Perchlorobenzylidenecyclohexa-2,5-diene

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Abstract. $C_{13}Cl_{12}$, FW 569·53, triclinic, $P\bar{1}$, $a = 8·415$ (4), $b = 8·598$ (2), $c = 15·410$ (4) Å, $\alpha = 90·38$ (2), $\beta = 97·13$ (3), $\gamma = 118·58$ (2)°, $V = 968·76$ Å³, $Z = 2$, $D_c = 1·952$ g cm⁻³, $\mu = 16·5$ cm⁻¹, λ(Mo $K\alpha$) = 0·7107 Å. The structure has been solved by *MULTAN* and refined by block-diagonal least squares to $R = 0·047$ for 2415 independent reflections. The most important steric interaction in the molecule occurs between Cl(19) and Cl(25).

Introduction. The title compound, synthesized by Ballester, Riera & De la Fuente (1970), is an isomer of perchlorodiphenylmethane (PDM) (I), to which it transforms at 206°C (after melting). PDM has a certain interest since it is a precursor of an inert free radical $(C_6Cl_5)_2CCl$ (PDM radical) (Ballester, Riera, Castañer, Badia & Monso, 1971).

$$\begin{array}{c} Cl \\ | \\ R—C—R \\ | \\ Cl \end{array}$$
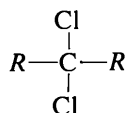
(I) PDM $(R = C_6Cl_5)$

Colourless tabular crystals were obtained from a hexane solution by slow evaporation. The dimensions of the selected crystal were $0·2 \times 0·3 \times 0·4$ mm. Approximate cell parameters were determined from Weissenberg photographs and a morphological study and were refined by least squares from diffractometer observations. The intensities of 2942 independent reflections were measured on a Nonius CAD-4 computer-controlled four-circle diffractometer. In-

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Table 1. *Instrumental settings for the data collection*

Source: Mo K (graphite-monochromatized)
Scan: ω–2θ
θ range: $1° \le \theta \le 35°$
Scan range: $\Delta\omega = (1 + 0·35 \tan \theta)°$
Aperture: $(1·6 + 0·7 \tan \theta)°$
Maximum scan time: 80 s
Number of independent reflections: 2942
Total observed reflections $|I > 2\sigma(I)|$: 2415

strumental settings are given in Table 1. No absorption correction was applied (crystal mounted along c, $\mu r < 0·7$). The space group $P\bar{1}$ was confirmed by the values of the statistical averages $\langle |E^2 - 1| \rangle$, $\langle |E| \rangle$ and the $N(z)$ distribution (Howells, Phillips & Rogers, 1950).

The structure was solved by direct methods with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974). 171 reflections with $|E| \ge 1·91$ were used in the phase-determining procedure. An E map calculated with the set of signs with the highest figures of merit revealed peaks corresponding to 12 Cl and 6 C atoms. A subsequent electron density synthesis revealed the positions of the remaining atoms. Weighted isotropic and anisotropic block-diagonal least-squares refinement (Ahmed, Hall, Pippy & Huber, 1966) gave a final R of 0·047 for all observed reflections. The function minimized was $\sum w(||F_o| - |F_c||)^2$, where $w = (7·2 + |F_o| + 0·01|F_o|^2)^{-1/2}$ and the R index was defined as $\sum ||F_o| - |F_c||/\sum |F_o|$.

Table 2. *Fractional atomic coordinates $(\times 10^4)$ with standard deviations in parentheses*

|	x	y	z
C(1)	6599 (7)	7090 (6)	6806 (3)
C(2)	7557 (7)	6482 (7)	6361 (4)
C(3)	6702 (7)	4768 (7)	5956 (4)
C(4)	4848 (7)	3655 (6)	5989 (4)
C(5)	3845 (7)	4271 (7)	6389 (4)
C(6)	4724 (7)	5987 (7)	6795 (4)
C(7)	7539 (7)	8905 (6)	7266 (4)
C(8)	8683 (6)	9402 (6)	8022 (3)
C(9)	9178 (6)	8158 (6)	8482 (4)
C(10)	10866 (7)	8689 (7)	8887 (4)
C(11)	12299 (7)	10593 (7)	8980 (4)
C(12)	11508 (7)	11819 (7)	8818 (4)
C(13)	9809 (7)	11247 (6)	8404 (4)
Cl(14)	9847 (2)	7856 (2)	6325 (1)
Cl(15)	7902 (2)	4021 (2)	5420 (1)
Cl(16)	3777 (2)	1524 (2)	5513 (1)
Cl(17)	1555 (2)	2915 (2)	6416 (1)
Cl(18)	3525 (2)	6756 (2)	7320 (1)
Cl(19)	7107 (2)	10344 (2)	6727 (1)
Cl(20)	7540 (2)	5954 (2)	8486 (1)
Cl(21)	11535 (2)	7202 (2)	9292 (1)
Cl(22)	13947 (2)	10991 (2)	8249 (1)
Cl(23)	13504 (2)	11099 (2)	10082 (1)
Cl(24)	12943 (2)	14018 (2)	9156 (1)
Cl(25)	8925 (2)	12684 (2)	8343 (1)
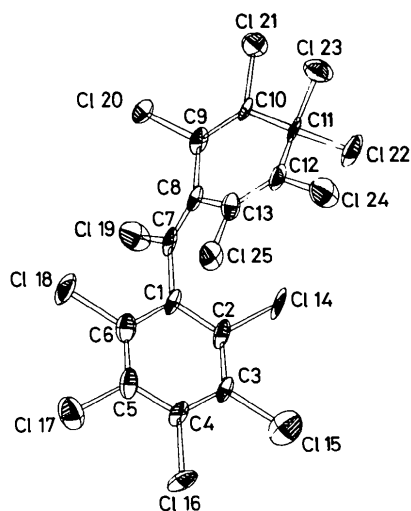
Fig. 1. A view of the molecule, drawn by the *ORTEP* program, showing thermal ellipsoids and the numbering of the atoms.

The scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The final difference map had no peaks greater than 0·42 e Å^{-3}.

Atomic coordinates are given in Table 2.* Fig. 1 shows a view of the molecule and the numbering of the atoms (*ORTEP*, Johnson, 1965).

Discussion. Figs. 2 and 3 show the bond distances and angles of the molecule. In the pentachlorophenyl group the mean values of the C—C and C—Cl bond distances and of the C—C—C and C—C—Cl bond angles (1·392, 1·717 Å and 119·9, 119·9° respectively) are similar to those found in related compounds, *e.g.* hexachlorobenzene (Brown & Strydom, 1974) and decachlorobiphenyl (Pedersen, 1975; Gali, 1975); the benzene ring is planar.

In the perchlorocyclohexa-2,5-diene group, all bond distances are comparable with the corresponding ones in hexachlorocyclohexa-2,5-dienone (Gali, Miravitlles & Font-Altaba, 1975). However, angles differ significantly, due to the strong interaction between Cl(19) and Cl(25) (Table 3). The angles Cl(19)—C(7)—C(8), C(7)—C(8)—C(13) and C(8)—C(13)—Cl(25) are increased to 124·2, 125·0 and 120·0° respectively. The ring shows a boat conformation, as can be inferred from the torsion angles (Table 4), due in part to *sp*^{3} hybridization of C(11) and also to the steric interaction of the Cl atoms (Table 3). Fig. 4 shows a perspective view of the unit cell.
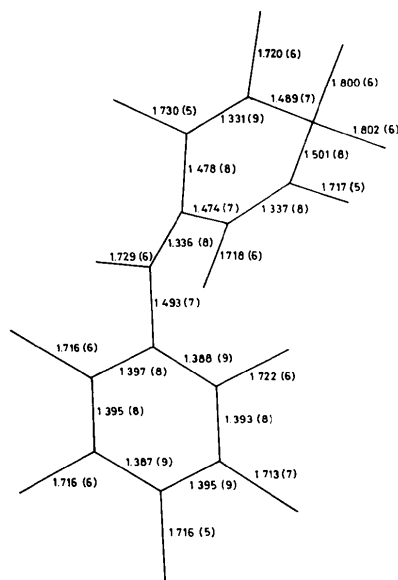
Fig. 2. Bond lengths (Å)
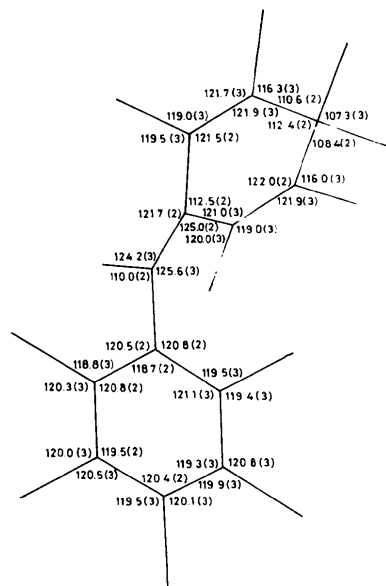
Fig. 3. Bond angles (°).

Table 3. Cl—Cl *intramolecular distances* (Å)

Cl(14)—Cl(15)	3·115 (2)	Cl(21)—Cl(22)	3·445 (2)
Cl(15)—Cl(16)	3·111 (3)	Cl(21)—Cl(23)	3·097 (2)
Cl(16)—Cl(17)	3·102 (3)	Cl(22)—Cl(23)	2·901 (3)
Cl(17)—Cl(18)	3·118 (2)	Cl(22)—Cl(24)	3·433 (3)
Cl(18)—Cl(19)	3·419 (2)	Cl(23)—Cl(24)	3·093 (2)
Cl(19)—Cl(25)	3·059 (2)	Cl(24)—Cl(25)	3·088 (2)
Cl(20)—Cl(21)	3·081 (2)		

Table 4. *Selected torsion angles* (°)

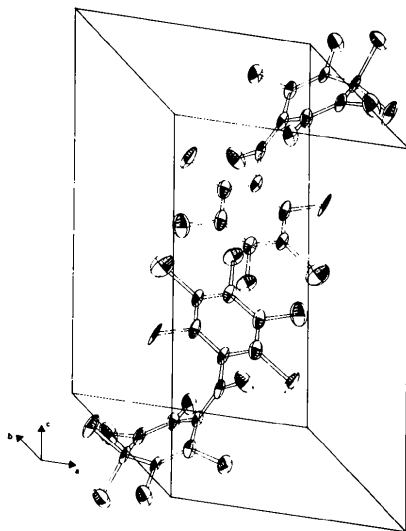|	
C(2)–C(1)–C(7)–C(8)	74·1 (8)
C(1)–C(7)–C(8)–C(9)	−1·5 (9)
C(7)–C(8)–C(9)–C(10)	−141·9 (6)
C(7)–C(8)–C(13)–C(12)	142·3 (6)
C(8)–C(9)–C(10)–C(11)	−6·4 (9)
C(9)–C(10)–C(11)–C(12)	−16·7 (8)
C(10)–C(11)–C(12)–C(13)	17·3 (8)
C(11)–C(12)–C(13)–C(8)	5·2 (9)

Fig. 4. A perspective view of the unit cell.

providing the crystals, and to Enraf–Nonius, who kindly supplied us with a CAD-4 automatic diffractometer.

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Choline *O*-Sulphate*

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Abstract. $C_5H_{13}NO_4S$, $M_r = 183·22$, monoclinic, $P2_1/c$, $a = 8·391$ (2), $b = 8·674$ (2), $c = 11·317$ (2) Å, $\beta = 97·29$ (1)°, $D_m = 1·50$, $D_c = 1·49$ g cm^{-3}, $Z = 4$, $R = 0·034$ on 1240 observed data. The molecule exists as a zwitterion with a *gauche* conformation in the choline chain; electrostatic stability is achieved through intra- and intermolecular interactions.

Introduction. $(CH_3)_3NCH_2CH_2OSO_3$ was prepared by the method of Stevens & Vohra (1955), and good air-stable crystals were grown by vapour-diffusion methods in an ethanol/water system. Preliminary photographic work showed that the cell dimensions and space group were in agreement with those previously reported (Okaya, 1966); the presence of pseudo-symmetry was indicated with the majority of the *l*-odd reflections weak in intensity.

A crystal (0·48 × 0·36 × 0·10 mm) was mounted for cell refinement and data collection with **b** parallel to

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