

it is very much flattened with respect to an unsubstituted 1-3 diplanar cyclohexene, the mean distance of all 6 atoms to the mean plane being 0.11 Å.

The bond distances are in good agreement with those found in the structure of crotepoixide (Coggon, McPhail & Sim, 1969).

The epoxide ring is inclined at 103° to the C(5)-C(6)-C(1)-C(2) plane. The two acetyl groups are planar within experimental error, but twisted around their bond to the benzene ring; the dihedral angle H(2)-C(2)-O(17)-C(18) is -34° and H(3)-C(3)-O(20)-C(21) is +39°.

The benzene ring makes an angle of 22° with C(9)-C(10) and is inclined at 29° to the cyclohexene ring mean plane.

The closest intermolecular distances are 3.18 Å for O(8)···O(21) and 3.14 Å for C(9)···O(21). The other distances for intermolecular contacts correspond to normal van der Waals interactions.

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*Acta Cryst.* (1976). B32, 1591

## Tris-(*p*-methoxyphenyl)chloromethane

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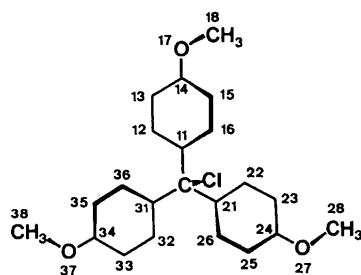
(Received 9 January 1976; accepted 31 January 1976)

**Abstract.** C<sub>22</sub>H<sub>21</sub>O<sub>3</sub>Cl, monoclinic, *P*2<sub>1</sub>/*c*; *a* = 14.912 (8), *b* = 10.303 (4), *c* = 12.489 (4) Å, β = 98.27 (6)°; *Z* = 4, *D<sub>m</sub>* = 1.300, *D<sub>x</sub>* = 1.304 g cm<sup>-3</sup>. The structure was refined to an *R* of 0.08. The molecule has the expected propeller geometry with the restriction that one methoxyl group is oriented contrariwise to the other two.

**Introduction.** The title compound (Schuchardt, Munich) forms colourless elongated prisms by slow evaporation of a benzene/petroleum ether solution in a flow of dry nitrogen. The lattice parameters and the intensities were measured at room temperature on an automatic four-circle Philips PW1100 diffractometer with graphite-monochromatized Mo *K*α radiation (λ = 0.7107 Å). With a crystal, 0.10 × 0.21 × 0.20 mm, sealed in a Lindemann capillary, 1338 independent reflexions were scanned in the θ-2θ mode (scan width 1.2° in 2θ; scan speed 0.02° s<sup>-1</sup>) out to a limit of sin θ/λ ≤ 0.55. The background was measured on each side of the peaks with a counting time of half the reflexion scan time. Three monitor reflexions were measured at intervals of 15 reflexions; no significant change in intensity was revealed. After the usual corrections, 862 reflexions with |*F<sub>o</sub>*| > 2σ(*F<sub>o</sub>*) were considered observed and used in the structure analysis. Absorption corrections

(μ = 2.28 cm<sup>-1</sup>) were not applied. The structure was solved by the heavy-atom method and refined by full-matrix least squares with anisotropic temperature factors for the non-hydrogen atoms. All the H atoms could be seen on a difference map, but some of the maxima were too diffuse for a precise coordinate determination. Fortunately at least one peak around each methyl group had a high enough density for a satisfactory coordinate assignment. This was sufficient to define the orientation of all the methyl groups, assuming tetrahedral symmetry for the C atoms. The calculated positions of the H atoms (Table 2) with C-H length 1.09 Å were included in the latter stages of the refinement, but were not refined. All the observed reflexions were included in each cycle; the final *R* was 0.08 with the weighting scheme: *w* = 0.053|*F<sub>o</sub>*| for |*F<sub>o</sub>*| ≤ 13.0, *w* = 1.0 for 13.0 < |*F<sub>o</sub>*| ≤ 64.0, and *w* = 75|*F<sub>o</sub>*|<sup>-1</sup> for |*F<sub>o</sub>*| > 64.0. The final atomic parameters are listed in Tables 1 and 2.\* The atom numbering is as follows (the first digit of the two digit numbers refers to the phenyl ring):

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31647 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.



**Discussion.** As part of our current interest in the structure and crystal packing of triphenylhalomethanes (Dunand & Gerdil, 1976), we have investigated the structure of triphenylchloromethane symmetrically substituted by electron-donating groups. Unlike the

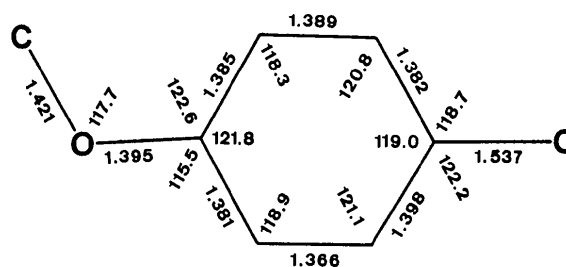


Fig. 1. Representative geometry of a *p*-methoxyphenyl group. Average values are taken over equivalent bonds and angles, assuming threefold symmetry for the three-ring system. Equivalence between the external C-C-O angles in the three aromatic rings is determined with respect to the orientation of the methoxy group. Mean values of the e.s.d.'s for bond distances (Å) and angles (°) are 0.016 and 1.0, respectively.

Table 1. *Final positional and thermal parameters (both  $\times 10^4$ ) with standard deviations in parentheses*

Thermal parameters are given in the form:  $\exp [-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)]$ .

	<i>x</i>	<i>y</i>	<i>z</i>	<i>b</i> <sub>11</sub>	<i>b</i> <sub>22</sub>	<i>b</i> <sub>33</sub>	<i>b</i> <sub>12</sub>	<i>b</i> <sub>13</sub>	<i>b</i> <sub>23</sub>
Cl	1132 (2)	686 (3)	894 (3)	69 (2)	67 (4)	109 (4)	20 (5)	-38 (4)	10 (6)
C	2046 (8)	-449 (11)	1473 (10)	43 (9)	77 (18)	57 (15)	16 (18)	-33 (16)	37 (21)
C(11)	1573 (8)	-1778 (12)	1577 (10)	41 (8)	76 (19)	47 (13)	-20 (18)	0 (14)	-15 (23)
C(12)	987 (10)	-2295 (12)	710 (11)	55 (10)	60 (18)	75 (16)	-15 (20)	-4 (16)	-14 (25)
C(13)	637 (10)	-3529 (14)	771 (11)	59 (11)	73 (17)	50 (15)	-48 (22)	-12 (18)	0 (23)
C(14)	848 (8)	-4229 (13)	1733 (10)	56 (9)	68 (17)	70 (13)	-31 (20)	27 (15)	-46 (24)
C(15)	1414 (8)	-3763 (10)	2610 (10)	60 (9)	9 (17)	66 (14)	-25 (15)	8 (16)	-8 (19)
C(16)	1772 (8)	-2511 (12)	2518 (10)	62 (9)	55 (18)	54 (13)	17 (20)	-8 (14)	-21 (21)
O(17)	485 (6)	-5474 (9)	1697 (7)	79 (7)	93 (13)	80 (10)	-63 (15)	27 (11)	-15 (16)
C(18)	697 (10)	-6244 (11)	2644 (12)	100 (11)	51 (18)	102 (17)	-49 (19)	55 (20)	36 (24)
C(21)	2435 (8)	81 (10)	2595 (9)	48 (9)	70 (14)	57 (12)	-15 (15)	-49 (13)	20 (19)
C(22)	1895 (10)	387 (12)	3378 (11)	56 (11)	61 (17)	134 (17)	3 (20)	113 (21)	-59 (26)
C(23)	2270 (10)	729 (12)	4400 (10)	82 (12)	87 (16)	54 (12)	-65 (23)	19 (17)	-74 (23)
C(24)	3201 (10)	752 (12)	4632 (12)	47 (11)	72 (15)	89 (19)	-51 (20)	-64 (20)	31 (25)
C(25)	3757 (10)	466 (13)	3878 (13)	46 (10)	121 (20)	101 (18)	10 (21)	76 (21)	25 (26)
C(26)	3362 (8)	144 (12)	2834 (10)	94 (10)	56 (18)	34 (12)	-43 (21)	-4 (14)	22 (20)
O(27)	3623 (7)	1038 (9)	5688 (9)	83 (7)	177 (14)	52 (11)	-85 (16)	17 (14)	-19 (17)
C(28)	3087 (11)	1076 (13)	6536 (12)	123 (14)	139 (22)	56 (17)	-70 (24)	41 (24)	11 (26)
C(31)	2699 (8)	-531 (13)	646 (10)	45 (8)	97 (20)	42 (12)	6 (21)	11 (15)	-34 (24)
C(32)	3037 (10)	570 (12)	173 (11)	85 (11)	67 (18)	65 (15)	-88 (24)	14 (18)	35 (25)
C(33)	3647 (11)	466 (15)	-526 (12)	85 (12)	110 (23)	75 (17)	-99 (25)	49 (20)	-51 (29)
C(34)	3951 (10)	-729 (16)	-789 (12)	54 (10)	157 (24)	50 (14)	-27 (25)	3 (17)	-68 (29)
C(35)	3643 (9)	-1850 (12)	-307 (12)	46 (10)	95 (19)	95 (16)	14 (20)	2 (16)	31 (26)
C(36)	3017 (8)	-1732 (12)	388 (10)	47 (9)	106 (19)	76 (14)	23 (20)	54 (16)	32 (22)
O(37)	4524 (7)	-727 (10)	-1570 (8)	77 (7)	140 (15)	95 (11)	-36 (18)	41 (13)	-1 (22)
C(38)	4729 (10)	-1947 (17)	-2022 (14)	89 (13)	158 (27)	176 (21)	1 (27)	139 (25)	-1 (37)

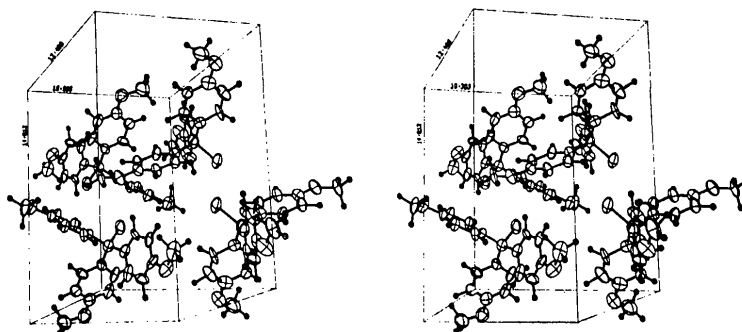


Fig. 2. Stereoscopic view of the contents of one unit cell, showing the molecular packing. The origin (rear upper-left corner) of the plotted cell contour is translated by  $(-\frac{1}{2}, -\frac{1}{2}, 0)$  with respect to the coordinate system fixing the positions *x*, *y*, *z* of the basic molecule (lower-left molecule).

Table 2. *Calculated positional parameters of hydrogen atoms* ( $\times 10^4$ )

*B* is equal to the isotropic thermal parameter of the carbon atom to which H is bonded. An asterisk denotes *observed* values taken from a difference synthesis.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )
H(121)	805	-1686	-44	4.66
H(131)	225	-3979	71	3.23
H(151)	1579	-4387	3344	3.30
H(161)	2224	-2139	3238	3.91
H(181)	326	-7155	2591	4.30
H(182)	551	-5721	3379	4.30
H(183)*	1477	-6472	2771	4.30
H(221)	1149	404	3134	4.32
H(231)	1847	949	5058	4.69
H(251)	4511	508	4088	4.61
H(261)	3779	-83	2200	3.12
H(281)	2721	2003	6549	5.29
H(282)*	2556	248	6479	5.29
H(283)	3520	976	7337	5.29
H(321)	2790	1540	311	4.86
H(331)	3960	1309	-832	5.74
H(351)	3882	-2792	-479	4.27
H(361)	2762	-2643	726	3.68
H(381)*	4088	-2551	-2206	6.13
H(382)	5235	-2495	-1482	6.13
H(383)	5005	-1809	-2801	6.13

unsubstituted triphenylchloromethane, the title compound does not display molecular threefold symmetry in the solid state. The overall conformation has the expected propeller shape with the restriction that one methoxyl group is oriented contrariwise with respect to the other two. Average bond distances and angles for a *p*-methoxyphenyl are given in Fig. 1. The C-Cl length 1.860 Å is greater than the sum of covalent radii. The C-C-Cl angle, averaged over the three bonds to the aryl groups, is 106.2 (6)°, whereas greater variations are encountered among the central C-C-C angles, with an average value of 112.5 (3.5)°. Even when no bulky *ortho* substituents (Sabacky, Johnson, Martin & Paul, 1969) are present the magnitude of the central C-C-C angle is largely fixed by steric interference between the phenyl groups, and does not seem to be much

affected by a central substituent atom, be it Cl or Br. The central angles only decrease from 112.6 to 112.2° in the series triphenylmethane (Riche & Pascard-Billy, 1974), triphenylchloromethane, triphenylbromomethane (Dunand & Gerdil, 1976) and triphenylmethane-benzene molecular complex (Allemand & Gerdil, 1975).

The central C atom lies significantly out of the least-squares planes of the adjacent phenyl rings at distances 0.13, 0.17 and 0.06 Å, respectively, the rings bending slightly away from the Cl atom. None of the methoxyl groups is coplanar with the adjacent aromatic ring; the twist angles are 2.8, 11.8 and 10.8° with rings 1, 2 and 3, respectively. In the same order, the twist angles of the phenyl rings about the central bonds are 42.6, 40.8 and 43.6° (the twist angle is 90° when the plane through the C-C-Cl fragment lies perpendicular to the mean plane of the phenyl ring). By consideration of intramolecular interatomic contacts, the torsion of a given phenyl ring about the central bond is controlled by the balance of two types of steric interference involving the *ortho* H atoms. The *exo* H atom is in close contact with the Cl atom at an average distance of 2.78 (5) Å, whereas the *endo* H atom penetrates the  $\pi$ -electron cloud of the neighbouring ring at a mean distance 2.40 (5) Å from C(1).

The molecular packing is depicted in the stereo view of Fig. 2. There is no direct contact between Cl atoms; the nearest approach occurs at 4.027 Å through a centre of symmetry.

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*Acta Cryst.* (1976). **B32**, 1593

## Structure Cristalline de FeUS<sub>3</sub>

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(Reçu le 5 janvier 1976, accepté le 21 janvier 1976)

**Abstract.** FeUS<sub>3</sub>, orthorhombic, *Cmcm*,  $a=3.795$  (3),  $b=11.626$  (5),  $c=8.717$  (4) Å,  $Z=4$ ,  $D_m=6.68$ ,  $D_x=6.73$  g cm<sup>-3</sup>. The crystal structure is of the UI<sub>3</sub> (or PuBr<sub>3</sub>) type, Fe being in octahedral sites, which are unoccupied in UI<sub>3</sub>.

**Introduction.** Les chalcogénures ternaires FeUS<sub>3</sub>, FeUSE<sub>3</sub> et MnUSE<sub>3</sub> constituent une famille de composés isotypes dans les systèmes binaires UX<sub>2</sub>-MX (M=élément de transition 3d; X=S, Se). Des monocristaux de FeUS<sub>3</sub> ont été obtenus par transport en