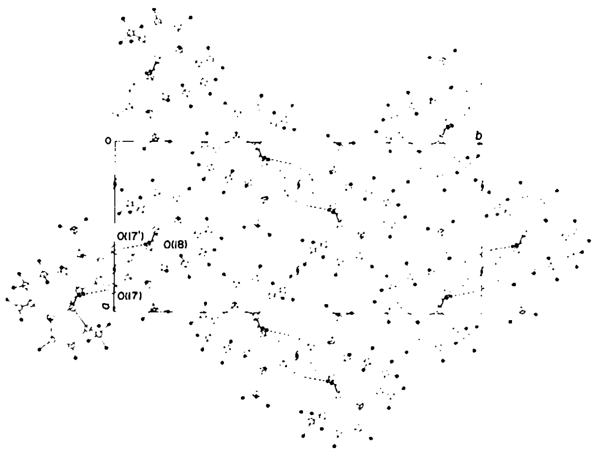

Fig. 3. Molecular packing viewed down **c**. The broken lines indicate the O–H···O hydrogen bonds.

from the plane. The structure of the lactone ring is essentially the same as in (II); the five atoms, C(6)–O(16)–C(12).O(17)–C(11), are coplanar (±0·02 Å) and only C(7) deviates, by 0·52 Å, from the plane. In both five-membered rings, the bond angles at the C(sp^3) atoms are systematically smaller than the tetrahedral value, the average being 102°.

The molecular packing viewed down **c** is shown in Fig. 3. The hydroxyl group O(18)–H(18) of a molecule is hydrogen-bonded to the carbonyl O(17') of an adjacent molecule in a helical sequence; thus, an infinite molecular column is formed along **c** through O–H···O hydrogen bonds. The hydrogen-bond distances are O(18)···O(17') = 2·850 and H(18)···O(17') = 1·97 Å. Molecular columns are stacked with normal van der Waals contacts.

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References

Asher, J. D. M. & Sim, G. A. (1965). *J. Chem. Soc.* pp. 1584–1594.

Fujimoto, Y., Shimizu, T. & Tatsuno, T. (1976*a*). *Chem. Pharm. Bull.* **24**, 365–367.

Fujimoto, Y., Shimizu, T. & Tatsuno, T. (1976*b*). *Tetrahedron Lett.* pp. 2041–2044.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Sakurai, T., Iwasaki, H., Watanabe, Y., Kobayashi, K., Bando, Y. & Nakamichi, Y. (1974). *Rep. Inst. Phys. Chem. Res.* **50**, 75–91.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1978). B**34**, 1011–1014

Perchloro-1,2,3-triphenylnaphthalene

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Abstract. $C_{28}Cl_{20}$, monoclinic, $P2_1/a$, $a = 23{\cdot}426$ (4), $b = 13{\cdot}284$ (2), $c = 11{\cdot}592$ (2) Å, $\beta = 98{\cdot}29$ (7)°, $Z = 4$, $D_x = 1{\cdot}945$ g cm^{-3}. The structure has been determined by direct methods and refined to $R = 0{\cdot}059$ for 5607 independent reflexions. The naphthalene system is very distorted owing to repulsions among its bulky substituents.

Introduction. Transparent, colourless crystals suitable for X-ray studies were grown by slow evaporation of a toluene solution at room temperature. Preliminary unit-cell parameters and the space group were obtained from Weissenberg photographs.

The dimensions of the crystal selected were 0·3 × 0·3 × 0·3 mm. Precise lattice parameters were determined by least-squares refinement of the θ values for 25 reflexions measured with a Philips PW 1100 four-circle diffractometer, on which all further experimental work was carried out. Monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å), pulse-height discrimination and the ω-scan mode [scan width = 1·3° (θ), scan speed = 0·043° (θ) s^{-1}] were used. 10419 independent reflexions were collected in the range $2° \le \theta \le 30°$; 4812 of these were considered as unobserved according to the criterion $I < 2\sigma(I)$, and were not used in calculations. Only Lorentz and polarization corrections were made.

The structure was solved by direct methods using the *MULTAN* system of computer programs (Main, Woolfson, Lessinger, Germain & Declercq, 1974). Overall temperature ($B = 3{\cdot}037$ Å^2) and scale factors were determined and used to compute normalized structure factors by the program *NORMAL* included in *MULTAN*. 284 reflexions with $E \ge 2{\cdot}2$ were used to determine the starting sets. An E map calculated with the set of signs with the highest figures of merit revealed peaks for 42 atoms. A subsequent electron density synthesis revealed the positions of the remaining atoms, and showed that the compound is a naphthalene derivative. Refinements were applied by the program *NRC*-10 of Ahmed, Hall, Pippy & Huber (1966). The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, with the weights of Cruickshank (1965): $w = 1/(a + |F_o| + c|F_o|^2)$, where $a = 2F_{\rm min}$, and $c = 2/F_{\rm max}$. After ten cycles of block-diagonal least-squares calculation with individual isotropic and five cycles with anisotropic thermal parameters, the refinement was terminated at $R = 0{\cdot}059$ for 5607 reflexions ($R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$). The final atomic parameters are listed in Table 1. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). Fig. 1 shows a view of the molecule (*ORTEP*, Johnson, 1965) and the numbering of the atoms.*

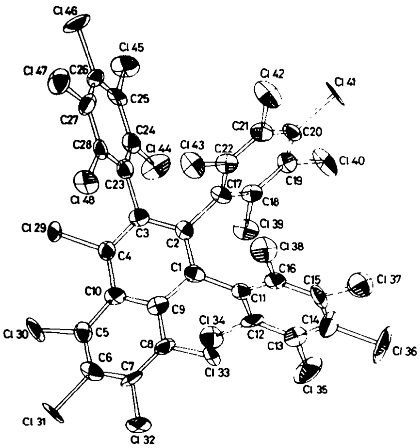

Fig. 1. View of the molecule drawn by *ORTEP* showing thermal ellipsoids and the numbering of the atoms.

Table 1. *Final atomic parameters* (×10^5) *with standard deviations in parentheses*

	x	y	z		x	y	z
C(1)	16021 (23)	39667 (37)	32523 (44)	C(25)	6635 (28)	8519 (40)	−2717 (55)
C(2)	14555 (21)	33411 (37)	23154 (44)	C(26)	1933 (25)	12768 (43)	−9634 (48)
C(3)	9311 (22)	27774 (39)	22129 (46)	C(27)	−421 (24)	21587 (48)	−6152 (52)
C(4)	6317 (24)	27165 (41)	31671 (53)	C(28)	1904 (21)	26258 (40)	4180 (53)
C(5)	4473 (26)	33973 (42)	51324 (50)	Cl(29)	610 (6)	18655 (12)	30170 (15)
C(6)	5154 (25)	42109 (47)	58732 (49)	Cl(30)	239 (8)	24325 (13)	54945 (16)
C(7)	8860 (24)	50108 (45)	57236 (48)	Cl(31)	1277 (8)	42656 (16)	70266 (15)
C(8)	12302 (25)	49387 (43)	48510 (50)	Cl(32)	8999 (7)	60503 (12)	65774 (15)
C(9)	11944 (23)	40973 (39)	40818 (46)	Cl(33)	16542 (8)	59647 (12)	46659 (17)
C(10)	7540 (23)	33767 (39)	41422 (50)	Cl(34)	22989 (7)	30564 (13)	53733 (13)
C(11)	22045 (24)	43689 (39)	35484 (48)	Cl(35)	35695 (8)	36992 (20)	59647 (19)
C(12)	25662 (25)	39557 (44)	45204 (49)	Cl(36)	40592 (11)	53731 (28)	45286 (32)
C(13)	31322 (30)	42556 (57)	48099 (58)	Cl(37)	32781 (11)	63950 (17)	24648 (26)
C(14)	33596 (31)	49829 (67)	41606 (77)	Cl(38)	20057 (8)	57208 (12)	18217 (16)
C(15)	30082 (31)	54255 (50)	32158 (67)	Cl(39)	25936 (6)	21206 (10)	27924 (11)
C(16)	24359 (29)	51002 (43)	29236 (52)	Cl(40)	33869 (6)	21882 (14)	8864 (15)
C(17)	18357 (22)	32650 (39)	13661 (44)	Cl(41)	29933 (7)	32998 (12)	−14490 (13)
C(18)	23712 (22)	27694 (37)	15273 (42)	Cl(42)	18185 (8)	43687 (16)	−18626 (14)
C(19)	27295 (21)	27799 (41)	6601 (51)	Cl(43)	10471 (6)	44371 (11)	620 (13)
C(20)	25580 (25)	32716 (40)	−3902 (46)	Cl(44)	14659 (7)	7788 (11)	16578 (16)
C(21)	20231 (25)	37495 (41)	−5784 (44)	Cl(45)	9386 (9)	−2839 (12)	−6337 (17)
C(22)	16733 (21)	37621 (39)	2923 (46)	Cl(46)	−1139 (9)	6727 (14)	−21994 (15)
C(23)	6873 (22)	22544 (40)	11129 (49)	Cl(47)	−6388 (6)	26703 (15)	−14432 (15)
C(24)	9037 (24)	13355 (39)	7620 (50)	Cl(48)	−1449 (6)	36723 (11)	8871 (15)

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* Lists of structure factors, anisotropic thermal parameters and perchlorophenyl mean-plane equations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33182 (50 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. It has been found (Ballester, Castañer & Armet, 1977) that perchlorodiphenylacetylene undergoes thermal dimerization giving, among other products, an unknown white chlorocarbon melting at about 350 °C. Structural elucidation of this product by spectral and chemical techniques (IR, UV, mass) posed insurmountable problems. Its empirical formula is $C_{28}Cl_{20}$, as ascertained by elemental and mass spectrum analysis. No indication of either double or triple bonds was found (IR).

On account of the extremely high thermal and chemical stability of this chlorocarbon, degradation studies were not possible (it withstands hot sulphuric and nitric acids, as well as their hot mixtures). For this reason it was considered that only X-ray analysis could provide the decisive data necessary concerning its structure.

The naphthalene ring system is very distorted owing to steric interactions among the bulky substituents: whereas the closest approach between Cl atoms in a molecule is about 3 Å (Brown & Strydom, 1974), the adjacent α-substituents would be separated by only 2·4 Å in the hypothetical planar model of naphthalene (Ballester & Olivella, 1974).

Therefore, it was expected that certain angles would differ significantly from their regular values, and out-of-mean-plane displacements of the α-C atoms and their Cl substituents would also occur, as in 1,4,5,8-tetrachloronaphthalene (Gafner & Herbstein, 1962) and octachloronaphthalene (Gafner & Herbstein, 1963).

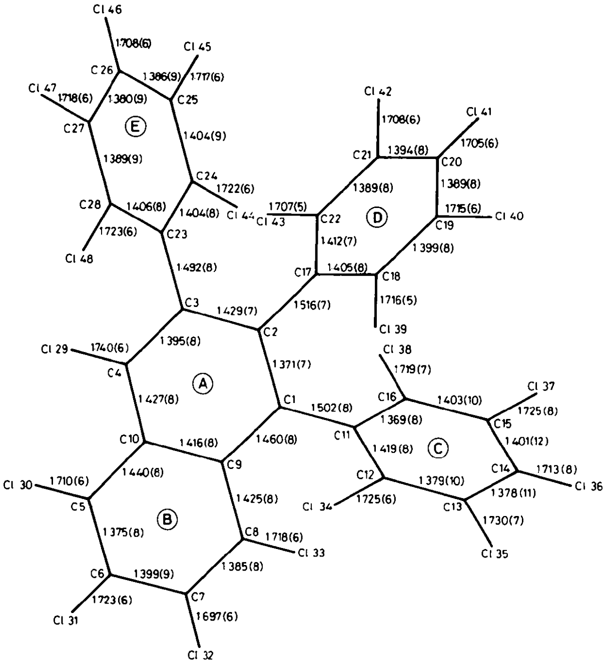

Fig. 2. Bond lengths (Å).

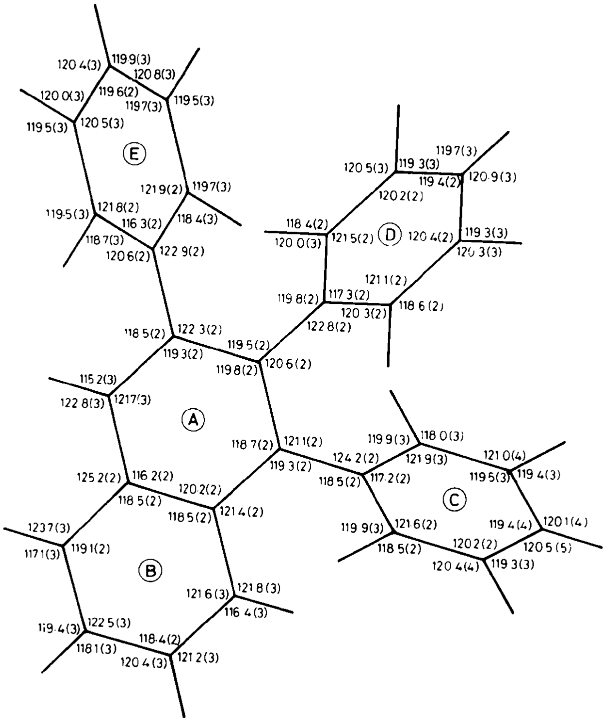

Fig. 3. Bond angles (°).

Table 2. *Least-squares mean plane and deviations* ($\times 10^3$ Å) *of atoms from this plane*

X, Y and Z are coordinates in Å referred to an orthogonal system of axes having X and Y along **a** and **b**, and Z along **c***. Fractional coordinates x, y, z in the system are related to X, Y, Z by the matrix equation

$$\begin{vmatrix} 23\cdot426 & 0 & 0 \\ 0 & 13\cdot284 & 0 \\ -1\cdot67369 & 0 & 11\cdot47088 \end{vmatrix} \begin{vmatrix} x \\ y \\ z \end{vmatrix} = \begin{vmatrix} X \\ Y \\ Z \end{vmatrix}.$$

Atoms included in the least-squares calculation are indicated by asterisks.

Naphthalene: plane *AB*

$$0\cdot5587X - 0\cdot6129Y + 0\cdot5588Z - 0\cdot4169 = 0$$

C(1)*	231 (5)	C(10)*	89 (6)
C(2)*	36 (5)	Cl(29)	−204 (2)
C(3)*	−248 (5)	Cl(30)	643 (2)
C(4)*	−67 (6)	Cl(31)	126 (2)
C(5)*	213 (6)	Cl(32)	−563 (2)
C(6)*	46 (6)	Cl(33)	−553 (2)
C(7)*	−202 (6)	C(11)	855 (6)
C(8)*	−171 (6)	C(17)	76 (5)
C(9)*	46 (5)	C(23)	−743 (6)

One of the largest compressions occurs between Cl(29) and Cl(30) [Cl(29)–Cl(30) = 2·982 Å, and Cl(29)–C(4)–C(10), C(4)–C(10)–C(5) and C(10)–C(5)–Cl(30) are increased to 122·8, 125·2 and 123·7°

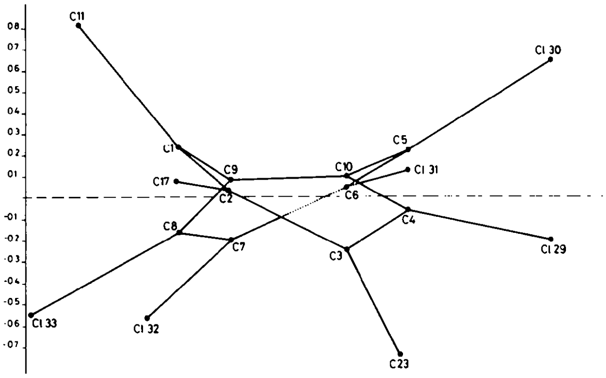

Fig. 4. Naphthalene rings: conformation and least-squares plane (vertical scale in Å).

Table 3. *Selected torsion angles* (°) *of naphthalene rings*

C(9)–C(1)–C(2)–C(3)	4·5 (8)
C(1)–C(2)–C(3)–C(4)	12·4 (8)
C(2)–C(3)–C(4)–C(10)	−14·8 (8)
C(3)–C(4)–C(10)–C(9)	−0·2 (8)
C(1)–C(9)–C(10)–C(4)	17·4 (8)
C(5)–C(6)–C(7)–C(8)	5·3 (9)
C(6)–C(7)–C(8)–C(9)	−2·9 (9)
C(7)–C(8)–C(9)–C(10)	−6·7 (9)
C(8)–C(9)–C(10)–C(5)	13·7 (8)
C(6)–C(5)–C(10)–C(9)	−11·5 (8)
C(3)–C(4)–C(10)–C(5)	−176·8 (6)
C(6)–C(5)–C(10)–C(4)	164·9 (6)
C(7)–C(8)–C(9)–C(1)	172·9 (5)
C(2)–C(1)–C(9)–C(8)	160·6 (5)
C(1)–C(9)–C(10)–C(5)	−165·8 (5)
C(8)–C(9)–C(10)–C(4)	−163·1 (5)

respectively (Figs. 2 and 3)]. Cl(29) and Cl(30), like Cl(33) and the pentachlorophenyl group, are displaced from the mean plane of naphthalene in opposite senses. However, α and β adjacent substituents deviate in the same sense. The deviations of the naphthalene C atoms follow, but are smaller than, those of the substituents on these atoms (Table 2). The distortion of naphthalene is such that the benzene rings individually tend to be planar. The symmetry of naphthalene is lost, but the resulting conformation maintains a pseudo twofold axis perpendicular to the best ring plane (Fig. 4), as in octachloronaphthalene.

Benzene rings *C*, *D* and *E* (Fig. 2) are planar. In general, Cl atoms in *ortho* positions [Cl(34), Cl(38), Cl(39), Cl(44) and Cl(48)] deviate more than other Cl atoms from the mean plane because they suffer more steric compression owing to the arrangement of the pentachlorophenyl groups in the molecule. Angles between the naphthalene mean plane and the benzene rings are variable, depending on steric interactions between some Cl atoms in the α position and *ortho* Cl atoms. The naphthalene plane and benzene ring *D* are nearly perpendicular (95°). Rings *C* and *E* make angles of 71·8 and 77·3° respectively with naphthalene. Selected torsion angles are given in Table 3.

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References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1966). NRC Crystallographic Programs for the IBM/360 System. National Research Council, Ottawa, Canada.

Ballester, M., Castañer, J. & Armet, O. (1977). In preparation.

Ballester, M. & Olivella, S. (1974). *Polychloroaromatic Compounds*, p. 41, edited by H. Suschitzky. New York: Plenum.

Brown, G. M. & Strydom, O. A. W. (1974). *Acta Cryst.* B**30**, 801–804.

Cruickshank, D. W. J. (1965). *Computing Methods in Crystallography*, edited by J. S. Rollett, pp. 112–116. Oxford: Pergamon Press.

Gafner, G. & Herbstein, F. H. (1962). *Acta Cryst.* **15**, 1081–1092.

Gafner, G. & Herbstein, F. H. (1963). *Nature* (*London*), **200**, 130–132.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. P. (1974). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.