bond lengths shows a good fit to a straight line (correlation 0·989, intercept 10^{-3}) with a slope of 0·88, indicating that the parameter standard deviations are satisfactorily estimated. Compared with the bond lengths from the room-temperature X-ray structure study (Gieren & Dederer, 1977) the distances in the neutron study at 120 K are larger than the corresponding X-ray values, with one exception. The S—O bond is 0·007 (8) Å shorter. This could be explained by the presence of O lone-pair electrons. A similar effect, where the O shifts more into its lone-pair region than into a C—O bond has been reported by Coppens & Vos (1971). The largest differences compared to the X-ray study occur in the X—H distances, which are on average 0·13 (2) Å longer in the present study. This is the usual asphericity shift of the H atom in the conventional X-ray refinement (Coppens, 1974). The N—H distance [1·018 (8) Å] is 0·15 (4) Å longer than the corresponding X-ray value and seems to be typical for the case of an N—H···O hydrogen bond, where the N is sp^2 hybridized (Lehmann, Verbist, Hamilton & Koetzle, 1973). Compared to other hydrogen bonds of the same type (reviewed by Olovsson & Jönsson, 1976) the H···O distances of the present work indicate medium to strong intermolecular hydrogen bonding, and a weak intramolecular bond.

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Structure of Trimethyl 4,5,6-Trichloro-1,2,3-benzenetricarboxylate, $C_{12}H_9Cl_3O_6$

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Abstract. $M_r = 355·56$, monoclinic, $P2_1/c$, $a = 13·649$ (6), $b = 5·819$ (2), $c = 19·667$ (8) Å, $\beta = 109·09$ (2)°, $V = 1476$ (1) Å^3, $Z = 4$, $D_x = 1·600$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5·978$ mm^{-1}, $F(000) = 720$, room temperature. Final $R = 0·059$ for 2103 observed reflections. The molecular structure is strongly influenced by the three vicinal methoxycarbonyl groups as well by three vicinal Cl atoms attached to the benzene ring. The central ester group is twisted 34·9 (1)° out of the best plane through the benzene ring. The other two groups are twisted 68·7 (1) and 66·6 (1)° out of this plane. The average

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C—Cl bond distance is 1·719 (4) Å and the intramolecular Cl···Cl contacts are 3·099 (2) and 3·098 (2) Å.

Introduction. The attempt to obtain dimethyl 1,4,5,6-tetrachloro-7,7-dimethoxybicyclo[2.2.1]hepta-2,5-diene-2,3-dicarboxylate by cycloaddition of tetrachlorodimethoxycyclopentadiene and dimethyl 2-butynedioate was unsuccessful. The product of the reaction was trimethyl 4,5,6-trichloro-1,2,3-benzenetricarboxylate (trimethyl 4,5,6-trichlorohemimellitate) already described in the literature (Diekmann, 1963; Lemal, Gosselink & McGregor, 1966). The X-ray analysis of this compound was carried out to obtain the accurate data for this type of molecule. The conformational and intramolecular effects of three vicinal ester groups and the other three vicinal Cl atoms on the benzene ring are of particular interest in the sense of molecular distortion due to overcrowding.

Experimental. White crystals of title compound grown from methylene dichloride. Intensity data collected from a crystal of dimensions 0·05 × 0·19 × 0·28 mm on a Philips PW 1100 four-circle diffractometer, θ–2θ scanning technique, scan range 1·20°, scan rate 0·04° s^{-1}. Unit-cell parameters obtained from least-squares analysis of 20 reflections with 2θ values ranging from 30 to 60°. Absent reflections $h0l$, $l \neq 2n$ and $0k0$, $k \neq 2n$ confirmed $P2_1/c$ space group. Out of 2236 reflections scanned within a quadrant $\pm h, k, l (h$ −16–15, k 0–6, l 0–23) up to $\sin\theta/\lambda = 0{\cdot}610$ Å^{-1}, 2103 unique reflections had $I > 3\sigma(I)$ and were used in crystal-structure analysis. Three standard reflections (311, $\bar{4}02$, 014) measured every 2 h showed an unsystematic variation with average 1·4 (4)%. Corrections applied for Lorentz and polarization effects. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Of 21 non-H atoms 19 obtained from E map calculated with 284 largest E values ($E \geq 1{\cdot}44$). Subsequent calculations performed mainly with XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Remaining non-H atoms as well as all H atoms located from difference maps. Block-diagonal least-squares refinement with anisotropic thermal parameters for non-H and isotropic for H atoms (U_H kept fixed in last cycle of refinement) gave R and R_w of 0·059 and 0·071 respectively; function minimized $\sum w(|F_o|-|F_c|)^2$ with $w = 0{\cdot}174/\sigma^2(F_o)$ giving $S =$ 1·00. Max. and mean $\Delta/\sigma \leq 0{\cdot}3$ and 0·1 respectively. A final difference map revealed no residuals greater than 0·35 e Å^{-3}, except six irregular maxima up to 0·80 e Å^{-3} near Cl atoms. Scattering factors of Cromer & Mann (1968) used for non-H atoms, those of Stewart, Davidson & Simpson (1965) for H. Anomalous-dispersion factors taken from *International Tables for X-ray Crystallography* (1974). All calculations performed on a Univac 1110 computer at the University Computing Centre of Zagreb.

Discussion. The final atomic coordinates are listed in Table 1.* Fig. 1 shows a perspective view of the molecule with the atomic numbering. Intramolecular bond lengths and angles are listed in Table 2. Deviations, from the best plane through the benzene ring, of six atoms bonded to the ring are given in Fig. 1.

The mean C—Cl bond distance of 1·719 (4) Å is, as expected, nearer to 1·709 than to 1·737 Å, the average values estimated by Rudman (1971) for aromatic compounds containing *ortho* and isolated C—Cl bonds respectively. The intramolecular Cl···Cl distances of 3·099 (2) and 3·098 (2) Å are somewhat smaller than the average non-bonded distance of 3·14 Å for *ortho* Cl atoms also estimated by Rudman (1971).

Neither exocyclic nor endocyclic angles of the benzene ring are significantly different from 120°. On the other hand, the atoms directly bonded to the ring deviate significantly from the benzene mean plane (Fig. 1) indicating a strong intramolecular overcrowding. The benzene ring is planar within ±0·012 (4) Å. None of the methoxycarbonyl groups is coplanar with the benzene ring. The twist angle τ about the C(ring)–C(methoxycarbonyl) bonds is similar for the two lateral groups and different for the central group: τ_1[C(1)–C(11)] = 68·7 (1), τ_3[C(3)–C(31)] = 66·6 (1) and τ_2[C(2)–C(21)] = 34·9 (1)°. Reported values for hemimellitic acid (Mo & Adman, 1975) are just the opposite: the central group is twisted (87·2°) much more than the two lateral groups (9·6 and 4·1°). It is obvious that the twisting angles are easily varied to accommodate intramolecular strain.

In general the analogous bond lengths and angles of the ester groups are similar but there are also some differences. The C—C=O angles of the lateral ester groups are larger [125·2 (4) and 126·4 (3)°] than that of the central group [123·2 (3)°]. These differences are still greater for the C—C—O angles: 109·3 (3) and 108·6 (3)° for the lateral groups and 112·1 (4)° for the central ester group. The last angle is in the range of 112–114°, quoted as typical in unstrained esters (Byrn, Curtin & Paul, 1972).

The carbonyl atoms O(11) and O(31) from the lateral ester groups have intramolecular O···Cl contacts of 3·205 (3) and 3·173 (4) Å respectively, which are close to the sum of the van der Waals radii (3·20 Å). There is an intermolecular O(11)···Cl(1) contact of 3·101 (4) Å, which may participate in the

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* Lists of structure factors, anisotropic thermal parameters, mean planes, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38955 (22 pp.). Copies may be obtained through The Executive Secrtary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

stabilization of the three-dimensional network of the structure. The packing of the molecules is illustrated in Fig. 2.

The C—H bond lengths fall within the range 0·83 (6) to 1·13 (9) Å.

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Table 1. *Fractional positional parameters* (×10⁴) *and equivalent isotropic temperature factors* (×10²) *of non*-H *atoms*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$(Å²)
Cl(1)	2822 (1)	1570 (2)	8253 (1)	5·13 (3)
Cl(2)	4227 (1)	4959 (2)	7778 (1)	5·68 (4)
Cl(3)	4379 (1)	4662 (2)	6244 (1)	5·61 (4)
O(11)	2787 (3)	2544 (6)	4800 (2)	6·4 (1)
O(12)	3539 (3)	−850 (6)	5202 (2)	6·5 (1)
O(21)	1509 (3)	−3880 (5)	5906 (2)	6·3 (1)
O(22)	1333 (3)	−1285 (6)	5034 (2)	7·2 (1)
O(31)	1716 (3)	−3067 (7)	7562 (2)	7·9 (2)
O(32)	541 (2)	−957 (5)	6736 (2)	5·4 (1)
C(1)	2962 (3)	1266 (6)	5992 (2)	3·6 (1)
C(2)	2309 (3)	−214 (6)	6207 (2)	3·7 (1)
C(3)	2254 (3)	−109 (6)	6896 (2)	3·7 (1)
C(4)	2864 (3)	1460 (7)	7389 (2)	3·6 (1)
C(5)	3500 (3)	2969 (7)	7181 (2)	3·7 (1)
C(6)	3554 (3)	2853 (6)	6488 (2)	3·6 (1)
C(11)	3068 (3)	1114 (7)	5257 (2)	4·4 (2)
C(12)	3637 (6)	−1314 (12)	4499 (3)	10·0 (3)
C(21)	1676 (3)	−2005 (7)	5707 (2)	4·6 (1)
C(22)	791 (5)	−2999 (10)	4500 (3)	9·9 (3)
C(31)	1500 (3)	−1597 (7)	7113 (2)	4·5 (1)
C(32)	−301 (4)	−2324 (11)	6829 (3)	7·7 (2)

Table 2. *Bond lengths* (Å) *and angles* (°) *involving non*-H *atoms*

C(1)–C(2)	1·400 (6)	C(21)–O(21)	1·205 (5)
C(1)–C(6)	1·394 (5)	C(21)–O(22)	1·321 (5)
C(2)–C(3)	1·383 (6)	C(22)–O(22)	1·462 (6)
C(3)–C(4)	1·392 (5)	C(3)–C(31)	1·510 (6)
C(4)–C(5)	1·387 (6)	C(31)–O(31)	1·194 (6)
C(5)–C(6)	1·390 (6)	C(31)–O(32)	1·330 (4)
C(1)–C(11)	1·502 (6)	C(32)–O(32)	1·457 (7)
C(11)–O(11)	1·193 (5)	C(4)–Cl(1)	1·720 (4)
C(11)–O(12)	1·333 (6)	C(5)–Cl(2)	1·716 (4)
C(12–O(12)	1·456 (8)	C(6)–Cl (3)	1·721 (4)
C(2)–C(21)	1·498 (5)		

C(2)–C(1)–C(6)	118·5 (3)	C(5)–C(6)–C(1)	120·9 (4)
C(2)–C(1)–C(11)	121·7 (3)	C(5)–C(6)–Cl(3)	119·6 (3)
C(6)–C(1)–C(11)	119·8 (4)	C(1)–C(6)–Cl(3)	119·5 (3)
C(1)–C(2)–C(3)	120·8 (3)	C(1)–C(11)–O(11)	125·2 (4)
C(1)–C(2)–C(21)	121·2 (4)	C(1)–C(11)–O(12)	109·3 (3)
C(3)–C(2)–C(21)	118·0 (4)	O(11)–C(11)–O(12)	125·5 (4)
C(2)–C(3)–C(4)	120·0 (4)	C(2)–C(21)–O(21)	123·2 (3)
C(2)–C(3)–C(31)	120·8 (3)	C(2)–C(21)–O(22)	112·1 (4)
C(4)–C(3)–C(31)	119·1 (4)	O(21)–C(21)–O(22)	124·8 (4)
C(3)–C(4)–C(5)	119·9 (4)	C(3)–C(31)–O(31)	126·4 (3)
C(3)–C(4)–Cl(1)	120·5 (3)	C(3)–C(31)–O(32)	108·6 (3)
C(5)–C(4)–Cl(1)	119·6 (3)	O(31)–C(31)–O(32)	125·0 (4)
C(4)–C(5)–C(6)	119·8 (3)	C(11)–O(12)–C(12)	115·6 (4)
C(4)–C(5)–Cl(2)	120·2 (3)	C(21)–O(22)–C(22)	115·7 (4)
C(6)–C(5)–Cl(2)	120·0 (3)	C(31)–O(32)–C(32)	116·7 (4)

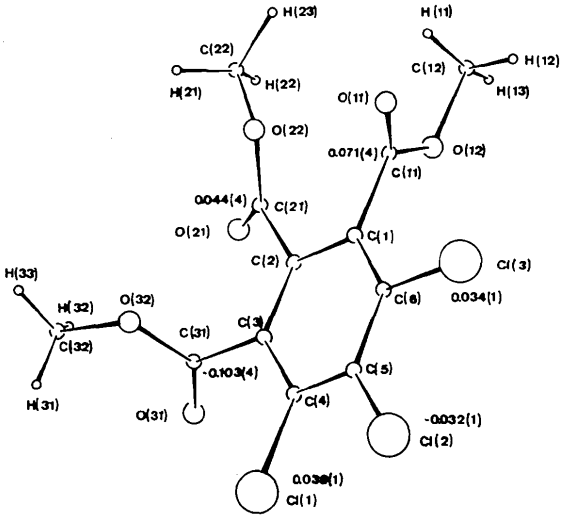

Fig. 1. Perspective drawing of the molecule viewed along [010]. Displacements (Å) of the atoms directly bonded to the ring from its mean plane are denoted.

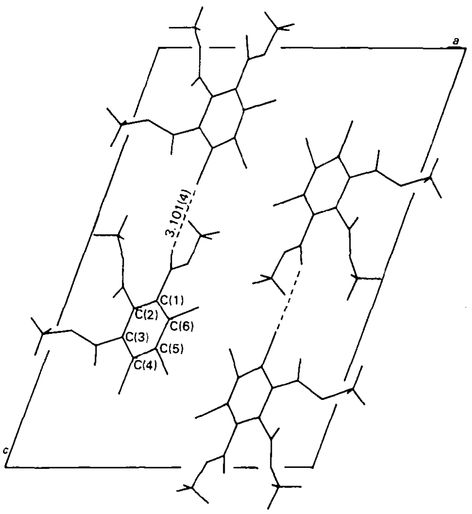

Fig. 2. Molecular packing along [010]. The intermolecular O(11)···Cl(1) contact (Å) is shown.

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Low-Temperature Structure of 1,2,3,4,5,6,7,8-Octahydroanthracene, $C_{14}H_{18}$

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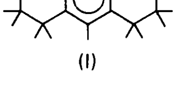
Abstract. $M_r = 186.30$, monoclinic, $P2_1/c$, $a = 9.354\,(3)$, $b = 6.550\,(2)$, $c = 9.622\,(3)$ Å, $\beta = 116.99\,(3)°$, $V = 525.3$ Å^3, $Z = 2$, $D_x = 1.18$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0.71069$ Å, $\mu = 0.071$ mm^{-1}, $F(000) = 204$, $T \simeq 110$ K. Final $R = 0.038$ for 1593 observed data. There is a good correspondence between the experimentally determined values of the bond lengths, bond angles and torsion angles and the estimates of these values from molecular-mechanics calculations on the achiral conformation.

Introduction. Empirical force-field calculations on 1,2,3,4,5,6,7,8-octahydroanthracene (I) reveal the existence of two equally stable conformations. Depending on the mutual orientation of the half-chair forms of the outer cyclohexene rings the point group is $2/m$ (achiral) or 222 (chiral) (Baas, unpublished results). The present paper describes the low-temperature form (the α form) of the title compound (I). It is designated as the α form because at 318K a martensite-type transformation to a β form is observed (Vatulev & Prikhot'ko, 1965).



(I)

Experimental. (I) obtained from Dr D. Tavernier (University of Gent, Belgium), recrystallized from acetone at 278 K. Crystal of approximate dimensions 0·3 × 0·4 × 0·4 mm enclosed in a thin-walled capillary and cooled down by a cold N_2 gas stream to 110 (2) K. Systematic absences, $h0l$ for l odd and $0k0$ for k odd, indicated space group $P2_1/c$. Cell parameters obtained from diffractometer angular settings of 25 centered reflections. Data collected for $hk \pm l$ ($h_{\max} = 13$, $k_{\max} = 9$, $l \pm 14$) with $\theta_{\max} = 32.00°$ on a Nonius CAD-4 diffractometer (graphite-monochromated Mo $K\alpha$ radiation). These gave 1817 independent data of which 1593 reflections with $I > \sigma(I)$ used in subsequent calculations. Three reflections measured every 2 h of X-ray exposure time, intensity variation within 3%. Extinction and absorption neglected. Structure solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1971); H atoms located in a difference map. Refinement (x, y, z of all atoms, anisotropic temperature factors for heavy atoms, isotropic for H) by full-matrix least squares on F (XRAY72, Stewart, Kruger, Ammon, Dickinson & Hall, 1972) with equal weights given to all reflections converged at $R = 0.038$. In final cycle shifts in parameters all $< 0.1\,\sigma$. Final difference map showed peaks of about 0·40 e Å^{-3} halfway between bonded C atoms. Molecular-mechanics calculations performed using *DELPHI* (van de Graaf, Baas & van Veen, 1980), empirical force field from Brückner, Allegra, Albinati & Ferina (1980).

Discussion. Positional and isotropic thermal parameters are given in Table 1.* The structure is shown in Fig. 1 with the atom numbering used here. Bond lengths, bond angles and torsion angles of the carbon skeleton are given in Fig. 2 together with the values obtained with the empirical force-field calculations. All observed

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38924 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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