Insecticides. Part II.† Crystal Structures of 1,1-Bis-(p-chlorophenyl)-2,2,2-trichloroethane (p,p'-DDT) and 1-(o-Chlorophenyl)-1-(p-chlorophenyl)-2,2,2-trichloroethane (o,p'-DDT)

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)-2.2.2-trichloroethane. (I). was determined from three-dimensional diffractometer data with 900 reflections. $Z = 4$ in the orthorhombic unit cell, space group $Pca2_1$ with dimensions, $a = 996.3(1)$. $b = 1920.0(2)$. and $c = 788.7(1)$ pm. The structure was refined by least-squares to R 0.091. The crystal structure of 1-(o-chlorophenyl)-1-(p-chlorophenyl)trichloroethane. (II). was determined from three-dimensional diffractometer data with 2649 reflections by the symbolic addition procedure. $Z = 4$ in the triclinic unit cell, space group $P\bar{1}$, with dimensions $a = 1201.6(1)$. $b = 1248.1(1)$. $c = 1093.8(1)$ pm. $\alpha = 105.43(3)$. $\beta = 89.93(3)$. $\gamma = 109.03(3)$*. Least-squares refinement was halted at R 0.048. These two isomers resemble one another. The diameter and projected areas of the two apices and the overall heights are similar but the overall length and distance between the two electronegative centres are not.

STEREOCHEMISTRY is thought to play a role in the mode of action of DDT. In order to learn more, the crystal structure determinations of two isomers of DDT [(I) p,p'- and (II) o,p'-] have been carried out. A preliminary communication on p,p'-DDT has already been made.[1]
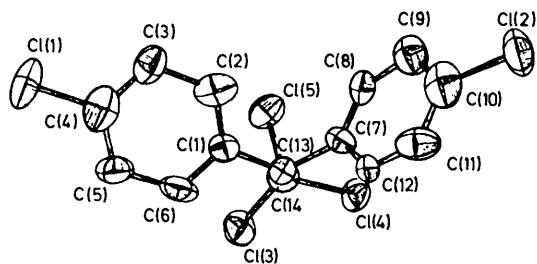
FIGURE 1 The structure of (I) viewed parallel to C(13)–C(14)

DISCUSSION

The central tetrahedral carbon atom, C(13) in (I) (Figures 1 and 2) joins the two p-chlorophenyl groups to

in the plane perpendicular to C(13)–C(14) results in the phenyl rings lying ' extended ' in the plane, then in the
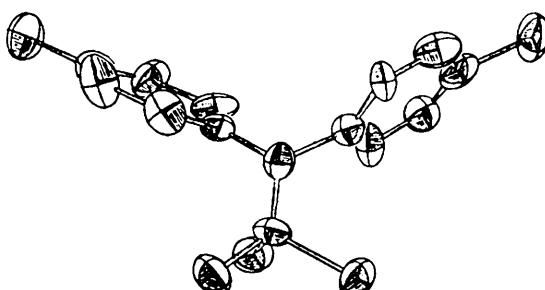

FIGURE 2 The structure of (I) viewed perpendicular to C(13)–C(14)

DDT molecule, the phenyl rings have torsion rotations of 4 [anticlockwise with respect to the C(13)–C(1) direction] and 33° [anticlockwise with respect to the C(13)–C(7)]. If viewed down C(13)–C(14), the trichloro-group is rotated slightly away from the staggered configuration
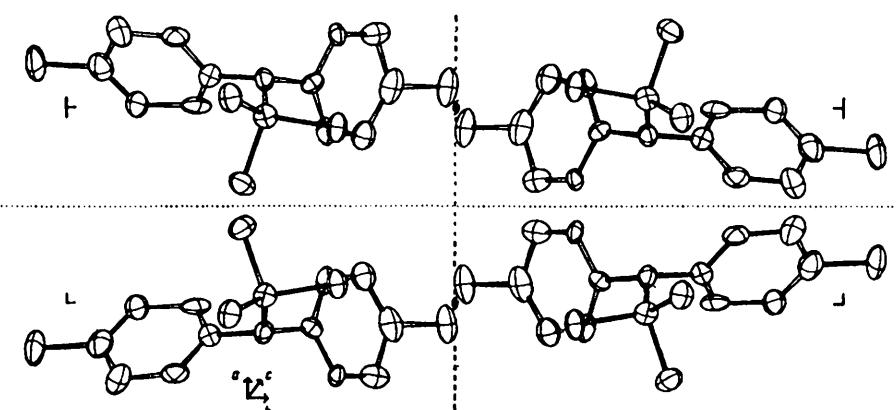

FIGURE 3 Packing of (I) in a unit cell viewed perpendicular to c

the trihalogenomethyl group. If an ideal ' butterfly ' configuration of the molecule is one where the projection

with respect to the chlorophenyl groups. Figure 3 shows the packing arrangement about c.

The o,p'-isomer of DDT (II) contains two molecules in the asymmetric unit. (Figures 4 and 5). The butterfly configuration is distorted with phenyl rings having

† Part I, T. P. DeLacy and C. H. L. Kennard, preceding paper.
[1] T. P. DeLacy and C. H. L. Kennard, Chem. Comm., 1971, 1208.
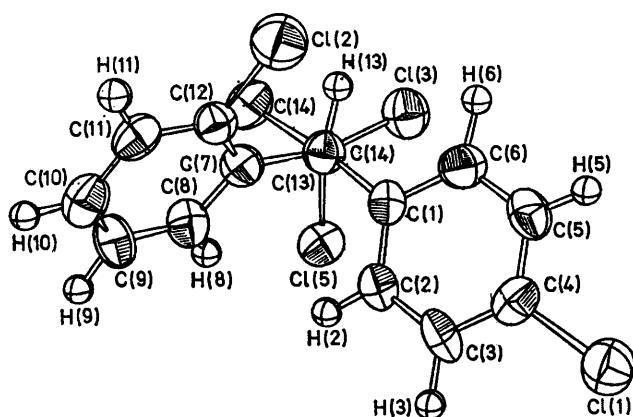
FIGURE 4 The structure of (II) viewed parallel to C(13)–C(14)
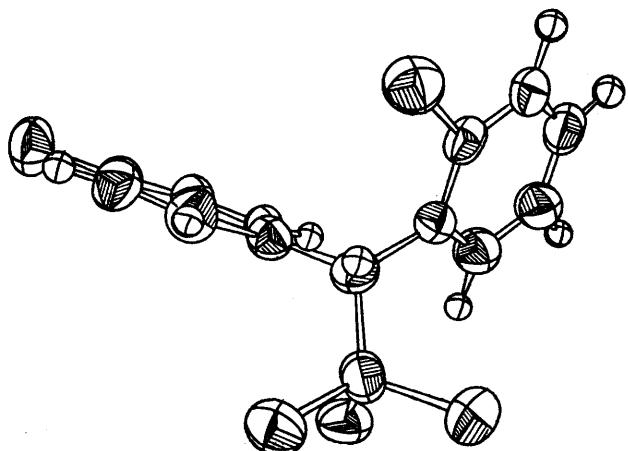
FIGURE 5 The structure of (II) viewed perpendicular to C(13)–C(14)
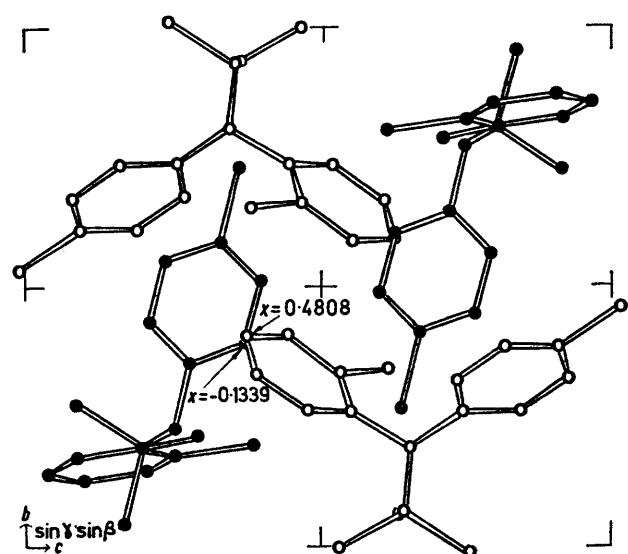
FIGURE 6 Packing of (II) in a unit cell viewed perpendicular to *a*

torsional rotations of 4 [clockwise with respect to the C(13)–C(1) direction] and 47° [anticlockwise with respect to the C(13)–C(7)]. The phenyl Cl–C bond distances (174·3 and 174·1 pm) and paraffinic Cl–C bonds are longer than usual (170 pm), with Cl(5)–C(14) (178·7
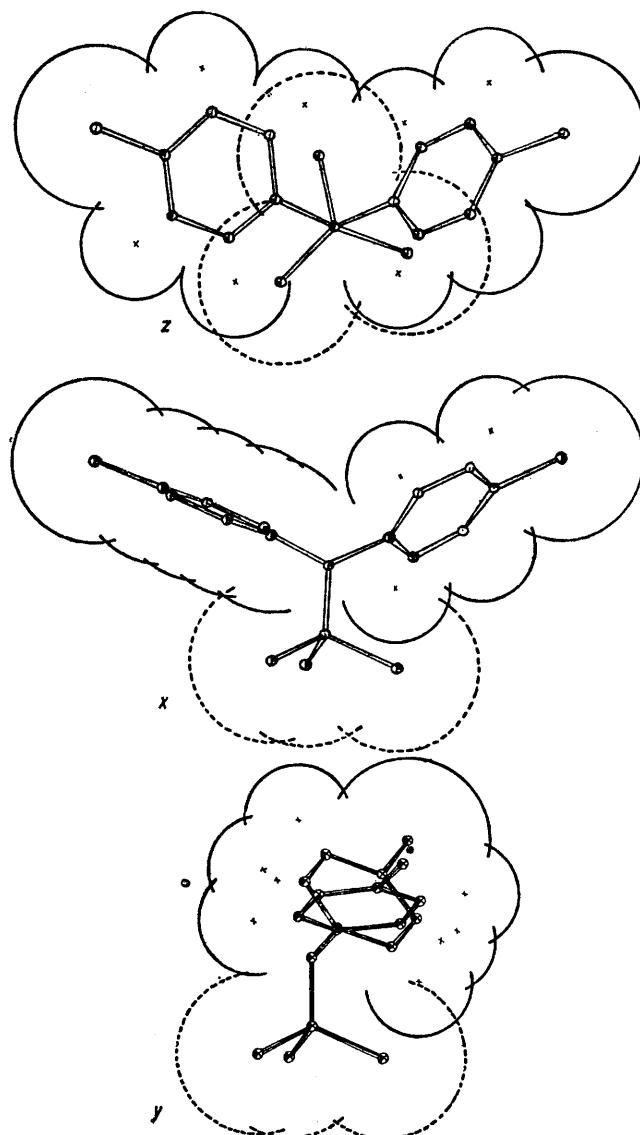
FIGURE 7 Three plane projections of (I) with the van der Waals circumferences drawn in

pm) being the longest. Van der Waals interactions appear to govern the packing (Figure 6).

Extensive structure activity studies have been carried out on DDT analogues. Rogers *et al.*[2] have suggested that the bulky CCl_3 group inhibits rotation of the phenyl rings and forces them to take up a ' butterfly ' configuration in which the planes are coplanar. In

[2] E. F. Rogers, H. D. Brown, I. M. Rasmussen, and R. E. Neal, *J. Amer. Chem. Soc.*, 1953, **75**, 2991.

contrast, Riemschneider and Otto[3] argued that some ability of the phenyl groups to rotate was a requirement for activity.

However, it was postulated that free rotation was required to permit ' almost planar ' configuration. For this reason, the inactivity of o,o'-DDT is said to be due
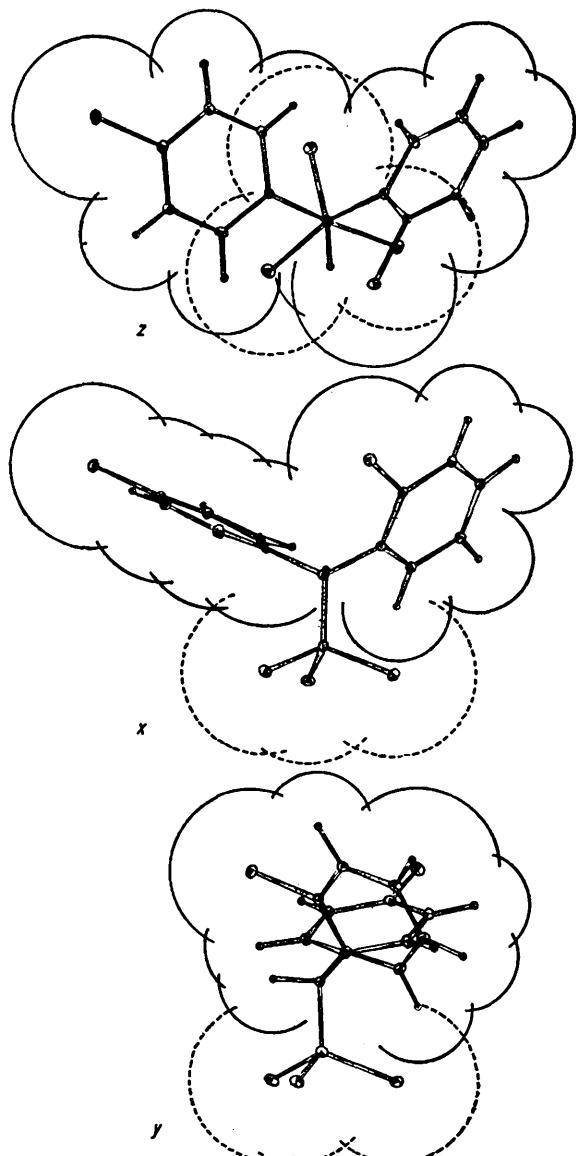

FIGURE 8   Three plane projections of (II) with the van der Waals circumferences drawn in

to two of the chlorine substituents restricting rotation. But o,p'-DDT does have such restriction, yet is a perfectly good insecticide.[4]

Figures 7 and 8 show the three-plane projections of

[3] R. Riemschneider and H. D. Otto, Z. Naturforsch., 1954, 9b, 95.
[4] R. L. Metcalf, 'Organic Insecticides,' 1955, Wiley, New York.
[5] C. J. Johnson, ORTEP plotting program.

both isomers. They were obtained from the refined atomic parameters for the molecules, by use of a thermal ellipsoid plotting programme ORTEP,[5] with a scale of one inch to 100 pm, and a view distance of infinity (parallel projection). The van der Waals circumferences of the periphery atoms in each projection were drawn in to scale.

The van der Waals dimensions of the plane projections are: (a) diameters of the apices, 655 (I) and 645 pm (II); (b) projected areas of the trichloromethane groups,

TABLE 1

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

Atom	x/a	y/b	z/c
Cl(1)	−0·1120(6)	−0·0395(2)	−0·3374(9)
Cl(2)	−0·0501(6)	0·4880(2)	−0·5961(9)
Cl(3)	−0·0252(5)	0·2097(2)	0·2409(8)
Cl(4)	0·0537(4)	0·3464(2)	0·1338(8)
Cl(5)	0·1907(3)	0·2285(2)	0·0004(8)
C(1)	−0·0848(9)	0·1814(6)	−0·1576(16)
C(2)	−0·1766(12)	0·1363(7)	−0·0930(19)
C(3)	−0·1873(17)	0·0699(7)	−0·1494(23)
C(4)	−0·1035(14)	0·0450(7)	−0·2711(26)
C(5)	−0·0059(15)	0·0892(10)	−0·3441(29)
C(6)	−0·0065(13)	0·1601(7)	−0·2862(23)
C(7)	−0·0653(10)	0·3141(6)	−0·2140(16)
C(8)	0·0552(17)	0·3335(7)	−0·2875(27)
C(9)	0·0621(15)	0·3823(9)	−0·4123(25)
C(10)	−0·0541(19)	0·4189(8)	−0·4507(26)
C(11)	−0·1783(15)	0·3999(8)	−0·3749(20)
C(12)	−0·1824(14)	0·3477(5)	−0·2578(20)
C(13)	0·0313(14)	0·2572(7)	0·0599(19)
C(14)	−0·0728(12)	0·2538(6)	−0·0798(18)

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

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cl(1)	262(7)	18(1)	278(10)	8(2)	−38(7)	−12(2)
Cl(2)	324(9)	22(1)	172(7)	−15(2)	−12(8)	14(2)
Cl(3)	185(4)	29(1)	139(6)	2(1)	−1(4)	19(2)
Cl(4)	155(4)	25(1)	155(6)	−9(1)	−9(4)	−10(1)
Cl(5)	101(3)	40(1)	226(6)	13(1)	−29(4)	−17(1)
C(1)	61(9)	25(3)	102(16)	2(4)	−11(10)	7(17)
C(2)	119(14)	25(3)	111(10)	−2(5)	44(13)	24(7)
C(3)	186(20)	21(3)	168(24)	25(6)	28(18)	2(8)
C(4)	86(13)	32(4)	221(31)	15(5)	−53(19)	4(10)
C(5)	111(13)	50(6)	196(29)	15(8)	−23(17)	−23(13)
C(6)	118(15)	29(4)	141(29)	1(6)	25(18)	−6(9)
C(7)	92(10)	25(3)	90(17)	−16(6)	28(11)	−3(6)
C(8)	190(19)	18(3)	209(30)	−13(7)	26(22)	−16(9)
C(9)	129(17)	40(5)	180(32)	−10(7)	6(18)	−6(12)
C(10)	199(24)	29(4)	126(27)	−16(8)	32(18)	−17(8)
C(11)	163(16)	31(4)	107(24)	17(6)	−57(17)	−27(8)
C(12)	148(16)	15(3)	133(22)	2(4)	−1(16)	11(7)
C(13)	144(17)	23(3)	121(22)	1(6)	−9(16)	−3(7)
C(14)	102(13)	18(3)	117(20)	−13(4)	25(12)	−12(7)

(c) Interatomic distances (pm); estimated standard deviation Cl–C 1·5, C–C 3 pm

Cl(1)–C(4)	170	Cl(2)–C(10)	175
Cl(3)–C(13)	178	Cl(4)–C(13)	181
Cl(5)–C(13)	174		
C(1)–C(2)	135	C(7)–C(8)	138
C(2)–C(3)	135	C(8)–C(9)	136
C(3)–C(4)	136	C(9)–C(10)	138
C(4)–C(5)	141	C(10)–C(11)	142
C(5)–C(6)	143	C(11)–C(12)	136
C(1)–C(6)	134	C(7)–C(12)	137
C(13)–C(14)	151		
C(1)–C(14)	152	C(7)–C(14)	157

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

TABLE 1 (Continued)

(d) Interatomic angles (deg.); estimated standard deviation 1°

C(2)—C(1)—C(6)	118·8	C(8)—C(7)—C(12)	120·1
C(2)—C(1)—C(14)	118·7	C(8)—C(7)—C(14)	121·5
C(6)—C(1)—C(14)	122·5	C(12)—C(7)—C(14)	118·3
C(1)—C(2)—C(3)	121·8	C(7)—C(8)—C(9)	122·2
C(2)—C(3)—C(4)	121·0	C(8)—C(9)—C(10)	117·7
C(3)—C(4)—C(5)	120·0	C(9)—C(10)—C(11)	120·2
C(3)—C(4)—Cl(1)	121·4	C(9)—C(10)—Cl(2)	120·5
C(5)—C(4)—Cl(1)	118·6	C(11)—C(10)—Cl(2)	119·4
C(4)—C(5)—C(6)	115·8	C(10)—C(11)—C(12)	120·0
C(1)—C(6)—C(5)	122·1	C(11)—C(12)—C(7)	119·3
C(1)—C(14)—C(7)	113·6	Cl(5)—C(13)—Cl(4)	105·8
C(1)—C(14)—C(13)	112·8	Cl(5)—C(13)—Cl(3)	110·0
C(7)—C(14)—C(13)	115·4	Cl(5)—C(13)—C(14)	114·2
Cl(3)—C(13)—C(14)	110·4	Cl(4)—C(13)—Cl(3)	105·0
		Cl(4)—C(13)—C(14)	111·0

0·296 and 0·283 nm²; (c) heights of the projections (x and y), 870 and 880 pm; (d) lengths of the projections (x and z), 1400 and 1240 pm; and (e) distance between two electro-negative chlorine atoms 1040 and 710 pm.

This means that the diameter and projected areas of the two apices and the overall heights of the two isomers are within experimental error but the overall lengths and distance between the two electronegative centres are not.

EXPERIMENTAL

1,1-Bis-(p-chlorophenyl)-2,2,2-trichloroethane, (I)

Crystal Data.—$C_{14}H_9Cl_5$, $M = 345·5$. Orthorhombic, $a = 996·3(1)$ $b = 1920·0(2)$, $c = 788·7(1)$ pm *, $U = 1·509$ nm³, $D_m = 1·55$ (flotation), $Z = 4$; $D_c = 1·56$, $F(000) = 712$. Space group, $Pca2_1(C_{2v}^5$, No. 29). Cu-K_α radiation $\lambda = 154·18$ cm; $\mu(Cu-K_\alpha) = 86·65$ cm⁻¹.

A needle-shaped crystal (0·1 × 0·1 × 0·4) mm was mounted with the (001) face perpendicular to the ϕ axis of a Picker four-circle diffractometer. The cell dimensions were derived from seven axial reflections (0,0,2, 0,0,4, 0,6,0, 0,7,0, 0,11,0, 2,0,0, and 4,0,0). Intensity data were collected up to 2θ 127°. A standard reflection, collected every 50 reflections was used to check crystal drift and decomposition, and showed a reduction of 20% over the collection period. 900 out of 1247 independent reflections were considered observed. The measured intensities were corrected for Lorentz and polarization factors, absorption (gaussian integration, grid size 8 × 8 × 8), and crystal decomposition. No corrections were made for extinction.

Structure Determination and Refinement.—The locations

---

* Previous values: $a = 1000$, $b = 1920$, $c = 784$ pm,[6,7] and $a = 996(4)$, $b = 1914(8)$, and $c = 785(4)$ pm.[8,9]

† See note about Supplementary Publications in Notice to Authors No. 7 in *J. Chem. Soc. (A)*, 1970, Issue No. 20

[6] H. Wild and E. Brandenberger, *Helv. Chim. Acta*, 1945, **28**, 1292.

[7] H. Wild and E. Brandenberger, *Helv. Chim. Acta*, 1946, **29**, 1024.

[8] M. Schneider and I. Fankuchen, *J. Amer. Chem. Soc.*, 1946, **68**, 2669.

[9] I. Fankuchen, M. Schneider and, G. Singer, *Science*, 1946, **103**, 25.

of the chlorine atoms were obtained by solution of a sharpened F^2 synthesis. Successive electron-density syntheses enabled location of the other chlorines and the carbon atoms. Full-matrix least-squares refinement, with unit weights, reduced R to 0·091 and R' to 0·106 (where $R' = [\Sigma w|F_o - F_c|^2/\Sigma|F_o|^2]^{\frac{1}{2}}$). A difference-Fourier synthesis failed to reveal unambiguously the positions of the hydrogen atoms.

Atomic parameters, and interatomic distances and angles are listed in Table 1. Observed and calculated structure amplitudes for both compounds are listed in Supplementary Publication No. SUP 20476 (14 pp., 1 microfiche).†

TABLE 2

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

Atom	x/a	y/b	z/c	B/Å²
Cl(11)	−0·0447(1)	0·2629(1)	0·5872(1)	•
Cl(12)	−0·0351(1)	0·8107(1)	0·7257(1)	
Cl(13)	0·3827(1)	0·7949(1)	0·8325(1)	
Cl(14)	0·3318(1)	0·9687(1)	1·0297(1)	
Cl(15)	0·2846(1)	0·7325(1)	1·0511(1)	
C(11)	0·1073(4)	0·6491(4)	0·8023(4)	
C(12)	0·0309(4)	0·5690(4)	0·8573(4)	
C(13)	−0·0171(4)	0·4490(4)	0·7903(5)	
C(14)	0·0141(4)	0·4119(4)	0·6690(5)	
C(15)	0·0889(4)	0·4895(4)	0·6131(4)	
C(16)	0·1339(4)	0·6085(4)	0·6803(4)	
C(17)	0·0641(4)	0·8186(3)	0·9522(4)	
C(18)	0·0632(4)	0·8380(4)	1·0836(5)	
C(19)	−0·0268(5)	0·8695(4)	1·1481(5)	
C(110)	−0·1164(5)	0·8822(4)	1·0825(6)	
C(111)	−0·1179(4)	0·8643(4)	0·9534(6)	
C(112)	−0·0280(4)	0·8327(4)	0·8892(4)	
C(113)	0·1560(4)	0·7810(3)	0·8729(4)	
C(114)	0·2795(4)	0·8170(4)	0·9439(4)	
H(12)	0·007	0·592	0·944	5·0
H(13)	−0·077	0·391	0·838	5·0
H(15)	0·104	0·464	0·534	5·0
H(16)	0·182	0·667	0·646	5·0
H(18)	0·124	0·826	1·129	5·0
H(19)	−0·023	0·881	1·241	5·0
H(110)	−0·196	0·906	1·128	5·0
H(111)	−0·174	0·872	0·908	5·0
H(113)	0·176	0·821	0·805	5·0
Cl(21)	0·2981(1)	0·4630(1)	1·0844(1)	•
Cl(22)	0·6558(1)	0·3405(1)	0·5890(1)	
Cl(23)	0·3204(1)	−0·0249(1)	0·6387(1)	
Cl(24)	0·3399(1)	−0·0152(1)	0·3806	
Cl(25)	0·1597(1)	0·0525(1)	0·5219(1)	
C(21)	0·3733(4)	0·2542(4)	0·7075(4)	
C(22)	0·2977(4)	0·3187(4)	0·7128(4)	
C(23)	0·2761(4)	0·3837(4)	0·8281(5)	
C(24)	0·3290(4)	0·3843(4)	0·9383(4)	
C(25)	0·4051(4)	0·3225(4)	0·9370(5)	
C(26)	0·4270(4)	0·2570(4)	0·8220(5)	
C(27)	0·4267(4)	0·2559(4)	0·4862(4)	
C(28)	0·3416(4)	0·2580(4)	0·4009(5)	
C(29)	0·3678(5)	0·3287(4)	0·3218(5)	
C(210)	0·4808(5)	0·4041(4)	0·3252(5)	
C(211)	0·5686(4)	0·4062(4)	0·4078(5)	
C(212)	0·5396(4)	0·3315(4)	0·4869(4)	
C(213)	0·4011(4)	0·1851(4)	0·5824(4)	
C(214)	0·3099(4)	0·0578(4)	0·5326(4)	
H(22)	0·266	0·324	0·640	5·0
H(23)	0·222	0·431	0·831	5·0
H(25)	0·422	0·326	1·017	5·0
H(26)	0·485	0·214	0·826	5·0
H(28)	0·261	0·202	0·397	5·0
H(29)	0·296	0·321	0·263	5·0
H(210)	0·506	0·462	0·270	5·0
H(211)	0·660	0·462	0·413	5·0
H(213)	0·472	0·160	0·593	5·0

TABLE 2 (Continued)

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

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cl(11)	149(2)	68(1)	122(2)	18(1)	−15(1)	−4(1)
Cl(12)	108(1)	124(2)	101(2)	58(1)	−13(1)	17(1)
Cl(13)	65(1)	117(1)	149(2)	33(1)	20(1)	−10(1)
Cl(14)	83(1)	77(1)	140(2)	17(1)	1(1)	−12(1)
Cl(15)	107(1)	127(2)	152(2)	55(1)	−21(1)	50(1)
C(11)	56(4)	67(5)	67(5)	31(4)	6(4)	11(4)
C(12)	82(5)	75(5)	85(6)	23(4)	20(4)	27(5)
C(13)	96(6)	72(5)	92(6)	21(4)	7(5)	23(5)
C(14)	81(5)	58(5)	104(7)	19(4)	−17(5)	14(5)
C(15)	81(5)	84(6)	73(5)	30(4)	−3(4)	7(5)
C(16)	59(4)	82(5)	90(6)	25(4)	4(4)	26(5)
C(17)	53(5)	53(4)	96(6)	17(3)	9(4)	19(4)
C(18)	89(6)	81(5)	93(6)	37(4)	21(5)	26(5)
C(19)	113(6)	81(5)	108(7)	23(4)	36(6)	25(5)
C(110)	87(6)	60(5)	154(9)	25(4)	41(6)	15(5)
C(111)	63(5)	69(5)	157(8)	23(4)	6(5)	15(5)
C(112)	66(5)	57(4)	88(6)	16(4)	6(4)	14(4)
C(113)	54(4)	61(4)	83(5)	21(3)	3(4)	18(4)
C(114)	70(5)	69(5)	100(6)	25(4)	8(4)	14(4)
Cl(21)	167(2)	101(1)	93(2)	57(1)	23(1)	25(1)
Cl(22)	72(1)	125(2)	133(2)	29(1)	3(1)	40(1)
Cl(23)	147(2)	88(1)	146(2)	38(1)	21(1)	52(1)
Cl(24)	146(2)	99(1)	113(2)	43(1)	28(1)	5(1)
Cl(25)	77(1)	91(1)	142(2)	11(1)	8(1)	23(1)
C(21)	64(5)	72(5)	86(6)	18(4)	3(4)	30(4)
C(22)	91(5)	78(5)	76(6)	24(4)	3(4)	35(4)
C(23)	95(6)	91(6)	86(6)	33(4)	−1(5)	33(5)
C(24)	93(6)	67(5)	85(7)	21(4)	15(5)	16(4)
C(25)	96(6)	94(6)	93(7)	27(5)	−11(5)	23(5)
C(26)	85(5)	99(6)	100(7)	42(4)	−1(5)	27(5)
C(27)	66(5)	73(5)	79(6)	17(4)	7(4)	22(4)
C(28)	83(5)	99(6)	97(6)	12(4)	−0(5)	36(5)
C(29)	102(6)	85(5)	97(6)	11(5)	−6(5)	33(5)
C(210)	130(7)	82(5)	78(6)	33(5)	22(5)	17(5)
C(211)	91(5)	75(5)	75(6)	31(4)	31(5)	6(4)
C(212)	65(5)	83(5)	62(5)	33(4)	6(4)	2(4)
C(213)	69(5)	74(5)	88(6)	22(4)	3(4)	22(4)
C(214)	104(6)	87(5)	92(6)	37(4)	25(5)	32(4)

* Defined as for Table 1.

(c) Interatomic distances (pm); estimated standard deviations, C–Cl 0·5, C–C 0·8, C–H 8 pm

	Molecule (1)	Molecule (2)
Cl(1)–C(4)	173·7	175·0
Cl(2)–C(12)	173·4	174·8
Cl(3)–C(14)	177·4	177·4
Cl(4)–C(14)	177·1	177·1
Cl(5)–C(14)	178·7	178·7
C(1)–C(2)	138·8	138·9
C(2)–C(3)	140·1	138·2
C(3)–C(4)	138·0	135·9
C(4)–C(5)	136·7	137·3
C(5)–C(6)	139·1	138·2
C(6)–C(1)	137·3	139·5
C(7)–C(8)	139·3	139·4
C(8)–C(9)	139·8	136·1
C(9)–C(10)	136·8	137·1
C(10)–C(11)	136·9	137·7
C(11)–C(12)	139·4	139·1
C(12)–C(7)	138·6	137·8
C(13)–C(1)	153·1	151·3
C(13)–C(7)	152·2	151·8
C(13)–C(14)	154·4	155·7
H(2)–C(2)	99	91
H(3)–C(3)	107	100
H(5)–C(5)	88	96
H(6)–C(6)	93	102
H(8)–C(8)	96	99
H(9)–C(9)	98	104
H(10)–C(10)	116	103
H(11)–C(11)	88	108
H(13)–C(13)	99	101

TABLE 2 (Continued)

(d) Interatomic angles (deg.); estimated standard deviation 0·4°, angles involving H atoms, 5°

	Molecule (1)	Molecule (2)
C(2)–C(1)–C(6)	118·6	118·0
C(2)–C(1)–C(13)	121·0	122·1
C(6)–C(1)–C(13)	120·3	119·9
C(1)–C(2)–C(3)	120·8	121·0
C(1)–C(2)–H(2)	123	121
C(3)–C(2)–H(2)	116	118
C(2)–C(3)–C(4)	118·6	119·6
C(2)–C(3)–H(3)	117	121
C(4)–C(3)–H(3)	124	120
C(3)–C(4)–C(5)	121·5	121·1
C(3)–C(4)–Cl(1)	118·4	119·6
C(5)–C(4)–Cl(1)	120·2	119·3
C(4)–C(5)–C(6)	119·0	119·6
C(4)–C(5)–H(5)	119	119
C(6)–C(5)–H(5)	122	122
C(5)–C(6)–C(1)	121·5	120·6
C(5)–C(6)–H(6)	123	117
C(1)–C(6)–H(6)	115	123
C(8)–C(7)–C(12)	116·8	115·4
C(8)–C(7)–C(13)	125·3	124·8
C(12)–C(7)–C(13)	117·9	119·6
C(7)–C(8)–C(9)	121·3	122·7
C(7)–C(8)–H(8)	118	116
C(9)–C(8)–H(8)	120	121
C(8)–C(9)–C(10)	120·2	120·5
C(8)–C(9)–H(9)	118	115
C(10)–C(9)–H(9)	122	125
C(9)–C(10)–C(11)	119·9	119·5
C(9)–C(10)–H(10)	125	124
C(11)–C(10)–H(10)	115	116
C(10)–C(11)–C(12)	119·8	118·8
C(10)–C(11)–H(11)	123	123
C(12)–C(11)–H(11)	118	118
C(11)–C(12)–C(7)	122·0	123·2
C(11)–C(12)–Cl(2)	117·6	115·9
C(7)–C(12)–Cl(2)	120·4	120·9
C(1)–C(13)–C(7)	110·7	111·7
C(1)–C(13)–C(14)	112·2	112·7
C(7)–C(13)–C(14)	115·7	115·3
C(1)–C(13)–H(13)	105	113
C(7)–C(13)–H(13)	112	108
C(14)–C(13)–H(13)	101	96
C(13)–C(14)–Cl(5)	113·7	113·9
C(13)–C(14)–Cl(4)	111·4	110·9
C(13)–C(14)–Cl(3)	109·8	109·2
Cl(5)–C(14)–Cl(4)	108·9	108·1
Cl(5)–C(14)–Cl(3)	106·2	106·7
Cl(4)–C(14)–Cl(3)	106·9	107·8

1-(o-*Chlorophenyl*)-1-(p-*chlorophenyl*)-2,2,2-*trichloroethane*, (II)

This isomer was separated from technical-grade DDT by repeated recrystallization from light petroleum and methyl alcohol.

Crystal Data.—$C_{14}H_9Cl_5$, $M = 354·5$. Triclinic, $a = 1201·6(1)$, $b = 1248·1(1)$, $c = 1093·8(1)$ pm, $\alpha = 105·43(3)$, $\beta = 89·93(3)$, $\gamma = 109·03(3)°$ †, $U = 1·460$ nm³, $D_m =$

† Previous values: $a = 2580$, $b = 1520$, $c = 1955$ pm, $\alpha = 104·15°$, $\beta = 95·40°$, $\gamma = 99·38°$.[10]

[10] H. Wild and E. Brandenberger, *Helv. Chim. Acta*, 1946. **29**, 1035.

1·61, $Z = 4$, $D_c = 1·59$, $F(000) = 712$. Space group $P\bar{1}$ $(C_i$, No. 2). Mo-K_α radiation, $\lambda = 71·07$ pm; $\mu(\text{Mo-}K_\alpha) = 9·4$ cm^{-1}.

A plate-shaped crystal $(0·3 \times 0·2 \times 0·05)$ mm was mounted in a general orientation on a Hilger and Watts four-circle diffractometer. 2649 out of 3600 independent reflections were collected up to 2θ 44°. No change in the intensity of the standard reflections was observed during data collection. The measured intensities were corrected for Lorentz and polarization factors, and for X-ray absorption (gaussian integration, grid size of $4 \times 4 \times 4$).

Structure Determination.—The structure was solved by the symbolic addition phase-determining technique using the largest 287 E values. Ten chlorine atoms were located from an initial E map calculated with 287 phased E values. Successive electron-density syntheses ennabled location of all the 28 carbons. Block-diagonal approximation and full-matrix least-squares refinement reduced R to 0·048 and R' to 0·044. All hydrogen atoms were located. Although the least squares had not converged, refinement was halted

at this stage. A final difference-Fourier failed to reveal any unaccounted electron density. A plot of I_c/I_o vs. I_o indicated that three low-angle high-intensity reflections were seriously effected by extinction. They were removed before the last cycle of refinement.

Atomic parameters and interatomic distances and angles are listed in Table 2.

The scattering factors used were those for chlorine, carbon,[11] and hydrogen.[12] No corrections were applied for anomalous dispersion. All calculations were carried out with local versions of standard programmes.[13]

[11] ' International Tables for X-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962, p. 201.
[12] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

[13] FAME–MAGIC (Symbolic Addition), Dewar and Stone, modified by R. C. Secombe, 1970; PREFOUR (Structure factor), FOURIER, J. Blount, 1966; ORFLS (Full-matrix least-squares), W. R. Busing, K. O. Martin, and H. A. Levy, 1964; DIGLS (Diagonal least-squares) for IBM 1130.