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## 2:1 Adduct of Perchloro-5,10-diphenyldibenzo[*a,e*]pentalene and Carbon Tetrachloride

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**Abstract.**  $C_{28}Cl_{18} \cdot \frac{1}{2}CCl_4$ ,  $M_r = 1051.4$ , monoclinic,  $C2/c$ ,  $a = 25.794$  (14),  $b = 8.676$  (4),  $c = 17.258$  (9) Å,  $\beta = 94.47$  (4)°,  $V = 3849$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $D_c = 1.81$  Mg m<sup>-3</sup>. The structure was solved with *MULTAN* and refined by block-diagonal least-squares methods. The final  $R$  value is 0.057 for 1610 observed reflections. The perchloro-5,10-diphenyldibenzo[*a,e*]pentalene (PDP) and carbon tetrachloride molecules are located at special positions

(inversion centres and twofold axes, respectively), with an occupancy factor of 0.5 for the carbon tetrachloride. The PDP molecule is slightly distorted due principally to repulsions between adjacent non-bonded Cl atoms.

**Introduction.** Ballester and co-workers have studied in detail the thermal reaction of perchlorodiphenylacetylene (Ballester, Castañer & Armet, 1979). Besides the initial compound, the reaction yields three products which are difficult to separate. The crystal structure of one of them, perchloro-1,2,3-triphenyl-naphthalene, has

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been already established by X-ray diffraction (Gali, Solans, Miravittles, Font-Altaba & Armet, 1978). The second unknown chlorocarbon compound, which is deep red in colour, was separated from the rest by fractional crystallization, advantage being taken of its solubility in carbon tetrachloride. The structure resolution of this product by spectral and chemical techniques (IR, UV, mass) posed insurmountable difficulties. For this reason, as in the case of perchloro-1,2,3-triphenyl-naphthalene, it was considered that only X-ray analysis could provide the decisive data necessary to elucidate its structure.

Red prismatic crystals of the title compound were kindly supplied by Professor M. Ballester. They were grown by slow evaporation of a chloroform solution at room temperature. A crystal  $0.2 \times 0.3 \times 0.4$  mm was used for the crystal-data and intensity measurements on a Syntex  $P2_1$  four-circle diffractometer. The unit cell was measured by automatically centring 15 independent reflections and refining the orientation matrix and unit-cell parameters by least squares. Intensities were collected with Mo  $K\alpha$  graphite-monochromated radiation using the  $\omega$ - $2\theta$  scan technique. 2858 independent reflections were collected in the range  $3^\circ \leq 2\theta \leq 47^\circ$ . 1248 of these were considered as unobserved according to the criterion  $I < 3\sigma(I)$ . Only Lorentz and polarization corrections were made.

The observed systematic absences indicated that the possible space groups are  $C2/c$  (centrosymmetric) and  $Cc$  (non-centrosymmetric). The statistical results of  $\langle |E|^2 - 1 \rangle$ ,  $\langle |E| \rangle$ , and the  $N(z)$  distribution (Howells, Phillips & Rogers, 1950) indicated that the most probable space group was  $C2/c$ , yet the structure was considered to be non-centrosymmetric during the resolution process. The phase problem was solved by direct methods (*MULTAN*; Main, Woolfson, Lessinger, Germain & Declercq, 1977), using 300 reflections with  $|E| \geq 1.62$  and 3000  $\sum_2$  relationships that generated 32 sets of starting phases. An  $E$  map computed with the phases from the set with the highest combined figure of merit revealed peaks for 18 Cl and 28 C atoms in chemically sensible positions and showed that the compound is a diphenyldibenzopentalene derivative with a symmetry centre in the C(1)–C(1') bond. Block-diagonal least-squares refinement (modified version of *SFLS*; Ahmed, Hall, Pippy & Huber, 1966) initially with isotropic thermal parameters gave an  $R = 0.122$ , with all observed reflections. A subsequent electron density synthesis showed the existence of two new maxima. The electron density of each of these two maxima is approximately half that of a Cl atom. A new maximum on a binary axis was found in a difference electron density map at the same refinement stage. These three maxima were assigned to the Cl(25), Cl(26) and C(24) atoms respectively, which together with the atoms related by symmetry built up the carbon tetrachloride molecule.

Table 1. Final atomic parameters ( $\times 10^5$ ) with standard deviations in parentheses

	x	y	z
C(1)	26454 (17)	19048 (53)	48136 (28)
C(2)	23484 (18)	15511 (54)	40602 (27)
C(3)	24196 (17)	5361 (55)	34568 (30)
C(4)	20469 (19)	4739 (60)	28212 (29)
C(5)	16223 (19)	14373 (61)	27794 (30)
C(6)	15456 (19)	24562 (58)	33980 (31)
C(7)	19090 (18)	25256 (56)	40259 (26)
C(8)	19241 (18)	34601 (55)	47440 (30)
C(9)	15243 (17)	46704 (57)	49013 (30)
C(10)	15791 (20)	61663 (58)	46332 (34)
C(11)	12161 (21)	72998 (57)	47969 (33)
C(12)	7963 (19)	69442 (58)	52037 (30)
C(13)	7433 (18)	54543 (63)	54834 (32)
C(14)	11043 (18)	43271 (58)	53216 (32)
Cl(15)	29600 (5)	-6276 (18)	34609 (8)
Cl(16)	21257 (6)	-8248 (20)	20827 (9)
Cl(17)	11842 (5)	13798 (21)	19828 (9)
Cl(18)	10011 (6)	36048 (21)	33193 (10)
Cl(19)	20940 (6)	66019 (18)	40999 (11)
Cl(20)	13058 (7)	91632 (16)	44797 (11)
Cl(21)	3442 (5)	83167 (18)	53688 (9)
Cl(22)	2367 (5)	50163 (20)	60360 (10)
Cl(23)	10445 (6)	24798 (17)	56678 (10)
C(24)	50000	35472 (197)	25000
Cl(25)	44497 (15)	23314 (46)	22035 (18)
Cl(26)	48320 (21)	47401 (82)	33038 (40)

Taking into account the electron density as well as the results obtained from the elemental analysis and mass spectrometry, we concluded that the occupation factor for the carbon tetrachloride atoms should be about 0.5.

Cycles of isotropic refinement, where the atoms Cl(25), Cl(26) and C(24) were introduced with an occupation factor of 0.5, were made; the refinement was continued with anisotropic thermal parameters for all the atoms until an  $R = 0.057$  with all observed reflections was reached.\*

The function minimized was  $\sum w(|F_o| - |F_c|)^2$  where  $w = \sigma(F)^{-2}$  and the  $R$  index was defined as  $\sum ||F_o| - |F_c|| / \sum |F_o|$ .

A final difference map showed no significant features. The final atomic positions are given in Table 1. Fig. 1 shows a view of the molecule and the numbering of the atoms, drawn by the *ORTEP* program (Johnson, 1965).

**Discussion.** Fig. 2 and Table 2 give the bond distances and angles of the PDP and carbon tetrachloride molecules respectively.

\* Lists of structure factors, anisotropic thermal parameters and least-squares mean planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34647 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.



