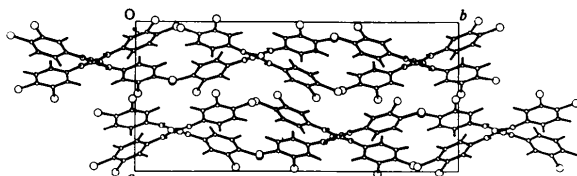


Table 4. Deviations (Å) of atoms from least-squares planes

Least-squares plane through the indicated atoms	Greatest deviations	Angle made with the urea plane (°)
Molecule A		
Urea N(1), N(2), O(1), C(25)	C(25) -0.001	
Phenyl I C(1)-C(6)	C(2) -0.013	16.32
Phenyl II C(7)-C(12)	C(7) 0.033	29.53
Molecule B		
Urea N(3), N(4), O(2), C(26)	C(26) -0.002	
Phenyl I C(13)-C(18)	C(15) -0.013	29.73
Phenyl II C(19)-C(24)	C(20) 0.021	39.67

N—C—O are larger, presumably as a result of the influence of the 3,4-dichlorophenyl substituents.

The authors are greatly indebted to Professors L.

Fig. 4. The arrangement of the molecules viewed along *c*.

Cavalca, M. Nardelli and B. Ribàr for their interest and to Dr A. Nikolić for the infrared spectra.

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Perchloro-*p*-xylene

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Abstract. C₈Cl₁₀, *M_r* = 450.62, *F*(000) = 1744, orthorhombic, *Pbca*, *a* = 16.436 (9), *b* = 18.600 (5), *c* = 9.792 (7) Å, *V* = 2993 (3) Å³, *Z* = 8, *D_c* = 1.999 g cm⁻³, λ(Mo *K*α) = 0.7107 Å, μ = 18.2 cm⁻¹. The structure was solved by direct methods and refined to *R* = 0.056 for 1749 observed reflections. Both the benzene ring and the trichloromethyl groups are distorted due to steric repulsions among the Cl atoms.

Introduction. C₈Cl₁₀ was synthesized by Ballester, Molinet & Castañer (1960), and was tentatively assigned a molecular conformation based on the UV-visible absorption spectrum (Ballester & Castañer, 1960).

These authors provided us with samples of perchloro-*p*-xylene in the form of pale greenish-yellow prismatic crystals obtained by slow evaporation of an ether solution.

A crystal of dimensions 0.2 × 0.2 × 0.4 mm was used for the data collection on a Nonius CAD-4 diffractometer. The cell dimensions were obtained by a least-squares refinement of the setting angles of 22 reflections. Mo *K*α radiation was used, mono-

chromatized by reflection from a graphite crystal, at a take-off angle of 5°. The ω–2θ technique was used with scan interval Δω = 0.70 + 0.35 tan θ. The background was measured before and after each reflection. The maximum scan time was 80 s, with 53.3 s used for the peak scanning and 13.3 s for each background. The intensity of 523 was chosen as a standard and it was measured every hour. Three reflections were selected for orientation control and measured every 100 reflections. 2723 independent reflections were collected within the range 1 < θ ≤ 30°, 1749 of which were considered as observed, by the criterion *I* > 2σ(*I*). The data were collected with the crystal mounted along the *c* axis. No absorption corrections were made (μ*r* < 0.5).

The structure was solved by the *MULTAN* system of computer programs (Main, Woolfson, Lessinger, Germain & Declercq, 1974), and refined by means of a block-diagonal least-squares procedure with a modified version of *SFLS* (Ahmed, Hall, Pippy & Huber, 1966), written by X. Solans. The function minimized was $w||F_o| - |F_c||^2$, where $w = (8.0 + |F_o| + 0.01|F_o|^2)^{-1/2}$. The refinement was terminated at *R* = 0.056 for all

observed reflections, where R is defined as $\sum ||F_o| - |F_c|| / \sum |F_o|$. * The scattering factors were taken from Forsyth & Wells (1959).

The final atomic parameters are listed in Table 1. Fig. 1 shows a view of the molecule drawn by the ORTEP program (Johnson, 1965) and the intramolecular distances. The molecular-geometrical calculations were performed with the CGMC computer program (Solans & Font-Altaba, 1975). The bond and torsion angles are shown in Figs. 2 and 3 respectively.

Discussion. The molecule of perchloro-*p*-xylene (PPX) has severe steric strain due to repulsion among the bulky Cl substituents, with short distances (mean value = 3.013 Å) between the geminal Cl atoms Cl(12) and Cl(13) [and Cl(16) and Cl(17)] and the ring Cl atoms Cl(10) and Cl(14) [and Cl(15) and Cl(9)] respectively. Almost identical distances between Cl atoms at analogous locations have been found in perchlorobenzylidene-2,5-cyclohexadiene (Galí, Solans, Miravittles & Plana, 1978), octachloronaphthalene (Gafner & Herbstein, 1963), perchloro-1,2,3-triphenyl-naphthalene (Galí, Solans, Miravittles, Font-Altaba & Armet, 1978), decachlorophenanthrene (Herbstein, Kapon & Merksamer, 1976), and decachloropyrene (Hazell & Jagner, 1976). These strains cause the C(1)–C(2)–Cl(9), C(4)–C(3)–Cl(10), C(4)–C(5)–Cl(14) and C(1)–C(6)–Cl(15) angles to be slightly increased (121.3°), while the C(2)–C(1)–C(6) and C(3)–C(4)–C(5) angles (115.0°) and the Cl(12)–C(8)–Cl(13) and Cl(16)–C(7)–Cl(17) angles (103.0°)

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33867 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	13781 (34)	62252 (28)	86438 (56)
C(2)	17832 (34)	68073 (32)	92748 (64)
C(3)	14209 (37)	74885 (34)	92923 (68)
C(4)	6613 (35)	76123 (31)	86580 (65)
C(5)	1858 (34)	69903 (30)	84569 (60)
C(6)	5466 (33)	63104 (28)	84255 (57)
C(7)	18204 (33)	55262 (30)	82770 (61)
C(8)	3387 (45)	83691 (32)	83524 (75)
Cl(9)	26544 (10)	66868 (10)	102081 (24)
Cl(10)	19031 (13)	81516 (10)	102217 (28)
Cl(11)	–1774 (12)	87190 (10)	97833 (19)
Cl(12)	–3070 (18)	83914 (11)	69125 (22)
Cl(13)	11324 (16)	89760 (11)	79008 (36)
Cl(14)	–8527 (9)	70410 (10)	83663 (23)
Cl(15)	–864 (8)	55759 (7)	82751 (19)
Cl(16)	14285 (10)	51179 (8)	67771 (18)
Cl(17)	28659 (9)	56732 (9)	78543 (21)
Cl(18)	17634 (12)	49125 (9)	96534 (20)

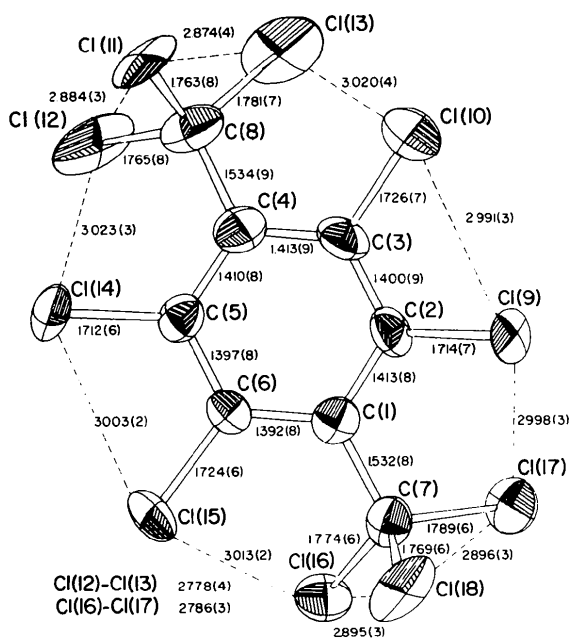


Fig. 1. A view of the molecule, drawn by the ORTEP program, showing thermal ellipsoids, the numbering of the atoms, the bond lengths (Å) and the most interesting intramolecular distances (Å).

are decreased relative to their normal values (120, 120 and 111° respectively), and the planarity of the *p*-xylene skeleton is lost (Fig. 4).

As the steric effects of Cl atoms in intramolecular interactions are similar to those of the methyl groups, the geometry of the trichloromethyl groups and the angles between the mean plane I (Fig. 4) and the bonds C(ar.)–Cl are similar to those obtained by Baenziger & Schultz (1973) in pentamethylbenzotrithloride.

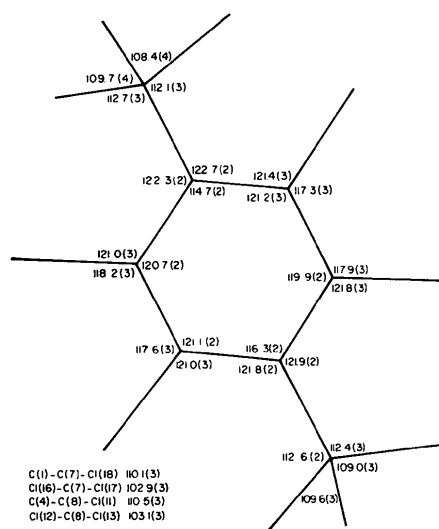


Fig. 2. Bond angles (°).

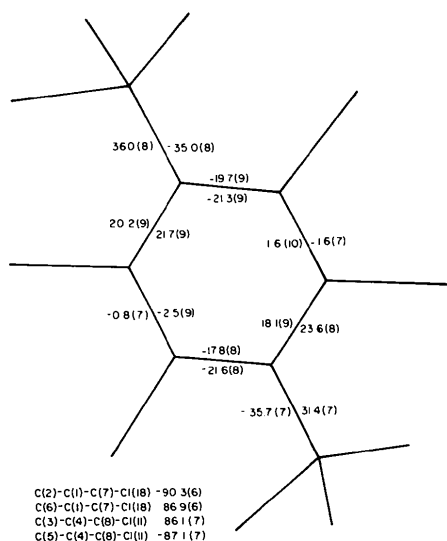


Fig. 3. Torsion angles ($^{\circ}$) less than 90° . A positive torsion angle $i-j-k-l$ ($j < k$) is defined such that when looking along $j-k$ a counter-clockwise rotation makes $i-j$ superimpose $k-l$.

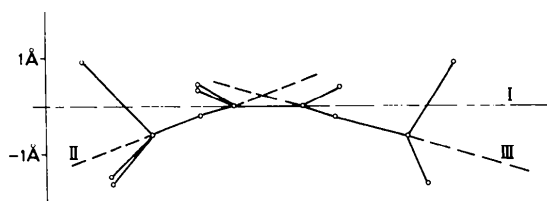


Fig. 4. Perchloro-*p*-xylene conformation and least-squares planes (vertical scale in Å). Plane I [C(2),C(3),C(5),C(6)], Plane II [C(1),C(2),C(6),C(7)] and Plane III [C(3),C(4),C(5),C(8)].

Slight differences between the PPX and the 2*H*,5*H*-octachloro-*p*-xylene (Silverman, Krukoniš & Yannoni, 1973) structures are due to the greater steric strain in our molecule.

The loss of planarity of the molecule was predicted by Ballester & Castañer from UV-visible absorption spectra. They suggested a 'chair-like' conformation. However we have found a 'boat-like' conformation which can be explained because the distortion of PPX is produced in such a way that the torsion angles Cl(9)-C(2)-C(3)-Cl(10), Cl(14)-C(5)-C(6)-Cl(15), C(1)-C(2)-C(3)-C(4) and C(4)-C(5)-

C(6)-C(1) in aromatic systems tend to be small (Fig. 3) as in perchloro-*p*-xylylene (Galí, Miravittles & Font-Altaba, 1976) and the mentioned molecular structures of octachloronaphthalene, perchloro-1,2,3-triphenylnaphthalene and perchlorobenzylidene-2,5-cyclohexadiene.

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