

1967; Gurtu, Chandler & Robertson, 1975; Cody, Shefter & Lehmann, 1976), and are presumably to avoid the short contact with the *ortho* O atom [O(10) in this structure].

Changes in the C—O lengths in the direction observed here also occur in acetal cleavage reactions, one bond becoming a double bond and the other being broken (Kirby & Martin, 1978). Compounds with more electronegative leaving groups may thus be considered to start (*i.e.* have ground states) farther along the reaction coordinate; we have recently demonstrated a simple relationship between bond length and reactivity in these compounds (Jones & Kirby, 1979).

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D. S. Motherwell; all other crystallographic programs used were written by Professor G. M. Sheldrick.

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2,2,3,3,4,6,7-Heptachloro-1,1,5-triethoxyindan

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Abstract. C₁₅H₁₅Cl₇O₃, *M_r* = 491.46, *F*(000) = 1984, orthorhombic, *P*2₁2₁2₁, *a* = 11.677 (2), *b* = 16.381 (8), *c* = 10.579 (2) Å, *V* = 2023.6 Å³, *Z* = 4, *D_c* = 1.61 Mg m⁻³, λ(Mo Kα) = 0.7107 Å. The structure was solved with *MULTAN* and refined by the block-diagonal least-squares method. The final *R* value is 0.040 for 2204 observed reflections. The five-membered ring is puckered with an 'envelope form'; moreover, bond distances are slightly influenced by the steric interaction between O and Cl substituents.

Introduction. Colourless prismatic crystals of this compound were kindly supplied by Professors Ballester and Riera (Ballester, Riera, Castañer & Juliá, 1978; Ballester & Olivella, 1974).

The structure was determined to locate the positions of the ethoxy groups in the molecule and to obtain molecular structural information as an aid to the elucidation of structure–toxicity relationships in this group of insecticides.

A crystal of 0.2 × 0.2 × 0.4 mm was used to obtain crystal data and intensity measurements on a Nonius CAD-4 diffractometer. The unit cell was determined by centring 25 independent reflections and refining the

Table 1. *Non-hydrogen atomic coordinates* (× 10⁵) *with standard deviations in parentheses*

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	38678 (31)	59130 (21)	78942 (43)
C(2)	31430 (31)	43820 (22)	76920 (42)
C(3)	39563 (33)	36927 (21)	81824 (45)
C(4)	52724 (32)	37793 (23)	101663 (47)
C(5)	58088 (32)	42757 (27)	110544 (46)
C(6)	57061 (36)	51249 (28)	109308 (46)
C(7)	50924 (33)	54731 (22)	99429 (45)
C(8)	45403 (30)	49681 (22)	90813 (43)
C(9)	46123 (29)	41169 (22)	92258 (42)
O(10)	45304 (25)	52388 (16)	68071 (30)
C(11)	54782 (40)	57914 (28)	68110 (56)
C(12)	59902 (43)	57483 (31)	54999 (54)
O(13)	32392 (24)	58894 (16)	81442 (32)
C(14)	25605 (46)	62279 (30)	71318 (60)
C(15)	22278 (50)	70660 (31)	75512 (73)
Cl(16)	19202 (8)	44441 (6)	86292 (13)
Cl(17)	26667 (9)	42044 (6)	61458 (11)
Cl(18)	49528 (10)	33761 (6)	70026 (13)
Cl(19)	31753 (10)	28210 (5)	86533 (14)
Cl(20)	55058 (11)	27403 (6)	102765 (15)
O(21)	64898 (27)	39563 (23)	119679 (35)
C(22)	58963 (45)	37262 (41)	131103 (61)
C(23)	67786 (66)	34528 (45)	140415 (65)
Cl(24)	64038 (13)	57249 (8)	120188 (14)
Cl(25)	50531 (10)	65211 (5)	98219 (13)

orientation matrix and unit-cell parameters by least squares. Intensities were collected with Mo $K\alpha$ radiation, monochromatized by reflection from a graphite crystal, at a take-off angle of 5° . The ω - 2θ technique was used with scan interval $\Delta\omega = (0.70 + 0.35 \tan \theta)$. The background was measured before and after each reflection. The maximum scan time was 80 s, with 53.3 s used for the peak scanning and 13.3 s for both backgrounds. The intensity of 014 was chosen as standard and was measured every hour. The reflections 754, 811, 864 chosen as orientation controls were measured every 50 reflections. 2729 independent reflections were collected within the range $1 < \theta \leq 30^\circ$, 2204 of which have been considered as observed, applying the condition $I > 2\sigma(I)$. The intensities were corrected for Lorentz and polarization effects only.

The structure was solved with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974), and an *E* map computed with the phases from the solution

with the highest combined figure of merit revealed peaks for 21 of the heaviest atoms.

The structure was isotropically refined by means of block-diagonal least squares with the modified version of *SFLS* (Ahmed, Hall, Pippy & Huber, 1966) written by Solans. The function minimized was $w||F_o| - |F_c||^2$, where $w = (4.0 + |F_o| + 0.01|F_o^2|)^{-1/2}$. A subsequent difference map revealed peaks for the four missing C atoms. After some anisotropic cycles of refinement, the hydrogen atoms were obtained by a new difference map.

The refinement was terminated at $R = 0.040^*$ for all observed reflections. The final non-hydrogen atomic parameters are listed in Table 1. Fig. 1 shows a view of the molecule and the numbering of the atoms, drawn by the *ORTEP* program (Johnson, 1965). The molecular geometry calculations were performed with the *CGMC* computer program (Solans & Font-Altaba, 1975). The bond distances and angles are shown in Figs. 2 and 3 respectively.

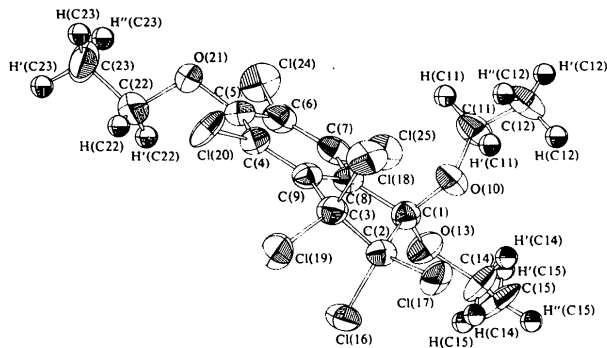


Fig. 1. A view of the molecule, drawn by the *ORTEP* program, showing thermal ellipsoids and the numbering of the atoms.

Discussion. The benzene ring is nearly planar [the largest atom-mean-plane distance is 0.026 (4) Å in C(9)]. The C-C distance (mean value = 1.392 Å), and C-C-C angles (120.0°) are typical values for a benzene ring. The C(8)-C(1) and C(9)-C(3) distances (1.519 Å) and the C(1)-C(8)-C(9)-C(3) torsion angle (2.3°) are typical of $C(sp^3)$ -C(aromatic) bonds.

* Lists of anisotropic temperature factors, fractional coordinates for the H atoms, structure factors, least-squares mean planes, distances and bond angles involving hydrogen atoms, have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34057 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

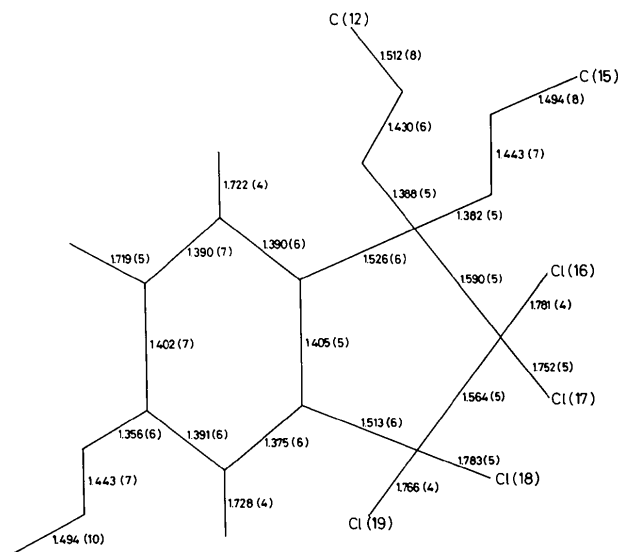


Fig. 2. Bond lengths (Å).

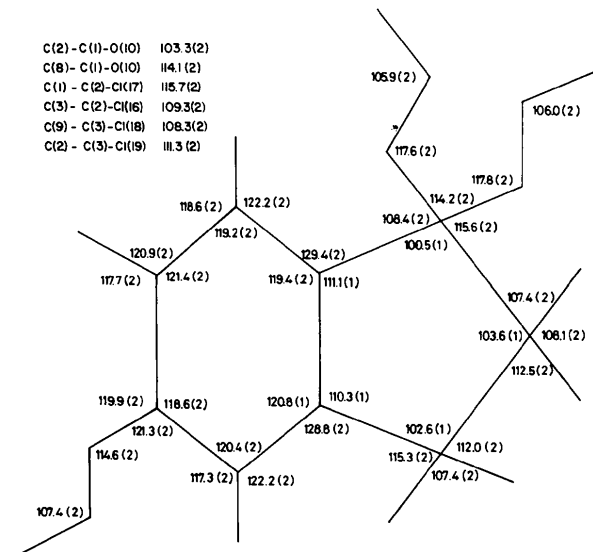


Fig. 3. Bond angles ($^\circ$).

Table 2. Selected intramolecular distances (Å)

O(10)—Cl(17)	2.845 (3)	Cl(17)—Cl(18)	3.129 (2)
O(10)—Cl(18)	3.098 (3)	Cl(18)—Cl(19)	2.861 (2)
O(13)—Cl(16)	2.883 (3)	Cl(19)—Cl(20)	3.221 (2)
O(13)—Cl(25)	2.951 (3)	Cl(20)—O(21)	2.914 (4)
Cl(16)—Cl(17)	2.859 (2)	O(21)—Cl(24)	2.899 (4)
Cl(16)—Cl(19)	3.036 (2)	Cl(24)—Cl(25)	3.097 (2)

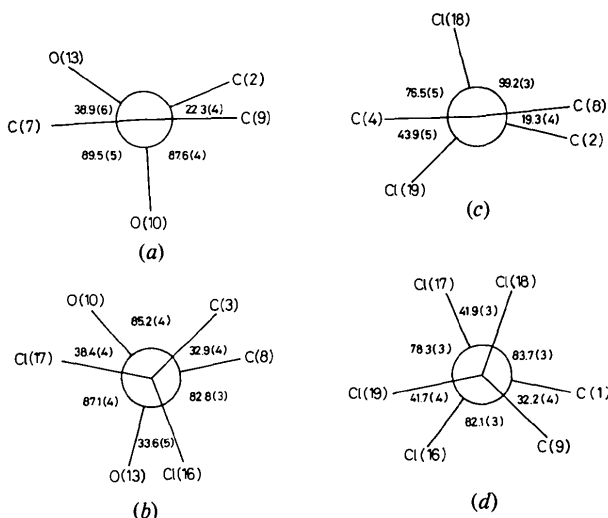


Fig. 4. Newman projection of four of the bonds of the five-membered ring, showing the torsion angles ($^{\circ}$) less than 90° . (a) Down the C(8)—C(1) bond, (b) C(2)—C(1), (c) C(9)—C(3), and (d) C(3)—C(2).

The Cl(16)—Cl(19), and Cl(17)—Cl(18) interatomic distances (3.08 Å) (Table 2) are similar to those found in other overcrowded perchlorinated compounds such as perchlorobenzylidenecyclohexa-2,5-diene (Galí, Solans, Miravittles & Plana, 1978), and hexachlorocyclohexa-2,5-dienone (Galí, Miravittles & Font-Altava, 1975), while the O(10)—Cl(17) and O(13)—Cl(16) distances (2.86 Å) are shorter than analogous distances in non-overcrowded systems (phenanthrene-2,3-dichloro-5,6-dicyano-*p*-benzoquinone, Herbstein, Kapon, Rzonzew & Rabinovich, 1978). As a consequence of this the dihedral angle between the C(1), C(8), C(9), C(3) and the C(1), C(2), C(3) least-squares mean planes (145.7°) is smaller than 165.3° , calculated by

Kitaigorodsky (1973) in cyclopentene, and the C(1)—C(2) and C(2)—C(3) bond distances (1.575 Å) are longer than the theoretical 1.54 Å of the C(sp^3)—C(sp^3) bond.

The five-membered ring presents a typical 'envelope form' of the substituted cyclopentenes. Because of overcrowding of the five-membered-ring substituents, the C(2)—C(1) and C(3)—C(2) bond torsion angles show a general trend to the skew conformation, instead of the usual tendency to present an eclipsed conformation (Eliel, 1962) (Fig. 4).

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