Insecticides. Part I. Crystal Structures of 1,1-Bis-(*p*-chlorophenyl)-2,2-dichlorocyclopropane and 1,1-Bis-(*p*-ethoxyphenyl)-2,2-dimethylpropane

By **T. P. DeLacy** and **C. H. L. Kennard,*** Department of Chemistry, University of Queensland, Brisbane, Queensland 4067, Australia

The crystal structure of 1,1-bis-(*p*-chlorophenyl)-2,2-dichlorocyclopropane, (I), was determined from three-dimensional diffractometer data with 2019 observed reflections by direct methods. $Z = 8$ in the monoclinic unit cell, space group $P2_1/a$, with dimensions, $a = 1735{\cdot}1(3)$, $b = 1570{\cdot}1(3)$, $c = 1111{\cdot}1(2)$ pm, and $\beta = 97{\cdot}09(1)°$. The refinement was halted at R 0·061. The x and z projections of the cyclopropane moiety in the molecule are 648 ± 5 pm long, with the projected area at 0·282 ± 5 nm^2. The height of the y projection is 880 pm. The crystal structure of 1,1-bis-(*p*-ethoxyphenyl)-2,2-dimethylpropane, (II), was determined from three-dimensional diffractometer data with 947 observed reflections by application of the tangent formula. $Z = 4$ in the orthorhombic unit cell, space group $Pca2_1$, with dimensions, $a = 2291{\cdot}2(2)$, $b = 1042{\cdot}4(1)$, and $c = 786{\cdot}9(1)$ pm. The refinement was halted at R 0·035. The main difference with the previous compound is that the ethoxy-groups take up a non-symmetrical *trans*-configuration. The x and y projection diameters of the neopentane group are 633 pm, projected area 0·288 nm^2, and height of y, 890 pm.

BY use of a stereochemical theory to fit organic molecules into interspaces of nerve membrane, Holan[1] has developed some highly active cyclopropane analogues of DDT. These insecticides are not readily attacked by the enzyme DDT-dehydrochlorinase. This enzyme, which occurs in resistent strains of insects, converts DDT into an active analogue DDE, dichlorodiphenyldichloroethylene. As a consequence, the cyclopropane insecticides are at present more active against DDT-resistant strains of insects than the parent DDT.

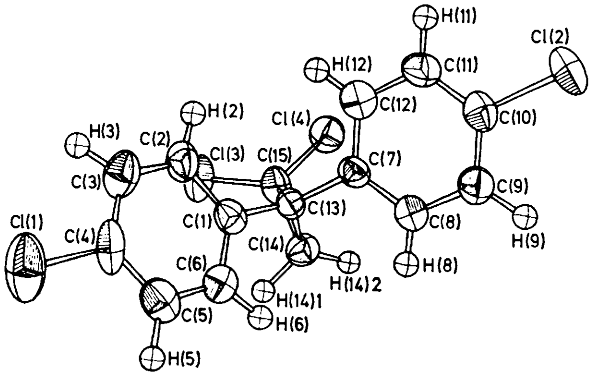

FIGURE 1 The structure of (I) viewed parallel to the bisector of C(15)–C(13)–C(14)

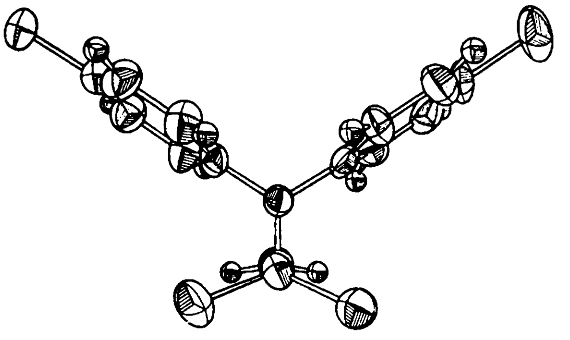

FIGURE 2 The structure of (I) viewed perpendicular to C(13)–C(14)

Holan [2] has also synthesised some non-halogenated insecticides of high activity and having a similar stereochemistry to DDT. These non-halogen containing molecules are more susceptible to biodegradation by the microbial population of the soil than DDT. We now report the crystal structure of 1,1-bis-(*p*-chlorophenyl)-2,2-dichlorocyclopropane (I) and 1,1-bis-(*p*-ethoxyphenyl)-2,2-dimethylpropane (II). A short communication on the second compound has already appeared.[3]

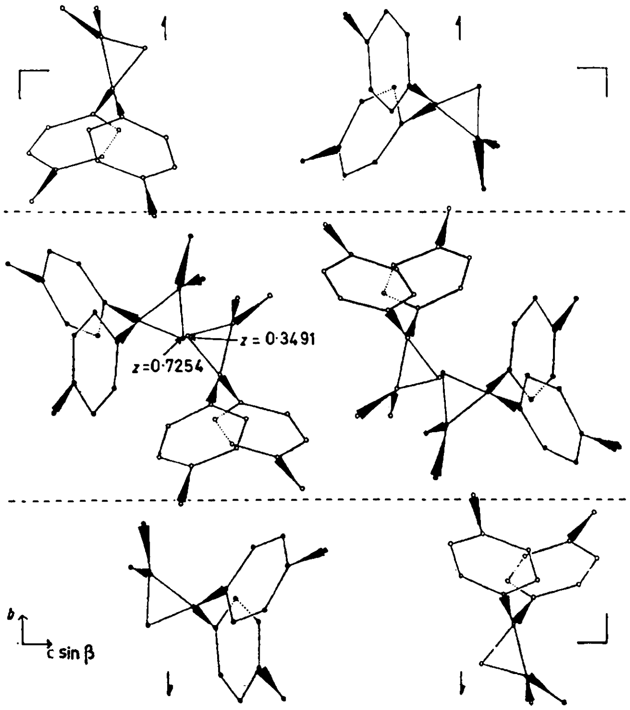

FIGURE 3 Packing of (I) in a unit cell viewed perpendicular to *c*

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[1] G. Holan, *Nature*, 1969, **221**, 1025.

[2] G. Holan, *Bull. World Health Organisation*, 1971, **44**, 355.

[3] T. P. DeLacy, C. H. L. Kennard, and G. Holan, *Chem. Comm.*, 1971, 930.

DISCUSSION

1,1-*Bis*-(p-*chlorophenyl*)-2,2-*dichlorocyclopropane* (I).—In the asymmetric unit, there are two crystallographically independent molecules. Two different views of one of these molecules are shown in Figures 1 and 2. The other molecule has essentially the same configuration.

The important atom, C(13), links the cyclopropane ring with the *p*-chlorophenyl groups. With no freedom of rotation on the vertical axis, the molecule is fixed in a symmetrical position about C(13); *i.e.* the C(14)–C(15) direction is perpendicular to a line from C(1) to C(7). The *p*-chlorophenyl groups take up a ' butterfly ' configuration with the plane of the ' flattened butterfly ' forming an angle of 10° with the C(14)–C(15) bond. The two phenyl groups are related by an approximate two-fold axis running through C(13), and a pesudo-mirror plane about the cyclopropane ring. The Cl–C(Ph) distances (175·3 and 174·2 pm) are larger than the reported value (170 pm). However, the Cl–C(cyclopropane) bond (175·2 pm) is similar to a Cl–C single bond. The C–C distances in and about the cyclopropane ring (150—151 pm) are all shorter than the normal C–C single bond, but in the expected range for ' bent ' bonds.

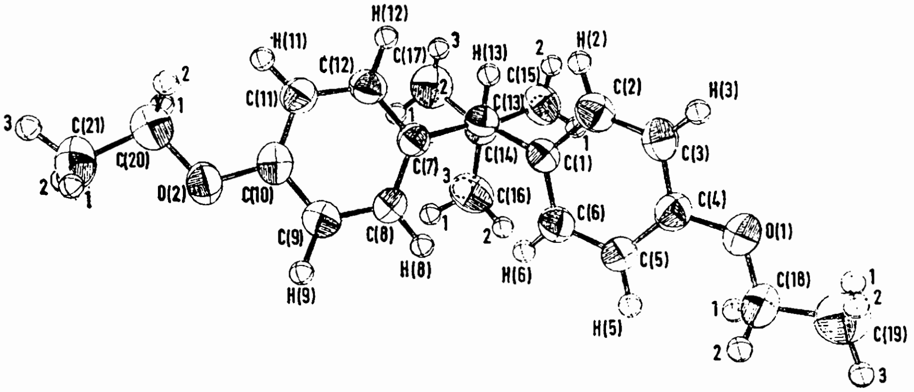

FIGURE 4 The structure of (II) viewed parallel to C(13)–C(14)

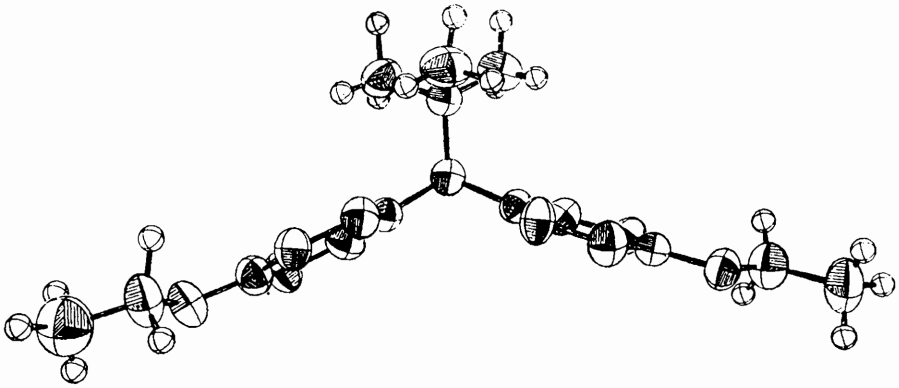

FIGURE 5 The structure of (II) viewed perpendicular to C(13)–C(14)

The cyclopropane ring imposes severe strain on the tetrahedral symmetry of its three near-neighbour carbon atoms. There is also considerable interaction between the cyclopropane moiety and the benzene ring systems. The molecules in the crystals (Figure 3) are held together only by intermolecular van der Waals interactions.

1,1-*Bis*-(p-*ethoxyphenyl*)-2,2-*dimethylpropane* (II).—The structure is illustrated in Figures 4 and 5. The central C(13) atom joins the two *p*-ethoxyphenyls to the neopentane with the two phenyl planes taking up a ' butterfly ' configuration. Excluding the ethoxy-groups, the molecule contains an approximate mirror plane, through H(13), C(13), C(14), C(16), and H(163). The ethoxy-groups take up a non-symmetrical ' *trans* '-configuration and lie almost in the plane of the attached benzene rings.

The C–C bond distances in the benzene rings, (137·0—139·9 ppm) are in the range expected. The C(13)–C(14) bond length (157·3 pm) is longer than the other C–C bonds in the neopentane grouping (152·4—153·4 pm). The C–O bonds adjacent to the benzene rings, O(1)–C(4) and O(2)–C(10) are considerably shorter (136·1 and 137·5 pm) than the O(1)–C(18) and O(2)–O(20) bonds (142·4 and 144·1 pm). This is consistent with there being some degree of electron delocalisation extending from the benzene system to the adjacent C–O bonds. Values (149·7 and 151·1 pm) for the C–C bond distances in the ethoxy-groups are significantly less than the normal C–C single bond.

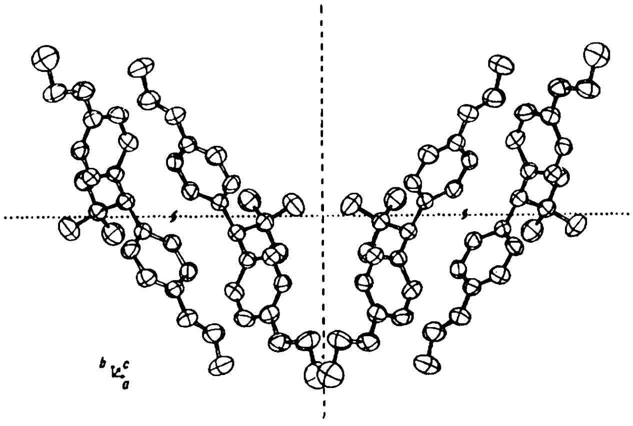

FIGURE 6 Packing of (II) in a unit-cell viewed perpendicular to *c*

The packing of the molecule in the crystal is governed only by intermolecular van der Waals forces (Figure 6).

Comparison of the Two Structures.—Figures 7 and 8 show the plane projections of both compounds viewed from infinity. The van der Waals circumferences for the periphery atoms in each projection were drawn by hand using the following radii: H 120, O 140, Cl 180, and benzene half-thickness 170 pm.

In discussing the general diaryl insecticide model, Holan states[1] that 'free rotation in the DDT series is only essential to place symmetrical dipoles at a certain position relative to the trihalogenomethyl group. This rigid symmetry requirement also exists for the halocyclopropanes.' In order to get model projections, Holan assumed that the phenyl rings take up a 'butterfly' configuration and that the plane of the 'flattened butterfly' is coplanar with the basal plane of the alkyl moiety. This was basically the configuration found in both compounds. He has also assumed, where possible, a staggered symmetrical configuration about the vertical axis C(13)–C(14) bond and the *p*-ethoxy-groups take up their most restricted rotation. However, in (II) the *p*-ethoxy-groups lie coplanar to the attached benzene rings and in a non-symmetrical *trans*-configuration.

Although the overall shape of the three projections of (I) are similar to those postulated by Holan,[1] the *y* projection is indented owing to the two dihydrogens in the cyclopropane ring. This minor change in shape is also reflected in the *z* projection. In making comparisons with Holan's model, it is assumed that the same van der Waals radii were used. The new results (648 ± 5 pm) for the diameter of the *x* and *z* projections of the cyclopropane moiety is larger than before (610 pm), and is larger than the upper limit (630 pm) set for highly active insecticides. The projected area of the cyclopropane (*z* projection) is 0·282 ± 5 nm^2 as against 0·54 nm^2 obtained by Holan. The distance (1005 pm) between the negative dipoles is well within the maximum limit (1100 pm) set by Holan. However, the distance (1370 pm) between the van der Waals outline of the negative dipoles is much greater than the model value of (1150 pm). The heights of the *y* projection are similar (880 and 900 pm).

The main differences in shape of projections of (II) are caused by ethoxy-groups taking up the non-symmetrical *trans*-configuration in the plane of the attached phenyl rings. The overall dimensions of the projections show some interesting results: the diameter of the *x* and *z* projections (633 pm) of the neopentane group is within the range (610—630 pm) for active insecticides. The projected area of this moiety is 0·288 nm^2. The height of the *y* projection is 890 pm.

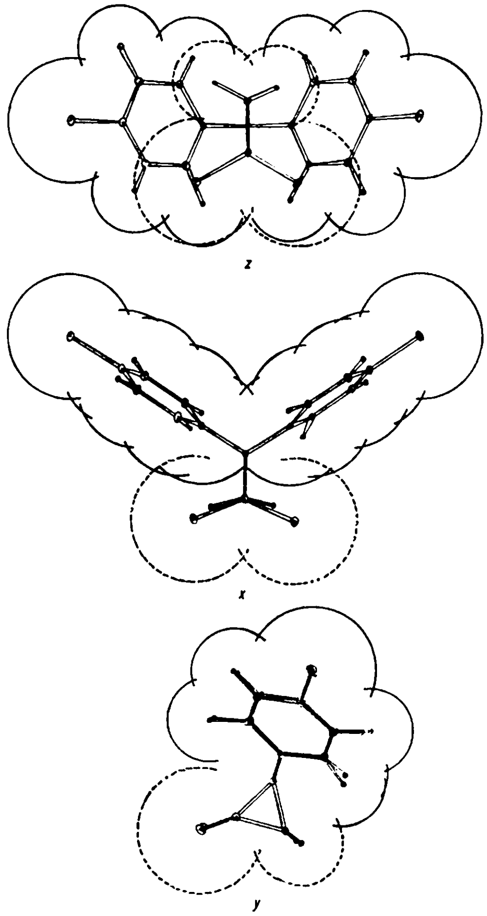

FIGURE 7 Three plane projections of (I) with the van der Waals circumferences drawn in

EXPERIMENTAL

1,1-*Bis*-(p-*chlorophenyl*)-2,2-*dichlorocyclopropane* (I)

Crystals were obtained by recrystallization from n-hexane. *Crystal Data.*—$C_{15}H_{10}Cl_4$, $M = 322$, Monoclinic, $a =$

1735·1(3), b = 1570·1(3), c = 1111·1(2) pm, β = 97·09(1)°, U = 3·004 nm^3, D_m = 1·47 (flotation), Z = 8, D_c = 1·466, $F(000)$ = 1344. Space group $P2_1/a$, (C^5_{2h}, No. 14). Cu-K_α radiation, λ = 154·18 pm; μ(Cu-K_α) = 70·09 cm^{-1}.

Intensity measurements were carried out on two box-shaped crystals, 0·25 × 0·15 × 0·02 and 0·15 × 0·1 × 0·1 mm, about the b axis. Cell dimensions were derived from diffractometer measurements of 0,2,0, 0,4,0, 0,6,0, 0,8,0, 2,0,0, 4,0,0, 6,0,0, 0,0,3, 0,0,4, and 0,0,7. Intensity data were collected on a Picker four-circle diffractometer by use of a 2θ—ω scanning mode. When the standard reflection, collected after every 50 reflections, had decreased to 80% of its initial value, the first crystal was replaced by a second which was used for further data collection.

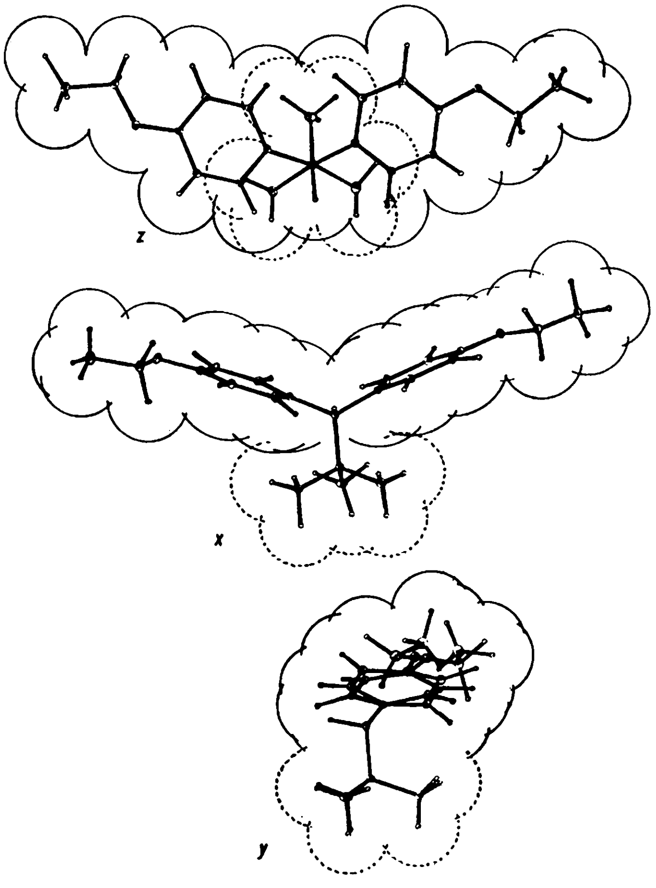

FIGURE 8 Three plane projections of (II) with the van der Waals circumferences drawn in

Intensities were corrected for Lorentz and polarization factors, and for crystal decomposition. Correction was made for X-ray absorption [gaussian integration, grid size (8 × 8 × 8)]. After application of an intercrystal scale-factor, the data sets from each crystal were combined.

Structure Determination.—A trial structure was obtained by use of the symbolic addition phase-determining procedure with 300 largest E values.

Eight chlorine atoms were located from the subsequent E map. Successive electron-density syntheses enabled location of all carbon atoms.

Full-matrix least-squares refinement with isotropic temperature factors for each of the 38 atoms, and unit weights for all observed reflections, reduced R to 0·069.

A difference-Fourier map indicated the positions of the hydrogen atoms, which were assigned equivalent isotropic temperature factors equal to those of neighbouring bonded atoms. Further refinement with unit weights reduced R and R' to 0·61 and 0·65 respectively [$R' = (\Sigma w|F_o - F_c|^2/\Sigma w|F_o|^2)^{\frac{1}{2}}$].

Because of limited computer core storage, each cycle of refinement was carried out in three steps. Although the refinement had not converged, it was halted as the essential structure features had been determined.

A final difference-Fourier failed to reveal unexplained electron density. A plot of I_c/I_o *vs.* I_c gave no indication of extinction effects.

The atomic co-ordinates and temperature factors are listed in Table 1. The observed and calculated structure amplitudes for both compounds are listed in Supplementary Publication No. SUP 20477 (13 pp., 1 microfiche).* The scattering factors used were those for chlorine, carbon,[4] and hydrogen.[5] No corrections were made for anomalous dispersion.

1,1-*Bis*-(p-*ethoxyphenyl*)-2,2-*dimethylpropane* (II)

Crystals were obtained by slow recrystallization from aqueous alcohol.

Crystal Data.—$C_{21}H_{28}O_2$, M = 312. Orthorhombic, a = 2291·2(2), b = 1042·4(1), c = 786·9(1) pm, U = 1·881

TABLE 1

(*a*) Atomic parameters for (I) with estimated standard deviations in parentheses

Atom	x/a	y/b	z/c	B/Å^2
(i) Molecule (1)				
Cl(1)	0·2711(2)	0·2441(2)	0·7440(3)	*
Cl(2)	0·4791(2)	0·2710(2)	−0·0576(2)	
Cl(3)	0·3691(2)	0·6055(2)	0·4936(2)	
Cl(4)	0·4260(2)	0·6077(2)	0·2597(2)	
C(1)	0·3234(6)	0·4152(6)	0·4356(8)	
C(2)	0·3831(6)	0·3917(7)	0·5292(8)	
C(3)	0·3670(7)	0·3408(7)	0·6238(9)	
C(4)	0·2910(8)	0·3090(6)	0·6239(9)	
C(5)	0·2351(7)	0·3275(7)	0·5335(10)	
C(6)	0·2504(6)	0·3796(6)	0·4385(8)	
C(7)	0·3757(5)	0·4204(6)	0·2332(8)	
C(8)	0·3298(6)	0·3900(8)	0·1310(9)	
C(9)	0·3609(6)	0·3441(7)	0·0407(9)	
C(10)	0·4386(6)	0·3250(6)	0·0566(9)	
C(11)	0·4859(6)	0·3532(7)	0·1571(10)	
C(12)	0·4548(6)	0·3988(8)	0·2465(9)	
C(13)	0·3394(5)	0·4677(6)	0·3289(8)	
C(14)	0·2834(6)	0·5360(6)	0·2865(8)	
C(15)	0·3607(6)	0·5578(6)	0·3491(8)	
H(2)	0·443(4)	0·418(8)	0·524(9)	4·0
H(3)	0·410(4)	0·321(8)	0·698(9)	4·5
H(5)	0·187(4)	0·303(8)	0·534(9)	4·8
H(6)	0·208(4)	0·405(8)	0·384(9)	3·9
H(8)	0·279(4)	0·399(8)	0·127(9)	3·9
H(9)	0·322(4)	0·313(8)	−0·042(9)	4·3
H(11)	0·539(4)	0·339(8)	0·167(9)	4·6
H(12)	0·499(4)	0·431(8)	0·327(9)	4·3
H(141)	0·243(4)	0·539(8)	0·337(9)	3·7
H(142)	0·271(4)	0·543(8)	0·187(9)	3·7

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* For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20.

[4] 'International Tables for X-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962, p. 201.

[5] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

TABLE 1 (*Continued*)

(ii) Molecule (2)

Atom	x/a	y/b	z/c	$B/\text{Å}_2$
Cl(1)	0·4450(2)	−0·0975(2)	−0·1987(3)	*
Cl(2)	0·5187(2)	0·1610(2)	0·6419(2)	
Cl(3)	0·1867(2)	0·1356(2)	0·0183(2)	
Cl(4)	0·2065(2)	0·2101(2)	0·2578(2)	
C(1)	0·3338(6)	0·0275(6)	0·1027(8)	
C(2)	0·3391(6)	−0·0589(7)	0·0969(8)	
C(3)	0·3744(7)	−0·0999(7)	0·0026(9)	
C(4)	0·4028(8)	−0·0483(6)	−0·0808(9)	
C(5)	0·4016(7)	0·0379(7)	−0·0754(10)	
C(6)	0·3666(6)	0·0755(6)	0·0181(8)	
C(7)	0·3509(5)	0·0938(6)	0·3140(8)	
C(8)	0·3615(6)	0·0388(8)	0·4112(9)	
C(9)	0·4154(6)	0·0592(7)	0·5143(9)	
C(10)	0·4534(6)	0·1356(6)	0·5146(9)	
C(11)	0·4460(6)	0·1891(7)	0·4158(10)	
C(12)	0·3947(6)	0·1686(8)	0·3162(9)	
C(13)	0·2950(5)	0·0656(6)	0·2043(8)	
C(14)	0·2155(6)	0·0324(6)	0·2245(8)	
C(15)	0·2228(6)	0·1217(6)	0·1683(8)	
H(2)	0·325(4)	−0·095(8)	0·162(9)	4·2
H(3)	0·377(4)	−0·160(8)	0·004(9)	4·4
H(5)	0·421(4)	0·071(8)	−0·144(9)	4·3
H(6)	0·357(4)	0·121(8)	0·015(9)	3·8
H(8)	0·326(4)	−0·000(8)	0·410(9)	5·3
H(9)	0·431(4)	0·019(8)	0·598(9)	5·5
H(11)	0·486(4)	0·241(8)	0·415(9)	4·7
H(12)	0·380(4)	0·209(8)	0·241(9)	3·9
H(141)	0·200(4)	0·023(8)	0·324(9)	5·9
H(142)	0·180(4)	0·003(8)	0·141(9)	5·9

(*b*) Anisotropic thermal parameters ($\times 10^4$) *

(i) Molecule (1)

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cl(1)	98(2)	50(1)	81(2)	−35(1)	20(2)	7(2)
Cl(2)	49(1)	53(1)	89(2)	6(1)	31(1)	−2(2)
Cl(3)	67(1)	45(1)	72(2)	−5(1)	7(1)	−4(1)
Cl(4)	45(1)	54(1)	85(2)	−16(1)	10(1)	8(1)
C(1)	31(4)	33(5)	63(9)	1(4)	13(5)	−11(5)
C(2)	41(5)	45(6)	61(9)	6(4)	9(6)	12(6)
C(3)	46(6)	42(6)	74(11)	−4(5)	−1(6)	8(7)
C(4)	67(7)	34(5)	39(9)	−16(5)	12(6)	−2(6)
C(5)	50(6)	38(6)	92(12)	−18(4)	25(7)	−23(7)
C(6)	36(5)	29(5)	78(10)	4(4)	3(6)	−7(6)
C(7)	25(4)	34(5)	67(9)	−1(4)	13(5)	4(5)
C(8)	33(4)	69(7)	73(10)	3(5)	9(6)	−10(7)
C(9)	35(5)	57(6)	71(10)	7(4)	2(6)	−3(7)
C(10)	41(5)	39(5)	63(10)	−1(4)	13(6)	1(6)
C(11)	34(4)	59(7)	104(11)	15(5)	18(7)	6(7)
C(12)	35(5)	57(6)	73(10)	3(5)	9(6)	−10(7)
C(13)	26(4)	32(5)	63(9)	2(4)	8(5)	1(6)
C(14)	34(5)	41(5)	78(10)	−6(4)	4(6)	−7(6)
C(15)	43(5)	36(5)	68(9)	−3(4)	15(6)	6(6)

* Defined as $T = \exp -(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{23}kl + 2\beta_{13}hl)$

(ii) Molecule (2)

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cl(1)	42(2)	80(1)	89(2)	17(1)	13(2)	−25(2)
Cl(2)	60(1)	85(1)	97(2)	6(1)	−18(1)	−27(2)
Cl(3)	43(1)	93(1)	86(2)	21(1)	−2(1)	−18(1)
Cl(4)	47(1)	52(1)	108(2)	9(1)	19(1)	−20(1)
C(1)	27(4)	42(5)	76(9)	−4(4)	7(5)	−5(5)
C(2)	34(5)	48(6)	74(9)	9(4)	8(6)	−9(6)
C(3)	42(6)	47(6)	83(11)	10(5)	2(6)	−9(7)
C(4)	37(7)	54(5)	67(9)	11(5)	8(6)	−15(6)
C(5)	37(6)	55(6)	80(12)	−5(4)	20(7)	−6(7)
C(6)	43(5)	39(5)	104(10)	0(4)	18(6)	−13(6)
C(7)	26(4)	55(5)	56(9)	7(4)	5(5)	−18(5)
C(8)	59(4)	42(7)	89(10)	−2(5)	−5(6)	5(7)
C(9)	59(5)	57(6)	90(10)	84(4)	16(6)	16(7)
C(10)	44(5)	52(5)	73(10)	4(4)	−3(6)	−18(6)
C(11)	48(4)	39(7)	82(11)	−16(5)	5(7)	−13(7)
C(12)	39(5)	36(6)	78(10)	−6(4)	11(6)	−9(7)
C(13)	42(4)	36(5)	79(9)	−7(4)	21(5)	−11(6)
C(14)	35(5)	63(5)	82(10)	11(4)	13(6)	−26(6)
C(15)	46(5)	59(5)	109(9)	−8(4)	26(6)	−17(6)

TABLE 1 (*Continued*)

(*c*) Interatomic distances (pm); estimated standard deviations: C–Cl 0·5, C–C 1, C–H 10 pm

	Molecule (1)	Molecule (2)
Cl(1)–C(4)	175·0	175·7
Cl(2)–C(10)	174·0	174·4
Cl(3)–C(15)	175·8	172·1
Cl(4)–C(15)	177·6	175·1
C(1)–C(2)	142	136
C(2)–C(3)	137	143
C(3)–C(4)	141	137
C(4)–C(5)	134	136
C(5)–C(6)	139	140
C(6)–C(1)	139	138
C(7)–C(8)	139	138
C(8)–C(9)	140	142
C(9)–C(10)	138	137
C(10)–C(11)	137	138
C(11)–C(12)	138	137
C(12)–C(7)	141	140
C(1)–C(13)	150	151
C(7)–C(13)	149	153
C(13)–C(14)	149	152
C(13)–C(15)	148	154
C(14)–C(15)	148	154
H(2)–C(2)	110	100
H(3)–C(3)	110	90
H(5)–C(5)	90	100
H(6)–C(6)	100	80
H(8)–C(8)	90	90
H(9)–C(9)	120	110
H(11)–C(11)	90	110
H(12)–C(12)	120	110
H(141)–C(14)	90	120
H(142)–C(14)	110	110

(*d*) Interatomic angles (deg.); estimated standard deviation: 0·5°, those involving H atoms, 10°

	Molecule (1)	Molecule (2)
C(13)–C(1)–C(2)	122·7	118·0
C(13)–C(1)–C(6)	119·8	123·5
C(2)–C(1)–C(6)	117·1	118·4
C(1)–C(2)–C(3)	121·1	121·5
H(2)–C(2)–C(1)	117	121
H(2)–C(2)–C(3)	122	117
C(2)–C(3)–C(4)	119·1	116·8
H(3)–C(3)–C(2)	124	118
H(3)–C(3)–C(4)	117	126
C(3)–C(4)–C(5)	120·8	123·6
Cl(1)–C(4)–C(3)	119·0	117·6
Cl(1)–C(4)–C(5)	120·2	118·9
C(4)–C(5)–C(6)	120·6	117·7
H(5)–C(5)–C(4)	119	118
H(5)–C(5)–C(6)	121	124
C(5)–C(6)–C(1)	121·1	121·9
H(6)–C(6)–C(5)	121	119
H(6)–C(6)–C(1)	116	118
C(13)–C(7)–C(12)	122·8	123·2
C(13)–C(7)–C(8)	119·9	117·0
C(8)–C(7)–C(12)	117·0	119·6
C(7)–C(12)–C(11)	121·8	120·0
H(12)–C(12)–C(7)	119	114
H(12)–C(12)–C(11)	119	125
C(12)–C(11)–C(10)	120·2	119·2
H(11)–C(11)–C(12)	120	122
H(11)–C(11)–C(10)	120	188

TABLE 1 (*Continued*)

	Molecule (1)	Molecule (2)
C(9)–C(10)–C(11)	120·3	122·4
Cl(2)–C(10)–C(11)	119·8	119·6
Cl(2)–C(10)–C(9)	119·8	117·8
C(10)–C(9)–C(8)	119·3	117·9
H(9)–C(9)–C(10)	117	115
H(9)–C(9)–C(8)	123	127
C(9)–C(8)–C(7)	121·8	120·0
H(8)–C(8)–C(9)	120	125
H(8)–C(8)–C(7)	118	114
C(1)–C(13)–C(7)	115·3	114·4
C(1)–C(13)–C(14)	117·8	118·2
C(1)–C(13)–C(15)	118·1	117·1
C(7)–C(13)–C(14)	116·9	118·8
C(7)–C(13)–C(15)	117·6	117·2
C(14)–C(13)–C(15)	60·2	60·6
Cl(3)–C(15)–C(13)	122·6	120·7
Cl(3)–C(15)–C(14)	119·4	117·9
Cl(3)–C(15)–Cl(4)	109·9	112·6
Cl(4)–C(15)–C(13)	120·1	119·2
Cl(4)–C(15)–C(14)	116·6	117·6
C(13)–C(15)–C(14)	60·1	58·9
H(141)–C(14)–C(13)	111	120
H(141)–C(14)–C(15)	114	122
H(141)–C(14)–H(142)	122	123
H(142)–C(14)–C(13)	115	116
H(142)–C(14)–C(15)	119	96
C(13)–C(14)–C(15)	59·7	60·5

TABLE 2

(*a*) Atomic parameters for (II) with standard deviations in parentheses

Atom	*x*/*a*	*y*/*b*	*z*/*c*	*B*/Å^2
C(1)	−0·1166(1)	−0·1677(3)	0·9235(5)	*
C(2)	−0·0923(1)	−0·2899(4)	0·9030(5)	
C(3)	−0·1107(1)	−0·3940(3)	0·9982(6)	
C(4)	−0·1151(1)	−0·3804(3)	1·1174(5)	
C(5)	−0·1785(1)	−0·2593(3)	1·1448(5)	
C(6)	−0·1589(1)	−0·1564(3)	1·0477(6)	
C(7)	−0·0681(1)	0·0549(3)	0·8800(5)	
C(8)	−0·0892(1)	0·1242(3)	1·0174(6)	
C(9)	−0·0625(1)	0·2347(4)	1·0736(5)	
C(10)	−0·0123(1)	0·2794(3)	0·9931(5)	
C(11)	0·0107(1)	0·2114(4)	0·8601(5)	
C(12)	−0·0167(1)	0·0998(3)	0·8069(5)	
C(13)	−0·0981(1)	−0·0625(3)	0·7997(5)	
C(14)	−0·1473(1)	−0·0231(3)	0·6707(5)	
C(15)	−0·1690(1)	−0·1443(3)	0·5827(6)	
C(16)	−0·1993(1)	0·0449(4)	0·7519(7)	
C(17)	−0·1210(2)	0·0635(4)	0·5335(7)	
C(18)	−0·2221(1)	−0·4825(4)	1·3051(6)	
C(19)	−0·2382(2)	−0·6163(5)	1·3620(8)	
C(20)	0·0560(1)	0·4529(4)	0·9631(6)	
C(21)	0·0691(2)	0·5777(4)	1·0490(8)	
O(1)	−0·1715(1)	−0·4892(2)	1·2006(4)	
O(2)	0·0092(1)	0·3930(2)	1·0562(4)	
H(2)	−0·061(1)	−0·295(3)	0·823(5)	2·9(1·1)
H(3)	−0·092(1)	−0·476(3)	0·997(5)	4·0(1·1)
H(5)	−0·211(1)	−0·252(3)	1·234(5)	3·0(1·1)
H(6)	−0·182(1)	−0·073(3)	1·048(5)	2·5(1·2)
H(8)	−0·127(1)	0·092(3)	1·080(5)	2·4(1·2)
H(9)	−0·076(1)	0·284(3)	1·192(5)	3·6(1·3)
H(11)	0·043(1)	0·254(3)	0·794(5)	5·4(1·1)
H(12)	−0·003(1)	0·050(3)	0·694(5)	2·9(1·1)
H(13)	−0·065(1)	−0·106(3)	0·724(5)	0·8(1·0)
H(181)	−0·257(2)	−0·437(4)	1·232(6)	3·9(1·3)
H(182)	−0·215(2)	−0·432(4)	1·424(6)	4·5(1·4)
H(201)	0·043(1)	0·460(4)	0·847(6)	2·5(1·2)
H(202)	0·090(2)	0·379(4)	0·945(6)	6·4(1·3)
H(191)	−0·245(2)	−0·653(5)	1·268(7)	6·4(1·6)
H(192)	−0·204(2)	−0·644(5)	1·406(7)	8·0(1·7)
H(193)	−0·278(2)	−0·620(5)	1·450(8)	7·0(1·7)
H(211)	0·041(2)	0·632(5)	1·029(7)	4·9(1·4)
H(212)	0·088(2)	0·555(5)	1·158(7)	3·6(1·6)
H(213)	0·103(2)	0·635(5)	0·994(8)	6·0(1·5)
H(151)	−0·186(2)	−0·201(4)	0·653(6)	3·9(1·4)
H(152)	−0·138(2)	−0·192(4)	0·540(7)	3·0(1·3)
H(153)	−0·202(2)	−0·121(4)	0·484(6)	3·6(1·4)
H(161)	−0·184(2)	0·120(4)	0·830(6)	6·4(1·5)
H(162)	−0·220(2)	−0·006(4)	0·853(6)	4·8(1·5)
H(163)	−0·222(2)	0·082(4)	0·661(7)	6·9(1·6)
H(171)	−0·119(2)	0·141(4)	0·605(7)	4·1(1·5)
H(172)	−0·153(2)	0·096(4)	0·458(6)	4·3(1·4)
H(173)	−0·092(2)	0·021(4)	0·487(6)	2·9(1·3)

(*b*) Anisotropic thermal parameters (× 10^4) *

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	18(1)	85(4)	150(7)	1(1)	−2(2)	−9(5)
C(2)	22(1)	105(5)	180(9)	8(2)	9(3)	12(6)
C(3)	28(1)	89(4)	223(10)	10(2)	6(3)	20(6)
C(4)	23(1)	100(5)	172(9)	−4(2)	−1(2)	8(6)
C(5)	20(1)	105(4)	182(9)	−3(2)	14(3)	−10(6)
C(6)	21(1)	91(4)	219(10)	2(2)	7(3)	−11(6)
C(7)	17(1)	84(4)	152(9)	−1(1)	−5(2)	−9(5)
C(8)	22(1)	102(4)	207(10)	−7(2)	18(3)	−10(6)
C(9)	28(1)	103(5)	176(9)	−4(2)	11(3)	−15(6)
C(10)	21(1)	81(4)	188(9)	2(1)	−8(3)	7(6)
C(11)	19(1)	92(5)	204(10)	−1(1)	−2(2)	−2(6)
C(12)	17(1)	104(4)	200(9)	3(2)	−1(3)	−21(6)
C(13)	18(1)	87(3)	173(8)	2(1)	7(2)	−9(6)
C(14)	20(1)	96(4)	173(9)	2(2)	−2(2)	4(5)
C(15)	28(1)	134(5)	231(10)	−2(2)	−18(3)	−23(7)
C(16)	27(1)	159(6)	269(12)	23(2)	−10(3)	−15(7)
C(17)	32(1)	152(6)	240(12)	3(2)	−12(3)	41(8)
C(18)	34(1)	124(4)	233(10)	−11(2)	16(3)	27(6)
C(19)	47(2)	164(7)	297(15)	−26(3)	29(5)	50(10)
C(20)	29(1)	107(5)	248(11)	−12(2)	2(3)	−1(6)
C(21)	40(1)	117(5)	325(13)	−21(2)	6(4)	−16(8)
O(1)	32(1)	103(3)	216(6)	0(1)	16(2)	32(4)
O(2)	25(1)	96(2)	190(5)	−9(1)	−2(2)	−14(4)

* Defined as before in Table 1.

(*c*) Interatomic distances (pm), estimated standard deviations C–C, C–O, 0·5; C–H, 5 pm

C(1)–C(2)	139·9	C(7)–C(12)	139·1
C(2)–C(3)	138·4	C(12)–C(11)	138·7
C(3)–C(4)	139·1	C(11)–C(10)	137·0
C(4)–C(5)	139·0	C(10)–C(9)	139·4
C(5)–C(6)	139·1	C(9)–C(8)	137·7
C(6)–C(1)	138·3	C(8)–C(7)	138·8
C(13)–C(1)	152·8	C(13)–C(7)	153·9
C(13)–C(14)	157·3	C(14)–C(16)	152·7
C(14)–C(15)	152·4	C(14)–C(17)	153·2
C(4)–O(1)	136·1	C(10)–O(2)	137·5
O(1)–C(18)	142·4	O(2)–C(20)	144·1
C(18)–C(19)	151·1	C(20)–C(21)	149·7
H(2)–C(2)	96	H(12)–C(12)	107
H(3)–C(3)	96	H(11)–C(11)	100
H(5)–C(5)	102	H(8)–C(8)	105
H(6)–C(6)	102	H(9)–H(9)	110
H(13)–C(13)	107	H(161)–C(16)	106
		H(162)–C(16)	106
		H(163)–C(16)	96
H(151)–C(15)	90	H(171)–C(17)	99
H(152)–C(15)	92	H(173)–C(17)	88
H(153)–C(15)	110	H(172)–C(17)	100
H(181)–C(18)	109	H(201)–C(20)	96
H(182)–C(18)	109	H(202)–C(20)	109
H(191)–C(19)	84	H(211)–C(21)	87
H(192)–C(19)	91	H(212)–C(21)	99
H(193)–C(19)	113	H(213)–C(21)	106

TABLE 2 (*Continued*)

(*d*) Interatomic angles (deg.); estimated standard deviation, 0·3°, those involving H atoms, 5°

C(13)–C(1)–C(2)	118·0
C(13)–C(1)–C(6)	125·8
C(2)–C(1)–C(6)	116·0
C(1)–C(2)–C(3)	122·1
H(2)–C(2)–C(1)	115
H(2)–C(2)–C(3)	122
C(2)–C(3)–C(4)	120·5
H(3)–C(3)–C(2)	124
H(3)–C(3)–C(4)	115
C(3)–C(4)–C(5)	118·7
O(1)–C(4)–C(3)	116·3
O(1)–C(4)–C(5)	125·1
C(4)–C(5)–C(6)	119·4
H(5)–C(5)–C(4)	117
H(5)–C(5)–C(6)	123
C(5)–C(6)–C(1)	123·3
H(6)–C(6)–C(5)	119
H(6)–C(6)–C(1)	116
C(7)–C(13)–C(1)	115·7
C(7)–C(13)–C(14)	112·2
C(1)–C(13)–C(14)	113·5
C(7)–C(13)–H(13)	105
C(1)–C(13)–H(13)	104
C(14)–C(13)–H(13)	105
C(13)–C(7)–C(12)	118·3
C(13)–C(7)–C(8)	125·3
C(12)–C(7)–C(8)	116·3
C(7)–C(12)–C(11)	122·7
H(12)–C(12)–C(7)	115
H(12)–C(12)–C(11)	121
C(12)–C(11)–C(10)	119·4
H(11)–C(11)–C(12)	123
H(11)–C(11)–C(10)	117
C(11)–C(10)–C(9)	119·4
O(2)–C(10)–C(11)	125·7
O(2)–C(10)–C(9)	114·8
C(10)–C(9)–C(8)	120·0
H(9)–C(9)–C(10)	117
H(9)–C(9)–C(8)	122
C(9)–C(8)–C(7)	122·1
H(8)–C(8)–C(9)	119
H(8)–C(8)–C(7)	119
C(13)–C(14)–C(15)	108·1
C(13)–C(14)–C(16)	114·3
C(13)–C(14)–C(17)	109·0
C(15)–C(14)–C(16)	108·7
C(15)–C(14)–C(17)	107·3
C(16)–C(14)–C(17)	109·1
H(161)–C(16)–C(14)	109
H(161)–C(16)–H(162)	95
H(161)–C(16)–H(163)	108
H(162)–C(16)–C(14)	115
H(162)–C(16)–H(163)	121
H(163)–C(16)–C(14)	107
H(151)–C(15)–C(14)	114
H(151)–C(15)–H(152)	102
H(151)–C(15)–H(153)	106
H(152)–C(15)–C(14)	111
H(152)–C(14)–H(153)	113
H(153)–C(15)–C(14)	111
O(1)–C(18)–C(19)	108·9
O(1)–C(18)–H(181)	108
O(1)–C(18)–H(182)	113
C(19)–C(18)–H(181)	112
C(19)–C(18)–H(182)	113
H(181)–C(18)–H(182)	111
H(191)–C(19)–C(18)	102
H(191)–C(19)–H(192)	110
H(191)–C(19)–H(193)	112
H(192)–C(19)–C(18)	101
H(192)–C(19)–H(193)	117
H(193)–C(19)–C(18)	114
C(4)–O(1)–C(18)	117·6
H(171)–C(17)–C(14)	96
H(171)–C(17)–H(172)	95
H(171)–C(17)–H(173)	128
H(172)–C(17)–C(14)	110
H(172)–C(17)–H(173)	118
H(173)–C(17)–C(14)	107
O(2)–C(20)–C(21)	107·3
O(2)–C(20)–H(201)	107
O(2)–C(20)–H(202)	107
C(21)–C(20)–H(201)	115
C(21)–C(20)–H(202)	121
H(201)–C(20)–H(202)	99
H(211)–C(21)–C(20)	109
H(211)–C(21)–H(212)	129
H(211)–C(21)–H(213)	96
H(212)–C(21)–C(20)	106
H(212)–C(21)–H(213)	100
H(213)–C(21)–C(20)	117
C(10)–O(2)–C(20)	117·1

nm^3, $D_m = 1·11$ (flotation), $Z = 4$, $D_c = 1·100$, $F(000) = 680$. Space group $Pca2_1$. μ(Cu-K_α) = 5·40 cm^{-1}.

A plate-shaped crystal, 0·4 × 0·2 × 0·05 mm, was mounted about the *c* axis. Cell dimensions were derived from diffractometer measurements. Intensity data were collected with a Picker four-circle diffractometer as before. Of the 1400 reflections collected up to $2\theta < 115°$, 947 were observed.

Intensities were corrected for Lorentz and polarization factors but not for absorption or extinction.

Structure Determination.—The structure was solved by the tangent method. The phases of 202 reflections with $|E| > 1·4$ were estimated and refined. After the final cycle of refinement, R_K was 0·246, where ($R_K = \Sigma||E_h|_{obs.} - |E_h|_{calc.}|/\Sigma|E_h|_{obs.}$). The complete molecule was recognizable from the E map.

Full-matrix least-squares refinement reduced R to 0·104 and R' to 0·115. A difference electron-density synthesis showed the positions of all the hydrogen atoms. Further refinement with unit weights reduced R to 0·035 and R' to 0·035. At this stage the refinement had not converged.

A final difference-Fourier failed to reveal any prominent features. A plot of I_c/I_o *vs.* I_o gave no indication of extinction.

Atomic co-ordinates and temperature factors are listed in Table 2.

The structures were determined, refined, and bond distances calculated with local versions of standard programmes.[6]

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[6] FAME-MAGIC (Symbolic Addition), Dewar and Stone, modified by R. C. Seccombe, 1970; PREFOUR (Structure Factor), FOURIER, J. Blount (1966); ORFLS (Full-Matrix Least-Squares), W. R. Busing, K. O. Martin, and H. A. Levy, 1964; C. J. Johnson, ORTEP, Plotting Program; DP Series, (Tangent Method), S. R. Hall and E. N. Maslen, modified by R. C. Seccombe, 1969; CRYSET (CDC parcel).