

Fig. 1. ORTEP (Johnson, 1965) drawing of the molecule. The numbering corresponds to that in Table 3.

Table 4. Atomic deviations (Å) from least-squares planes

E.s.d.'s are in parentheses.

Plane I		Plane II	
C(11)	-0.005 (3)	C(10a)	0.031 (2)
C(12)	0.018 (3)	C(3a)	0.068 (2)
O(1)	-0.005 (3)	C(4)	-0.288 (2)
O(2)	-0.006 (2)	C(4a)	0.279 (2)
		C(8a)	-0.151 (2)
Plane III		Plane IV	
C(4a)	0.008 (3)	S	-0.001 (1)
C(5)	-0.009 (3)	C(2)	0.004 (3)
C(6)	-0.004 (4)	C(3)	0.000 (3)
C(7)	0.015 (3)	C(3a)	-0.004 (3)
C(8)	-0.012 (3)	C(10a)	-0.005 (3)
C(8a)	-0.001 (3)		

Angles between planes (°) (e.s.d.'s ~0.03°)

I and II	30.4	II and III	39.5
I and III	36.1	II and IV	21.8
I and IV	51.8	III and IV	55.5

All the groups of atoms forming planes deviate significantly from planarity at the 95% level.

The structure comprises molecules hydrogen bonded around centres of symmetry forming dimers in a manner typical of carboxylic acids. The carboxyl group is *cis* to the thiophene ring. The least-squares planes for various portions of the molecule, Table 4, have been computed by PARST5 (Nardelli, Musatti, Domiano & Andreotti, 1965). The molecule is not planar, the torsion angle C(8a)—C(9)—C(10)—C(10a) being $-64.8 (3)^\circ$.

Most of the calculations were carried out with XRAY 70 (Stewart, Kundell & Baldwin, 1970). Thanks are due to the staff of the Computing Center of JEN (Madrid) for the facilities provided on the Univac 1100/80 computer.

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The Structure of 1,2,3,5-Tetrachlorobenzene

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Abstract. C₆H₂Cl₄, monoclinic, *P2₁/c*, *Z* = 8, *a* = 3.8530 (8), *b* = 24.010 (3), *c* = 17.226 (4) Å, β = 94.12 (2)°, *D_c* = 1.804 Mg m⁻³. The structure was solved by molecular-packing analysis and difference Fourier methods and refined to *R* = 0.047 for 2770 reflections.

Introduction. The crystal structure of the title compound was undertaken to provide a larger data base in connection with a study of Cl...Cl nonbonded interactions in crystals (Hsu & Williams, 1980). In the cited work, no evidence for nonbonded anisotropy above a threshold level was found in a study of five

perchlorohydrocarbons: hexachlorobenzene, octachloronaphthalene, octachloropentafulvalene, deca-chlorophenanthrene, and decachloropyrene. However, Wheeler & Colson (1976) and Munowitz, Wheeler & Colson (1977) claim that significantly anisotropic Cl...Cl interactions are present in one or more of the three crystal forms of *p*-dichlorobenzene.

Translucent crystals with good face development were grown with material obtained from the Aldrich Chemical Company; an approximately cubic crystal with an edge length of 0.2 mm was selected. X-ray diffraction data showed the crystal to be monoclinic, space group $P2_1/c$, which requires that there be two molecules in the asymmetric unit. 5127 independent reflections were collected with an Enraf-Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, using the θ - 2θ scan method. The intensities of three reference reflections remained constant throughout the data collection. Lorentz and polarization corrections were made in the usual manner and the data were reduced to observed structure factor amplitudes. No absorption correction was made; the absorption coefficient is 1.299 mm^{-1} and the transmission factors were estimated to range between 0.76 and 0.80.

A number of attempts were made to solve the phase problem by symbolic addition, using the computer programs *MULTAN* (Germain, Main & Woolfson, 1971) and *SHELX 76* (Sheldrick, 1976), without success. A molecular packing model was calculated with the computer program *PCK5* (Williams, 1969,

1979) and the resulting approximate Cl atom positions were used to calculate Fourier electron density difference maps. After several cycles of difference maps all atoms in the two molecules of the asymmetric unit were located.

After an initial refinement with *SHELX 76* in which the two molecules were treated as rigid, all nonhydrogen atoms were converted to anisotropic temperature factor format, with the H atoms isotropic. This final refinement adjusted 196 structural parameters, plus a scale factor, to minimize the *R* factor $\{R = [\sum w(F_o - F_c)^2 / \sum wF_o^2]^{1/2}\}$. The weights were defined as the squares of the reciprocals of the estimated standard deviations of the structure factors. The hydrogen scattering factors of Stewart, Davidson & Simpson (1965) were used. The final *R* factor was 0.047 for reflections significantly above background [2770 reflections with $F > 3\sigma(F)$]. The final electron density difference map showed no distinguishing features.*

Atomic parameters are given in Table 1.

Discussion. A thermal-ellipsoid plot (Johnson, 1965) of the asymmetric unit is shown in Fig. 1, with the atomic-numbering system and bond distances. The standard deviations are 0.004, 0.005, and 0.04 Å for C-Cl, C-C, and C-H bond distances. The average C-Cl and C-C distances are 1.729 and 1.378 Å respectively. All bond angles are within seven standard deviations of 120° . The largest deviation of the atoms from the best least-squares plane for the respective molecules is 0.011 Å.

Table 1. Final positional parameters and equivalent values for the temperature factors (Willis & Pryor, 1975)

	<i>x</i> ($\times 10^4$)	<i>y</i> ($\times 10^5$)	<i>z</i> ($\times 10^5$)	\bar{U} ($\text{\AA}^2 \times 10^3$)
C(1)	3080 (9)	3145 (15)	13682 (20)	4.6 (2)
C(2)	3352 (9)	5028 (13)	21268 (20)	4.0 (1)
C(3)	4749 (9)	1452 (15)	27016 (19)	4.3 (2)
C(4)	5855 (10)	-3798 (15)	25234 (23)	4.5 (2)
C(5)	5545 (10)	-5493 (15)	17609 (23)	4.8 (2)
C(6)	4158 (11)	-2108 (16)	11735 (24)	5.2 (2)
Cl(1)	1355 (3)	7476 (5)	6294 (6)	6.50 (5)
Cl(2)	1966 (3)	11597 (4)	23582 (6)	5.82 (5)
Cl(3)	5112 (4)	3531 (5)	36616 (6)	6.82 (6)
Cl(4)	6952 (3)	-12150 (4)	15282 (7)	6.88 (6)
H(1)	3855 (110)	-3417 (177)	6431 (255)	
H(2)	6454 (109)	-6260 (190)	29326 (235)	
C(7)	7328 (10)	23928 (15)	54655 (23)	4.9 (2)
C(8)	8163 (10)	18573 (16)	56995 (20)	4.4 (2)
C(9)	9312 (10)	14894 (14)	51499 (21)	4.4 (2)
C(10)	9643 (10)	16501 (16)	43953 (22)	4.8 (2)
C(11)	8795 (11)	21884 (16)	41845 (21)	5.2 (2)
C(12)	7634 (12)	25628 (17)	47115 (27)	5.5 (2)
Cl(5)	5852 (3)	28679 (5)	61211 (8)	7.38 (6)
Cl(6)	7804 (4)	16508 (5)	66450 (6)	7.35 (6)
Cl(7)	10449 (4)	8144 (4)	54107 (6)	6.80 (5)
Cl(8)	9195 (5)	24007 (5)	32362 (7)	8.50 (7)
H(3)	7142 (110)	29407 (196)	45924 (243)	
H(4)	10214 (116)	13925 (188)	40230 (247)	

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35543 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

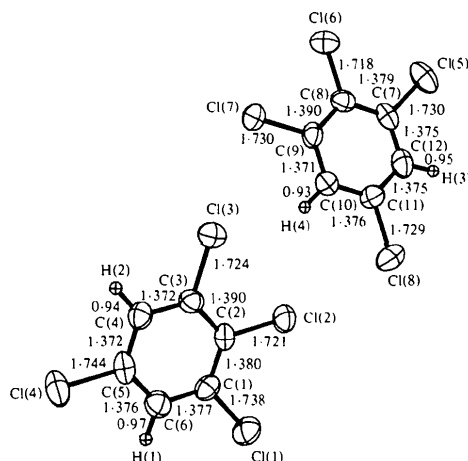


Fig. 1. A view of the asymmetric unit normal to the *bc* plane with *b* across the page. The thermal ellipsoids enclose 50% probability (Johnson, 1965). The atomic-numbering system and the bond distances (Å) are shown.

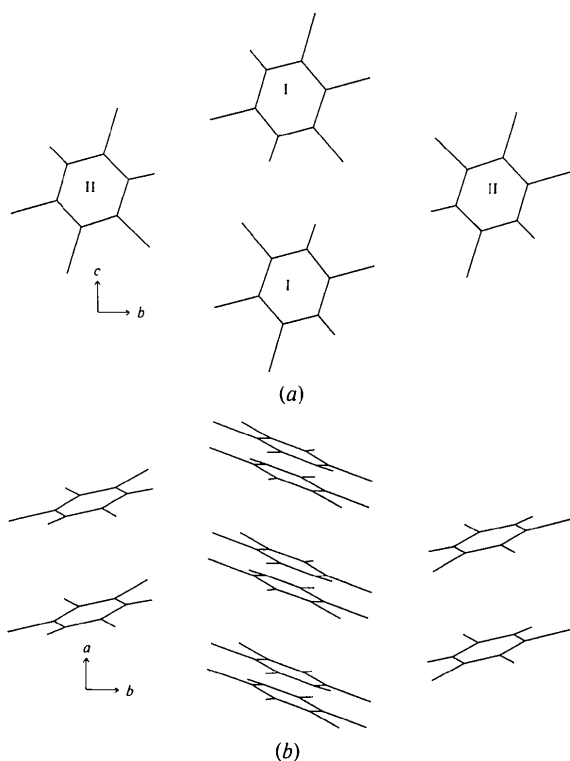


Fig. 2. The packing of the molecules around an inversion center. (a) A view along the short *a* lattice direction, looking down superimposed molecules arranged in stacks of type I or type II molecules. (b) A view along the *c* lattice direction showing the herringbone packing along *b*.

Intramolecular Cl...Cl repulsions produce minor distortions in the benzene ring. The bond angles C(1)–C(2)–C(3), C(3)–C(4)–C(5) and C(5)–C(6)–C(1) are less than 120° (118.1, 118.6, and 118.0°, e.s.d.'s 0.3°), while C(2)–C(3)–C(4), C(4)–C(5)–C(6) and C(6)–C(1)–C(2) are greater than 120° (121.3, 122.1, and 121.8°, e.s.d.'s 0.3°). Both molecules display similar trends.

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15-Oxosparteine Perchlorate Hemihydrate

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Abstract. C₁₅H₂₅N₂O⁺·ClO₄⁻·½H₂O, *P*22₁2₁, *a* = 9.071 (2), *b* = 11.096 (2), *c* = 16.793 (2) Å, *Z* = 4, *D_x* = 1.44 Mg m⁻³. The final *R* = 0.052, *R_w* = 0.062

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Fig. 2 illustrates the molecular packing. Type I and type II molecules stack in separate columns along the short-lattice-constant direction. The molecules, when viewed along *c*, show a herringbone type of packing. Along *b* the stacks are staggered. The intermolecular distances are in the normally expected range for nonbonded contacts; the shortest intermolecular distance is an H...Cl contact, at 3.025 Å. The shortest intermolecular Cl...Cl distance is 3.563 Å.

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for 1217 reflections. Two types of hydrogen bonds were observed: O(C15)...O(*W*) and N(1)...O(C1) of 2.854 (5) and 2.912 (6) Å respectively. The piperidine

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