, the bridge bond C(3a)—N(8) in (Ia) at 1.379 Å is considerably longer than in either single ring [1.339 Å in (II), 1.326 Å in (III)], and the opposite angle N(1)—C(2)—N(3) is correspondingly  $2.7^{\circ}$  larger in (Ia) than in (III). All ring bonds are intermediate between single and double.

The packing of molecules in the unit cell is illustrated in Fig. 2. The  $\pi$ -deficient triazine portion of the heterocycle is covered by a phenyl ring from a neighbouring molecule at  $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$  with contacts N(8)  $\cdots$  C(13') 3.379 Å and C(3a)  $\cdots$  C(12') 3.437 Å. However, the least-squares planes of the two rings are not parallel but intersect at  $25.6^{\circ}$ . Other intermolecular contacts are normal throughout.

We thank Dr T. A. Hamor for diffractometer facilities and the 3M Company for a research studentship (to E.J.G.). Most computations were carried out at the University of Aston Computer Centre, the University of Manchester Regional Computer Centre, and the

Fig. 2. Projection of the unit-cell contents along  $a$ .

Atlas Computer Laboratory. The initial direct methods work was kindly done by Dr G. M. Sheldrick.

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## A Toxaphene Component: 2,5,6-*exo*,8,8,9,10-Heptachlorodihydrocamphene

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**Abstract.** C<sub>10</sub>H<sub>11</sub>Cl<sub>7</sub>, triclinic,  $P\bar{1}$ ,  $a = 8.745$  (1),  $b = 11.156$  (1),  $c = 7.942$  (5) Å,  $\alpha = 99.83$  (3),  $\beta = 114.64$  (3),  $\gamma = 85.16$  (3) $^{\circ}$ ,  $Z = 2$ ,  $D_m$  (room temperature) = 1.76,  $D_x$  (85 K) = 1.81 g cm<sup>-3</sup>. The structure was determined from diffractometer data (Mo K $\alpha$ ) measured at 85 K, and refined (anisotropic  $B$ 's for

Table 1. *Final positional and thermal ( $\text{\AA}^2$ ) parameters*

Standard deviations are in units of the last digit. The anisotropic temperature factor is  $\exp[-\frac{1}{4}(a^*h^2 B_{11} + \dots + 2b^*c^*klB_{23})]$ .

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{11}$	$B_{22}$	$B_{33}$	$B_{12}$	$B_{13}$	$B_{23}$
Cl(11)	0.29746 (5)	0.48817 (4)	0.15457 (6)	1.14 (2)	0.92 (2)	1.13 (2)	-0.13 (1)	0.39 (1)	0.13 (1)
Cl(12)	0.01625 (6)	0.06312 (4)	0.32003 (7)	2.56 (2)	1.49 (2)	2.02 (2)	-0.49 (2)	1.56 (2)	0.20 (2)
Cl(13)	-0.21010 (6)	0.19160 (4)	-0.02245 (7)	1.04 (2)	1.53 (2)	2.43 (2)	-0.25 (1)	0.86 (2)	-0.06 (2)
Cl(14)	0.04601 (5)	0.35635 (4)	-0.27065 (6)	1.12 (2)	2.42 (2)	0.77 (2)	0.43 (1)	0.28 (1)	0.45 (1)
Cl(15)	0.40742 (5)	0.32712 (4)	-0.14272 (6)	1.10 (2)	2.08 (2)	1.09 (2)	-0.03 (1)	0.63 (1)	0.41 (1)
Cl(16)	0.67327 (5)	0.36453 (4)	0.34677 (6)	0.97 (2)	1.89 (2)	1.28 (2)	-0.27 (1)	0.19 (1)	0.07 (1)
Cl(17)	0.55723 (6)	0.03487 (4)	0.31709 (6)	1.87 (2)	1.44 (2)	1.57 (2)	0.47 (1)	0.56 (2)	0.64 (1)

Table 1 (*cont.*)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
C(1)	0.0774 (2)	0.3211 (2)	0.1120 (2)	0.94 (3)
C(2)	0.2458 (2)	0.3288 (2)	0.0934 (2)	0.87 (3)
C(3)	0.3706 (2)	0.2470 (2)	0.2453 (2)	0.95 (3)
C(4)	0.2522 (2)	0.2134 (2)	0.3341 (2)	1.06 (3)
C(5)	0.1242 (2)	0.1177 (2)	0.1989 (2)	1.16 (3)
C(6)	0.0110 (2)	0.1903 (2)	0.0379 (2)	1.04 (3)
C(7)	0.1372 (2)	0.3261 (2)	0.3236 (2)	1.09 (3)
C(8)	0.2296 (2)	0.2902 (2)	-0.1094 (2)	0.98 (3)
C(9)	0.5162 (2)	0.3165 (2)	0.4095 (3)	1.20 (3)
C(10)	0.4406 (2)	0.1369 (2)	0.1536 (3)	1.09 (3)
H(11)	-0.0021 (22)	0.3794 (16)	0.0546 (24)	0.58 (34)
H(41)	0.3225 (23)	0.1861 (17)	0.4551 (27)	1.33 (39)
H(51)	0.1731 (24)	0.0465 (18)	0.1542 (27)	0.65 (40)
H(61)	0.0187 (23)	0.1528 (17)	-0.0756 (27)	1.31 (38)
H(71)	0.1926 (24)	0.3987 (17)	0.3942 (27)	1.21 (39)
H(72)	0.0452 (24)	0.3125 (17)	0.3541 (26)	0.98 (38)
H(81)	0.2199 (19)	0.2075 (14)	-0.1421 (21)	0.23 (30)
H(91)	0.5767 (22)	0.2603 (16)	0.4969 (26)	0.89 (37)
H(92)	0.4742 (22)	0.3851 (17)	0.4636 (25)	0.66 (36)
H(101)	0.5151 (23)	0.1603 (16)	0.1159 (24)	0.67 (36)
H(102)	0.3548 (24)	0.0886 (17)	0.0532 (27)	1.27 (39)

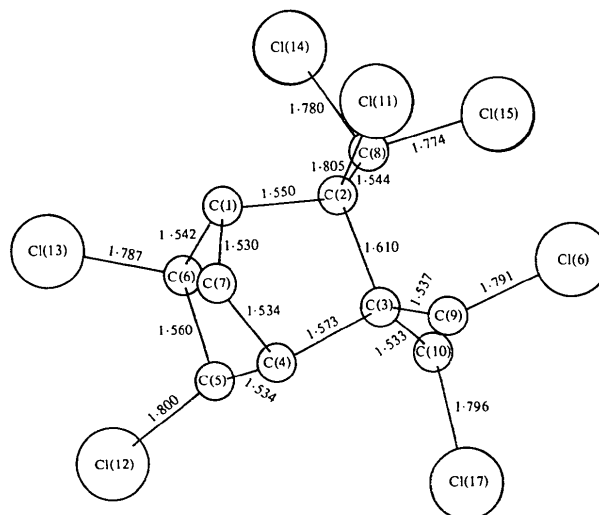


Fig. 1. C—C and C—Cl distances. E.s.d.'s are about 0.003 Å for C—C and 0.002 Å for C—Cl.

Cl, isotropic  $B$ 's for C and H) to  $R = 0.022$  for 2576 reflections. The molecule has an unusually long bond [1.610 (2) Å] between C atoms 2 and 3.

**Introduction.** 2,5,6-*exo*,8,8,9,10-Heptachlorodihydrocamphene (Fig. 1) was isolated by column chromatography and preparative gas chromatography from the insecticide toxaphene (Seiber, Landrum, Madden, Nugent & Winterlin, 1975) and recrystallized from hexane-ether (5:1). The crystals were large colorless parallelepipeds which were subsequently cut to a reasonable size. Exploratory oscillation and Weissenberg photographs taken at room temperature with Cu  $K\alpha$  radiation showed triclinic symmetry. Cooling to just above liquid nitrogen temperature caused no apparent phase change, indicating that the data could be collected at low temperature (85 K). The crystal used for intensity data had dimensions 0.35 × 0.20 × 0.09 mm; attached to it was a fragment amounting to about 1% of the main crystal, and misaligned with its  $c^*$  axis about 3° from the main  $c^*$ .

Cell dimensions were obtained from measurements with Mo  $K\alpha_1$  ( $\lambda = 0.71069$  Å) and Mo  $K\bar{\alpha}$  wave-

lengths. The reduced cell was determined by use of the program *TRACER II* (Lawton, 1973).

Intensity data up to  $2\theta = 55^\circ$  were measured with a Picker automated diffractometer by the  $\theta$ - $2\theta$  scan method ( $2^\circ \text{ min}^{-1}$  in  $2\theta$ , scan range  $2\theta_{\alpha_1} - 0.8^\circ$  to  $2\theta_{\alpha_2} + 0.8^\circ$ ) with graphite-monochromatized Mo  $K\alpha$  radiation, scintillation detector, pulse-height analyzer, automatic attenuation to below 10 000 c s $^{-1}$  and 20 s background counts at the extremes of each scan. A monitor reflection gradually suffered a 3% decline in intensity. The 2863 reflections measured were corrected for background, attenuation, and Lorentz and polarization effects. Estimated standard deviations were calculated for each recorded count ( $N$ ) using the formula  $s(N) = [N + (0.008N)^2]^{1/2}$ . The factor 0.008 reproduces the variance in the monitor reflection, uncorrected for decay.

Intensity statistics indicated the space group to be  $P\bar{1}$  and later refinement confirmed this choice. Long's (1965) sign-predicting program gave signs to 303  $E$ 's greater than 1.65. Cl positions determined from the  $E$  map produced a clear electron density map with all Cl

Table 2. Bond angles ( $^{\circ}$ )

C(2)—C(1)—C(6)	109.1 (0.1)	C(6)—C(5)—Cl(12)	114.7 (0.1)	C(5)—C(6)—H(61)	109.0 (1.2)
C(2)—C(1)—C(7)	101.8 (0.1)	C(1)—C(6)—C(5)	103.5 (0.1)	Cl(13)—C(6)—H(61)	102.9 (1.3)
C(6)—C(1)—C(7)	101.8 (0.1)	C(1)—C(6)—Cl(13)	109.3 (0.1)	C(1)—C(7)—H(71)	113.7 (1.2)
C(1)—C(2)—C(3)	102.3 (0.1)	C(5)—C(6)—Cl(13)	116.5 (0.1)	C(1)—C(7)—H(72)	110.7 (1.1)
C(1)—C(2)—C(8)	112.9 (0.1)	C(1)—C(7)—C(4)	94.4 (0.1)	C(4)—C(7)—H(71)	114.9 (1.3)
C(1)—C(2)—Cl(11)	105.3 (0.1)	C(2)—C(8)—Cl(14)	110.8 (0.1)	C(4)—C(7)—H(72)	111.7 (1.1)
C(3)—C(2)—C(8)	116.0 (0.1)	C(2)—C(8)—Cl(15)	113.5 (0.1)	H(71)—C(7)—H(72)	110.6 (1.7)
C(3)—C(2)—Cl(11)	113.4 (0.1)	Cl(14)—C(8)—Cl(15)	109.2 (0.1)	C(2)—C(8)—H(81)	110.6 (1.0)
C(8)—C(2)—Cl(11)	106.5 (0.1)	C(3)—C(9)—Cl(16)	114.1 (0.1)	Cl(14)—C(8)—H(81)	107.1 (1.0)
C(2)—C(3)—C(4)	101.3 (0.1)	C(3)—C(10)—Cl(17)	111.8 (0.1)	Cl(15)—C(8)—H(81)	105.4 (1.1)
C(2)—C(3)—C(9)	114.8 (0.1)	C(2)—C(1)—H(11)	112.9 (1.2)	C(3)—C(9)—H(91)	107.6 (1.1)
C(2)—C(3)—C(10)	111.7 (0.1)	C(6)—C(1)—H(11)	112.5 (1.1)	C(3)—C(9)—H(92)	109.9 (1.2)
C(4)—C(3)—C(9)	104.8 (0.1)	C(7)—C(1)—H(11)	117.6 (1.1)	Cl(16)—C(9)—H(91)	103.6 (1.3)
C(4)—C(3)—C(10)	114.3 (0.1)	C(3)—C(4)—H(41)	108.6 (1.3)	Cl(16)—C(9)—H(92)	108.6 (1.3)
C(9)—C(3)—C(10)	109.6 (0.2)	C(5)—C(4)—H(41)	112.5 (1.1)	H(91)—C(9)—H(92)	113.0 (1.6)
C(3)—C(4)—C(5)	110.7 (0.1)	C(7)—C(4)—H(41)	121.0 (1.2)	C(3)—C(10)—H(101)	110.8 (1.2)
C(3)—C(4)—C(7)	102.9 (0.1)	C(4)—C(5)—H(51)	114.3 (1.3)	C(3)—C(10)—H(102)	113.1 (1.3)
C(5)—C(4)—C(7)	100.5 (0.2)	C(6)—C(5)—H(51)	112.1 (1.2)	Cl(17)—C(10)—H(101)	104.5 (1.2)
C(4)—C(5)—C(6)	102.1 (0.1)	Cl(12)—C(5)—H(51)	104.7 (1.3)	Cl(17)—C(10)—H(102)	105.2 (1.2)
C(4)—C(5)—Cl(12)	109.2 (0.1)	C(1)—C(6)—H(61)	116.0 (1.2)	H(101)—C(10)—H(102)	111.0 (1.7)

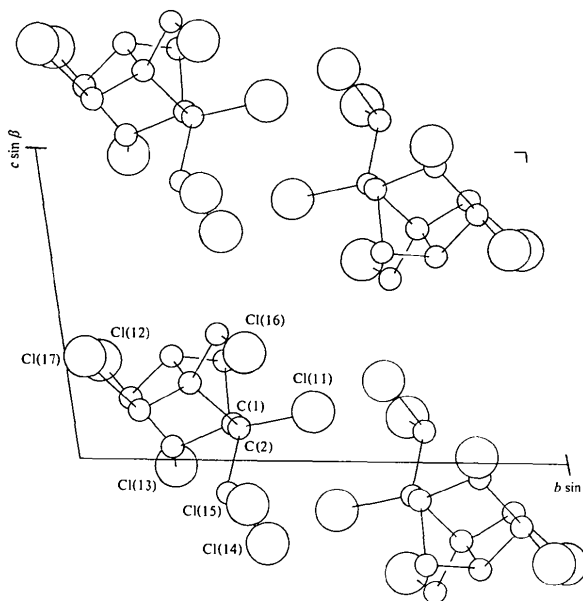
Table 3. C—H distances ( $\text{\AA}$ )

C(1)—H(11)	0.936 (18)	C(8)—H(81)	0.916 (16)
C(4)—H(41)	0.985 (19)	C(9)—H(91)	0.970 (19)
C(5)—H(51)	0.956 (21)	C(9)—H(92)	0.936 (19)
C(6)—H(61)	0.953 (20)	C(10)—H(102)	0.955 (20)
C(7)—H(71)	0.943 (20)	C(10)—H(101)	0.894 (21)
C(7)—H(72)	0.961 (22)		

and C atoms visible. Two cycles of isotropic full-matrix least-squares refinement, minimizing the quantity  $\sum (F_o - F_c)^2 / \sigma^2(F_o)$  for 2576 reflections with  $I_{net}/\sigma I_{net} > 3$ , reached an  $R$  of 0.087. H atom positions were then predicted using our program *HPOSN*. Further refinement with anisotropic thermal parameters for Cl and isotropic parameters for C and H converged with  $R = 0.022$ . \* Scattering factors for C and Cl are from Doyle & Turner (1968) and for H from Stewart, Davidson & Simpson (1965). Calculations were performed on a CDC 7600 computer using a program library developed in this laboratory. The atomic parameters are listed in Table 1. C—C and C—Cl distances are shown in Fig. 1. All bond angles are given in Table 2, and C—H distances in Table 3.

**Discussion.** We are interested in the title compound because of its presence in the insecticide toxaphene, one of the few chlorinated hydrocarbon insecticides still in use in the United States. Toxaphene is composed of

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33717 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 2. Projection of the structure along  $a$ .

approximately 175 polychlorinated compounds (Casida, Holmstead, Khalifa, Palmer & Wong, 1974) of which the present one constitutes 0.8% by weight (Landrum, Pollock, Seiber, Hope & Swanson, 1976). 2,2,5-*endo*,6-*exo*,6,8,10-Heptachloroborane is the only other toxic component which has been identified by X-ray diffraction (Palmer, Wong, Lundin, Khalida & Casida, 1975). The structure of the heptachlorodihydrocamphene reported here could not be conclusively determined using mass spectrometry and proton magnetic resonance spectroscopy.

The most interesting feature of the molecule is the abnormally long [1.610 (2)  $\text{\AA}$ ] C(2)—C(3) bond. This

large deviation from a normal  $sp^3$  C—C distance is apparently caused by the eclipsed (within  $4^\circ$ ) conformation of bulky C(2) and C(3) substituents, including a Cl atom and a dichloromethyl group at C(2) and two chloromethyl groups at C(3). Several other distances within the ring system are somewhat longer than normal, with no main contributing cause immediately apparent. The nonbonded C(1)—C(4) distance is only 2.25 Å (common for this type of ring system), suggesting the beginning of an approach to a bonding situation.

A projection of the unit-cell contents is shown in Fig. 2.

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## The Imidazole Complex of 2-Hydroxy-4,4,5,5-tetramethyl-1,3,2-dioxaphospholane 2-Sulphide\*

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**Abstract.**  $C_6H_{13}O_3PS$ .  $C_3H_4N_2$ , monoclinic,  $P2_1/c$ ,  $a = 13.229$  (3),  $b = 8.585$  (2),  $c = 12.456$  (3) Å,  $\beta = 108.31$  (2)°,  $Z = 4$ ,  $D_m = 1.318$ ,  $D_x = 1.314$  g cm $^{-3}$ ,  $F(000) = 560$ . The dioxaphospholane ring is in the half-chair conformation with C(1) and C(2) 0.373 and –0.236 Å from the O(2), P, O(3) plane. Asymmetry parameters  $\Delta C_2^P = 5.1^\circ$  and  $\Delta C_5^{C(2)} = 10.7^\circ$  are

observed. The imidazole and dioxaphospholane systems are linked into infinite chains through two strong hydrogen bonds O(1)⋯H(1)—N(1) 2.72 and O(1)—H(2)⋯N(2) 2.79 Å.

**Introduction.** Cell dimensions were obtained from a least-squares fit to the settings for 15 reflexions ( $\pm hkl$ ) on a Syntex  $P2_1$  diffractometer (Cu  $K\alpha$ ,  $\lambda = 1.54178$  Å). Intensity data were collected in the  $\theta$ – $2\theta$  mode ( $3.0 \leq 2\theta \leq 135.0^\circ$ ) with graphite-monochromated Cu  $K\alpha$  radiation. No absorption correction was applied [ $\mu(\text{Cu } K\alpha) = 31.1$  cm $^{-1}$ ]. After application of the acceptance

\* Stereochemistry of Organophosphorus Cyclic Compounds. VIII.

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