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Perchloro-*p*-xylylene

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Abstract. C_8Cl_8, triclinic, $P\bar{1}$, $a=10{\cdot}254$, $b=14{\cdot}912$, $c=8{\cdot}742$ Å, $\alpha=99{\cdot}40$, $\beta=96{\cdot}39$, $\gamma=99{\cdot}24°$, $V=1288{\cdot}613$ Å^3, $Z=4$, $D_x=1{\cdot}96$ g cm^{-3}, λ(Mo $K\alpha$)$=0{\cdot}7093$ Å. Final residual $R=0{\cdot}075$ for 2445 observed reflexions. The molecule is very distorted owing to steric interactions between methylene Cl atoms and the Cl atoms of the ring.

Introduction. C_8Cl_8 was synthesized by Ballester & Castañer (1960) during their research programme on perchlorinated organic compounds. Crystallographic study of C_8Cl_8 is especially interesting with regard to its conformation, and was initiated along with structural studies of similar compounds.

* This paper reports part of the research work undertaken to satisfy the requirements for a D. Sc. degree.

Crystals were obtained from hexane solution as light-yellow prisms elongated along **c**. The dimensions of the selected crystal were $0{\cdot}2\times0{\cdot}2\times0{\cdot}3$ mm. The unit-cell dimensions and space group were obtained from Weissenberg photographs and morphological study. Intensities were measured on a Philips single-crystal diffractometer with graphite-monochromated Mo $K\alpha$ radiation. 3650 independent reflexions were measured, of which 2649 had intensities greater than $3\sigma(I)$.

The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971). The normalization of F_o, symbolic addition procedure and Fourier *E* map were carried out by the programs of Declercq, Germain, Main & Woolfson (1973). An *E* map calculated with the set of signs with highest figures of merit revealed peaks for 31 atoms. A sub-

Table 1. *Final structure parameters*

Standard deviations are in parentheses. All values have been multiplied by 10^4. The temperature factor is in the form: $\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)]$.

	x	*y*	*z*	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
C(1)*A*	2653 (7)	3521 (5)	−54 (10)	70 (7)	35 (4)	115 (16)	26 (11)	3 (15)	27 (8)
C(2)*A*	2438 (7)	3571 (5)	−1744 (10)	63 (7)	44 (4)	124 (17)	17 (11)	−11 (16)	20 (8)
C(3)*A*	2179 (8)	4345 (6)	−2169 (11)	83 (8)	50 (4)	111 (18)	58 (13)	26 (18)	15 (9)
C(4)*A*	2096 (7)	5128 (5)	−927 (10)	86 (8)	42 (4)	86 (16)	20 (11)	−7 (16)	19 (8)
C(5)*A*	3110 (7)	5193 (5)	432 (10)	74 (7)	41 (4)	136 (17)	28 (12)	44 (16)	14 (8)
C(6)*A*	3353 (7)	4414 (5)	858 (10)	76 (8)	60 (4)	95 (16)	45 (12)	12 (16)	41 (9)
C(7)*A*	2145 (8)	2796 (6)	528 (11)	100 (9)	44 (4)	159 (19)	45 (13)	21 (19)	54 (10)
C(8)*A*	1154 (8)	5647 (5)	−1007 (12)	97 (10)	45 (4)	179 (20)	41 (14)	12 (20)	32 (10)
Cl(9)*A*	2748 (2)	2675 (1)	−3123 (3)	108 (2)	52 (1)	164 (5)	12 (3)	91 (5)	45 (3)
Cl(10)*A*	2160 (2)	4501 (2)	−4103 (3)	105 (2)	63 (1)	139 (5)	43 (4)	−4 (5)	24 (3)
Cl(11)*A*	462 (2)	5503 (2)	−2545 (3)	110 (2)	70 (1)	210 (6)	50 (4)	−1 (6)	21 (3)
Cl(12)*A*	1027 (2)	6489 (1)	505 (3)	105 (2)	52 (1)	204 (6)	37 (4)	53 (5)	53 (2)
Cl(13)*A*	4030 (2)	6256 (1)	1308 (3)	88 (2)	50 (1)	165 (5)	2 (3)	−8 (5)	12 (2)
Cl(14)*A*	4587 (2)	4443 (2)	2379 (3)	102 (2)	75 (1)	154 (5)	51 (4)	−22 (5)	43 (3)
Cl(15)*A*	2305 (3)	2751 (2)	2469 (4)	163 (3)	74 (2)	178 (6)	94 (4)	70 (7)	69 (4)
Cl(16)*A*	1229 (2)	1806 (1)	−594 (3)	120 (3)	47 (1)	217 (6)	42 (4)	35 (6)	21 (3)
C(1)*B*	3071 (9)	8292 (5)	4217 (10)	146 (10)	39 (4)	97 (18)	24 (12)	33 (20)	30 (10)
C(2)*B*	4429 (9)	8637 (5)	4059 (10)	117 (11)	36 (4)	108 (18)	8 (12)	30 (20)	18 (10)
C(3)*B*	4583 (8)	8956 (5)	2759 (11)	91 (8)	44 (4)	142 (19)	19 (13)	38 (18)	31 (9)
C(4)*B*	3502 (9)	8936 (5)	1588 (11)	146 (11)	30 (4)	151 (20)	24 (12)	48 (21)	−15 (10)
C(5)*B*	2378 (9)	9154 (6)	2296 (12)	132 (11)	49 (4)	57 (21)	60 (14)	−16 (22)	29 (11)
C(6)*B*	2108 (8)	8836 (6)	3546 (12)	82 (9)	55 (5)	178 (21)	23 (15)	45 (20)	37 (10)
C(7)*B*	2636 (10)	7502 (7)	4714 (12)	174 (13)	63 (6)	144 (22)	13 (17)	42 (26)	32 (14)
C(8)*B*	3519 (11)	8647 (7)	90 (13)	154 (15)	65 (6)	194 (24)	75 (18)	−5 (27)	7 (15)
Cl(9)*B*	5676 (2)	8743 (2)	5578 (3)	130 (3)	56 (1)	150 (5)	29 (4)	12 (6)	51 (3)
Cl(10)*B*	6223 (3)	9533 (2)	2561 (3)	124 (3)	61 (1)	236 (6)	79 (4)	79 (7)	37 (3)
Cl(11)*B*	4802 (5)	8298 (3)	−730 (4)	241 (6)	114 (3)	235 (8)	64 (7)	170 (10)	78 (6)
Cl(12)*B*	2133 (4)	8521 (2)	−1275 (4)	178 (4)	98 (2)	198 (7)	84 (6)	−34 (8)	−19 (5)
Cl(13)*B*	1501 (3)	9969 (2)	1635 (4)	148 (4)	65 (1)	289 (7)	92 (5)	−40 (8)	49 (4)
Cl(14)*B*	878 (3)	9161 (2)	4669 (4)	126 (3)	97 (2)	239 (7)	40 (6)	47 (7)	69 (4)
Cl(15)*B*	1012 (4)	7053 (2)	4744 (4)	162 (4)	84 (2)	245 (7)	61 (6)	50 (8)	−33 (4)
Cl(16)*B*	3722 (4)	6833 (2)	5365 (4)	235 (5)	61 (1)	217 (7)	75 (5)	8 (9)	57 (4)

sequent electron density synthesis revealed the positions of the remainder. Refinements were applied by the program NRC-10 of Ahmed, Hall, Pippy & Huber (1966). The function minimized was $\sum w(|F_o|-|F_c|)^2$, with the weights of Cruickshank (1965): $w=1/[a+|F_o|+c|F_o|^2]$, where $a=2F_{min}$, and $c=2/F_{max}$. After eight cycles of block-diagonal least-squares calculation with individual isotropic and four cycles with anisotropic thermal parameters, the refinement was terminated at $R=0{\cdot}075$ for 2445 reflexions, where R was defined as $\sum(||F_o|-|F_c||)/\sum|F_o|$. The final atomic and thermal parameters are listed in Table 1. The atomic scattering factors used were taken from *International Tables for X-ray Crystallography* (1962). Fig. 1 shows a view of the unit cell, drawn by the *ORTEP* program (Johnson, 1965), and the numbering of the atoms.*

Discussion. The two molecules in the asymmetric unit are distorted in the same way. They show a 'boat' form. Neither the alternative 'chair' nor the 'twisted-methylene-bond' (Ballester, Molinet & Rosa, 1961) forms have been observed in this compound. The molecules can be described by three mean planes, with C(2), C(3), C(5) and C(6) lying on least-squares mean-plane intersections (Table 3 and Fig. 2).

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31879 (13 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

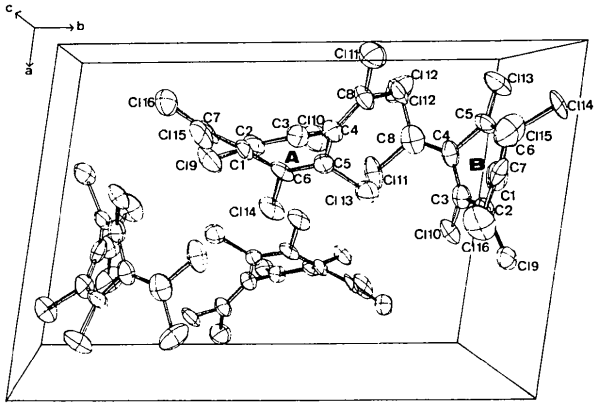

Fig. 1. Perspective view of the unit cell drawn by the *ORTEP* program, showing the thermal ellipsoids of the atoms.

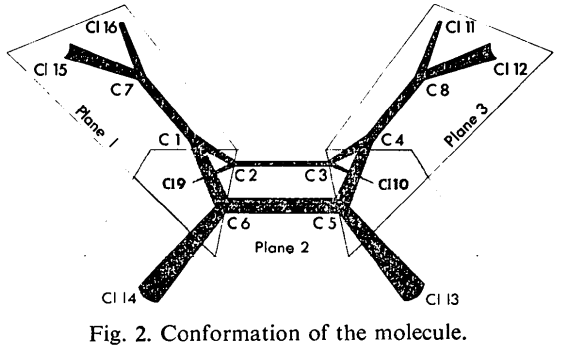

Fig. 2. Conformation of the molecule.

Table 2. *Intramolecular distances and angles of molecules A and B*

	Molecule *A*	Molecule *B*
C(1)—C(2)	1·485 (11) Å	1·434 (13) Å
C(2)—C(3)	1·326 (11)	1·338 (13)
C(3)—C(4)	1·479 (11)	1·489 (13)
C(4)—C(5)	1·468 (11)	1·424 (14)
C(5)—C(6)	1·327 (11)	1·300 (14)
C(6)—C(1)	1·464 (11)	1·503 (12)
C(1)—C(7)	1·325 (11)	1·347 (14)
C(4)—C(8)	1·334 (11)	1·313 (15)
C(2)—Cl(9)	1·742 (8)	1·706 (10)
C(3)—Cl(10)	1·742 (9)	1·713 (9)
C(5)—Cl(13)	1·717 (8)	1·756 (10)
C(6)—Cl(4)	1·720 (8)	1·762 (10)
C(7)—Cl(15)	1·700 (9)	1·696 (12)
C(7)—Cl(16)	1·703 (9)	1·720 (12)
C(8)—Cl(11)	1·690 (10)	1·681 (13)
C(8)—Cl(12)	1·700 (10)	1·715 (13)
Cl(9)—Cl(10)	3·112 (3)	3·129 (4)
Cl(10)–Cl(11)	3·225 (3)	3·204 (5)
Cl(11)–Cl(12)	2·837 (3)	2·807 (7)
Cl(12)–Cl(13)	3·168 (2)	3·252 (5)
Cl(13)–Cl(14)	3·116 (3)	3·174 (5)
Cl(14)–Cl(15)	3·171 (3)	3·178 (5)
Cl(15)–Cl(16)	2·826 (4)	2·863 (6)
Cl(16)–Cl(9)	3·169 (4)	3·172 (4)
	Molecule *A*	Molecule *B*
C(6)—C(1)–C(2)	110·1 (2)°	112·7 (3)°
C(6)—C(1)–C(7)	125·7 (2)	121·1 (2)
C(7)—C(1)–C(2)	123·6 (2)	125·5 (3)
C(1)—C(2)–C(3)	119·4 (3)	118·7 (4)
C(1)—C(2)–Cl(9)	119·5 (4)	120·5 (4)
Cl(9)—C(2)–C(3)	120·4 (3)	120·4 (5)
C(2)—C(3)–C(4)	118·2 (4)	116·1 (3)
C(2)—C(3)–Cl(10)	121·2 (5)	122·7 (4)
Cl(10)–C(3)–C(4)	119·9 (4)	120·2 (4)
C(3)—C(4)–C(5)	110·5 (2)	112·7 (3)
C(3)—C(4)–C(8)	125·5 (3)	120·5 (3)
C(8)—C(4)–C(5)	125·5 (3)	126·3 (3)
C(4)—C(5)–C(6)	118·2 (3)	119·6 (4)
C(4)—C(5)–Cl(13)	119·2 (4)	119·7 (5)
Cl(13)–C(5)–C(6)	122·2 (4)	120·0 (5)
C(5)—C(6)–C(1)	119·9 (4)	116·7 (4)
C(5)—C(6)–Cl(14)	120·3 (4)	124·2 (5)
Cl(14)–C(6)–C(1)	119·4 (4)	118·0 (4)
C(1)—C(7)–Cl(15)	124·2 (5)	125·1 (6)
C(1)—C(7)–Cl(16)	123·4 (5)	121·6 (6)
Cl(15)–C(7)–Cl(16)	112·2 (5)	113·2 (6)
C(4)—C(8)–Cl(11)	124·2 (5)	126·4 (7)
C(4)—C(8)–Cl(12)	122·0 (5)	121·9 (7)
Cl(11)–C(8)–Cl(12)	113·6 (5)	111·4 (6)

Intramolecular distances and angles of the molecules are given in Table 2. They are in agreement with the quinonoid electronic structure for C_8Cl_8 (Ballester & Castañer, 1960). C(1)–C(7), C(2)–C(3), C(4)–C(8) and C(5)–C(6) bond distances correspond to a double-bond distance, and angles C(6)–C(1)–C(2), C(3)–C(4)–C(5) are less than 120°, characteristic of a quinonoid structure. Selected torsion angles are given in Table 4. They show that molecular distortion of C_8Cl_8 does not affect

Table 3. *Least-squares mean planes and atom deviations* ($\times 10^3$ Å)

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

$$\begin{vmatrix} 10{\cdot}254 & -2{\cdot}394 & -0{\cdot}973 \\ 0 & 14{\cdot}718 & -1{\cdot}605 \\ 0 & 0 & 8{\cdot}538 \end{vmatrix} \begin{vmatrix} x \\ y \\ z \end{vmatrix} = \begin{vmatrix} X \\ Y \\ Z \end{vmatrix} .$$

Plane 1A $-0{\cdot}9367X+0{\cdot}3397Y+0{\cdot}0851Z-0{\cdot}0421=0$
Plane 1B $-0{\cdot}1167X+0{\cdot}3695Y+0{\cdot}9219Z-7{\cdot}4378=0$

	Molecule A	Molecule B
C(1)	−46 (7)	53 (9)
C(2)	11 (7)	−29 (9)
C(6)	28 (8)	−17 (10)
C(7)	−20 (9)	21 (11)
Cl(15)	7 (3)	−19 (4)
Cl(16)	20 (2)	−10 (3)

Plane 2A $-0{\cdot}9312X-0{\cdot}2227Y+0{\cdot}2886Z+3{\cdot}3490=0$
Plane 2B $0{\cdot}0381X+0{\cdot}8447Y+0{\cdot}5339Z-12{\cdot}1137=0$

	Molecule A	Molecule B
C(2)	−4 (7)	4 (9)
C(3)	4 (8)	−4 (9)
C(5)	−4 (8)	4 (9)
C(6)	4 (8)	−4 (10)
Cl(9)†	−720 (2)	664 (3)
Cl(10)†	−715 (2)	706 (3)
Cl(13)†	−667 (2)	766 (3)
Cl(14)†	−610 (2)	705 (3)

Plane 3A $-0{\cdot}5557X-0{\cdot}7309Y+0{\cdot}3961Z+6{\cdot}4645=0$
Plane 3B $-0{\cdot}1674X-0{\cdot}9806Y+0{\cdot}1022Z+12{\cdot}4421=0$

	Molecule A	Molecule B
C(3)	21 (8)	26 (8)
C(4)	−38 (8)	−49 (8)
C(5)	16 (8)	23 (9)
C(8)	−31 (9)	−38 (11)
Cl(11)	14 (3)	18 (4)
Cl(12)	18 (2)	21 (4)

Dihedral angles between planes

1A and 2A	34·8°	1B and 2B	36·9°
2A and 3A	37·4	2B and 3B	38·7

† Atom not included in least-squares calculation.

Table 4. *Selected torsion angles*

	Molecule A	Molecule B
C(7)—C(1)–C(2)–Cl(9)	−51·2°	−51·6°
Cl(9)—C(2)–C(3)–Cl(10)	0·9	−3·3
Cl(10)–C(3)–C(4)–C(8)	51·9	54·6
C(8)—C(4)–C(5)–Cl(13)	−49·7	−55·3
Cl(13)–C(5)–C(6)–Cl(4)	2·3	0·5
C(7)—C(1)–C(6)–Cl(14)	47·6	53·9

the double-bond torsion angles, which attain a maximum value of 3·3° in Cl(10)–C(3)–C(2)–Cl(9) of molecule B.

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