

Fe2	0.00000	-0.027433 (21)	0.25000	0.0525 (3)
O1B	0.08988 (15)	0.11400 (7)	0.22158 (11)	0.0653 (12)
C11B	0.01329 (23)	0.00908 (13)	0.15451 (15)	0.0599 (16)
C12B	-0.0127 (3)	-0.04931 (15)	0.14299 (19)	0.0785 (22)
C13B	0.0677 (3)	-0.08152 (13)	0.19150 (21)	0.0784 (23)
C14B	0.14239 (22)	-0.04303 (11)	0.23306 (16)	0.0598 (17)
C15B	0.10919 (20)	0.01393 (10)	0.21042 (13)	0.0484 (13)
C1B	0.16801 (19)	0.06976 (10)	0.23244 (14)	0.0501 (14)
C21B	0.23284 (20)	0.06863 (11)	0.31259 (15)	0.0521 (14)
C22B	0.20595 (22)	0.10055 (13)	0.36652 (18)	0.0661 (18)
C23B	0.2655 (3)	0.09817 (17)	0.43950 (20)	0.0885 (25)
C24B	0.3519 (3)	0.06354 (19)	0.45919 (19)	0.093 (3)
C25B	0.3808 (3)	0.03205 (16)	0.40646 (19)	0.0839 (21)
C26B	0.32353 (23)	0.03484 (13)	0.33397 (16)	0.0660 (17)
C31B	0.23728 (23)	0.08194 (12)	0.18059 (17)	0.0629 (18)
C32B	0.3100 (4)	0.12464 (18)	0.1970 (3)	0.111 (3)
C33B	0.3743 (5)	0.1361 (3)	0.1509 (4)	0.147 (5)
C34B	0.3656 (5)	0.1055 (3)	0.0884 (4)	0.134 (5)
C35B	0.2932 (5)	0.0644 (3)	0.0711 (3)	0.132 (4)
C36B	0.2293 (3)	0.05215 (18)	0.11698 (22)	0.099 (3)
HOA1	0.035	0.235	0.224	0.0642
HOA2	0.069	0.206	0.194	0.0642
HOB1	0.045	0.108	0.231	0.0770
HOB2	0.104	0.142	0.212	0.0770

Table 4. Summary of dimension ranges ( $\text{\AA}$ ,  $^\circ$ ) for (I)–(III)

	(I)	(II)	(III)
Fe—C <sub>cp</sub>	2.029(4)–2.053(3)	2.037(2)–2.057(2)	2.033(3)–2.052(2)
Mean	2.043(3)	2.048(2)	2.040(3)
C <sub>cp</sub> —C <sub>cp</sub>	1.406(4)–1.425(3)	1.388(3)–1.430(2)	1.405(5)–1.426(3)
Mean	1.419(3)	1.414(3)	1.418(4)
C <sub>cp</sub> —C <sub>sp</sub> <sup>3</sup>	1.515(3)	1.517(2)	1.516(3), 1.517(3)
C <sub>sp</sub> <sup>3</sup> —C <sub>sp</sub> <sup>3</sup>	1.518(3)	—	—
C <sub>ar</sub> —C <sub>sp</sub> <sup>3</sup>	1.526(3)	1.533(2)–1.534(2)	1.524(3)–1.543(3)
C <sub>sp</sub> <sup>3</sup> —O	1.441(3)	1.439(2)	1.427(3)–1.438(3)
C <sub>ar</sub> —C <sub>ar</sub>	1.362(4)–1.389(3)	1.363(4)–1.398(2)	1.334(10)–1.405(4)
Mean	1.380(3)	1.384(3)	1.374(6)
O—C—C <sub>sp</sub> <sup>3</sup>	108.69(17)	—	—
O—C—C <sub>ar</sub>	109.46(17)	105.1(1), 110.4(1)	106.0(2)–107.9(2)
O—C—C <sub>cp</sub>	106.83(17)	109.9(1)	107.9(2), 110.4(2)

1-Ferrocenyl-1-phenylethanol (I) was obtained from the reaction of acetylferrocene with phenyllithium, and crystals were grown by slow evaporation of a solution in CH<sub>2</sub>Cl<sub>2</sub>. Ferrocenyl(diphenyl)methanol (II) was obtained from the reaction of benzoylferrocene with phenyllithium; crystals were obtained by slow evaporation of a solution in CH<sub>2</sub>Cl<sub>2</sub>. Ferrocene-1,1'-diylbis(diphenylmethanol) (III) was obtained from the reaction of 1,1'-dibenzoylferrocene with phenyllithium; crystals were grown by slow evaporation of a solution in dichloromethane/petroleum (b.p. 313–333 K).

Data collection and cell refinement: Enraf-Nonius CAD-4 software. Data reduction, program used to solve and refine structure, software used to prepare material for publication: NRCVAX (Gabe, Le Page, Charland, Lee & White, 1989).

The space group for (I) was determined unambiguously from the systematic absences ( $h0l$  absent if  $h+l = 2n+1$ ,  $0k0$  absent if  $k = 2n+1$ ) as  $P2_1/n$ . Molecule (II) crystallized in the triclinic system; space group  $P1$  was assumed and confirmed by the analysis. The systematic absences for (III) ( $hkl$  absent if  $h+k = 2n+1$ ,  $h0l$  absent if  $l = 2n+1$ ) allow the space group to be either  $C2/c$  or  $Cc$ ; the former was assumed and confirmed by the analysis. In molecule (I) it was clear from early difference maps that the unsubstituted C<sub>5</sub> ring was disordered over two sites; occupancies of 0.87 and 0.13 were estimated from Fourier and difference Fourier maps. In all three structures, the H atoms attached to the C atoms were clearly visible in difference maps [except for the minor C<sub>5</sub> ring of (I)]; they were positioned geometrically (C—H 0.95  $\text{\AA}$ ) and included as riding atoms in the structure factor

calculations. The hydroxyl H atoms [disordered equally in (III) over two sites] were included in the structure factor calculations at the positions obtained from difference maps. The ORTEP diagrams were prepared using ORTEPII (Johnson, 1976).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55804 (77 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: L11034]

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## Structure of Dimethyl 9-Methyl-9,10-dihydro-9,10-ethenoanthracene-11,12-dicarboxylate

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## Abstract

There are two molecules per asymmetric unit and the two ester groups have different orientations in each of them; the C=C—C=O torsion angles are 1.9 and 2.4(6) $^\circ$  for the groups remote from and 91.7 and -91.4(5) $^\circ$  for the

groups adjacent to the 9-Me substituents. The remote ester groups are therefore fully conjugated with the C=C double bond [ $\cos^2(\text{angle}) = 1.0$ ] and the adjacent groups non-conjugated [ $\cos^2(\text{angle}) = 0$ ], presumably as a result of steric effects as in the 9-chloro derivative.

### Comment

The structure of the title compound was determined as part of a structural and photochemical study of dibenzobarrelene diesters (Pokkuluri, Scheffer & Trotter, 1989; Trotter & Yee, 1993). There are two nearly identical molecules per asymmetric unit (related by a rough pseudo-glide at  $z = 1/2$ ).

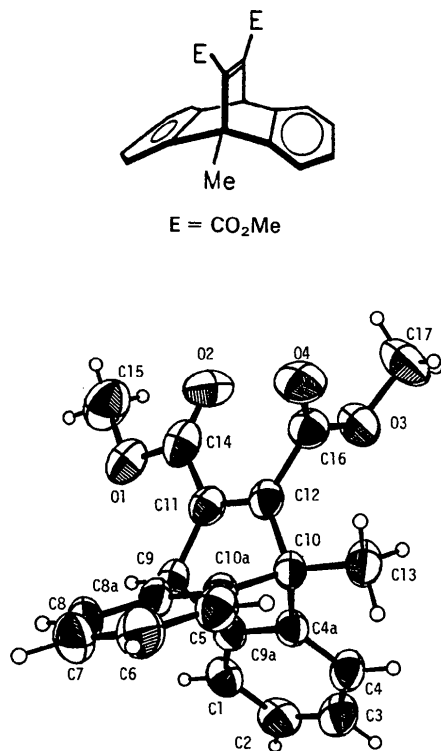


Fig. 1. View of the molecule (50% probability ellipsoids); chemical numbering system, except that Me is bonded to C10. (Molecule 1 is shown; stereoviews of both molecules are in the deposited material.)

### Experimental

#### Crystal data

C<sub>21</sub>H<sub>18</sub>O<sub>4</sub>

$M_r = 334.37$

Triclinic

$P\bar{1}$

$a = 16.164 (2) \text{ \AA}$

$b = 14.747 (1) \text{ \AA}$

$c = 8.3073 (6) \text{ \AA}$

$\alpha = 90.86 (7)^\circ$

$\beta = 112.82 (6)^\circ$

$\gamma = 108.74 (8)^\circ$

Cu K $\alpha$  radiation

$\lambda = 1.54056 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 14\text{--}21^\circ$

$\mu = 6.9 \text{ cm}^{-1}$

$T = 294 \text{ K}$

Plate

$0.4 \times 0.4 \times 0.2 \text{ mm}$

Colourless

$V = 1706.2 (3) \text{ \AA}^3$

$Z = 4$

$D_x = 1.302 \text{ Mg m}^{-3}$

#### Data collection

Nonius CAD-4F diffractometer

Absorption correction: analytical

$T_{\min} = 0.91$ ,  $T_{\max} = 0.80$

7008 measured reflections

7008 independent reflections

4292 observed reflections

$[3\sigma(I)]$

Crystal source: synthesis  
(see deposited material)

$\theta_{\max} = 75^\circ$

$h = -20 \rightarrow 20$

$k = 0 \rightarrow 18$

$l = -10 \rightarrow 10$

3 standard reflections

monitored every 150

reflections

intensity variation: none

#### Refinement

Refinement on  $F$

Final  $R = 0.066$

$wR = 0.084$

$S = 3.0$

4292 reflections

452 parameters

H-atom parameters not refined

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 0.87 \text{ e \AA}^{-3}$

Extinction correction: Coppins

Extinction coefficient:

$0.54 \times 10^4$

Atomic scattering factors

from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Data collection: CAD-4. Cell refinement: CAD-4. Data reduction: local programs. Program(s) used to solve structure: *MULTAN* (Main *et al.*, 1980). Program(s) used to refine structure: *ORFLS* (Busing, Martin & Levy, 1962). Molecular graphics: *ORTEPII* (Johnson, 1976).

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
C1	0.1104 (3)	-0.0350 (3)	-0.3202 (5)	0.056
C2	0.1413 (3)	-0.0379 (3)	-0.4563 (5)	0.068
C3	0.2310 (3)	-0.0381 (4)	-0.4202 (6)	0.069
C4	0.2932 (3)	-0.0365 (3)	-0.2484 (5)	0.058
C4a	0.2642 (2)	-0.0347 (3)	-0.1135 (4)	0.044
C5	0.2817 (3)	-0.2011 (3)	0.2016 (5)	0.053
C6	0.2142 (3)	-0.2759 (3)	0.2328 (6)	0.063
C7	0.1235 (3)	-0.2727 (3)	0.1918 (6)	0.065
C8	0.0988 (3)	-0.1954 (3)	0.1217 (5)	0.056
C8a	0.1660 (2)	-0.1211 (3)	0.0909 (5)	0.047
C9	0.1528 (3)	-0.0305 (3)	0.0151 (5)	0.050
C9a	0.1729 (3)	-0.0332 (3)	-0.1496 (5)	0.047
C10	0.3224 (2)	-0.0367 (3)	0.0841 (4)	0.042
C10a	0.2568 (2)	-0.1240 (3)	0.1287 (4)	0.044
C11	0.2354 (3)	0.0553 (3)	0.1488 (5)	0.050
C12	0.3216 (3)	0.0510 (3)	0.1835 (5)	0.047
C13	0.4212 (3)	-0.0393 (3)	0.1258 (5)	0.057
C14	0.2263 (3)	0.1376 (4)	0.2424 (6)	0.060
C15	0.1264 (4)	0.2100 (4)	0.2779 (8)	0.101
C16	0.4145 (3)	0.1187 (3)	0.3194 (5)	0.052
C17	0.5506 (4)	0.2580 (4)	0.3734 (7)	0.094
O1	0.1341 (2)	0.1258 (2)	0.1915 (4)	0.072
O2	0.2912 (3)	0.2026 (3)	0.3448 (4)	0.081
O3	0.4567 (2)	0.1924 (2)	0.2522 (3)	0.067
O4	0.4498 (2)	0.1042 (2)	0.4684 (3)	0.071
C1'	0.1107 (3)	0.4648 (3)	1.2232 (6)	0.057
C2'	0.1437 (3)	0.4682 (3)	1.4087 (6)	0.069
C3'	0.2346 (3)	0.4729 (3)	1.5094 (5)	0.071
C4'	0.2970 (3)	0.4725 (3)	1.4320 (5)	0.059
C4a'	0.2668 (2)	0.4693 (3)	1.2521 (4)	0.044

C5'	0.2887 (3)	0.2998 (3)	0.9692 (5)	0.052
C6'	0.2234 (3)	0.2232 (3)	0.8362 (6)	0.061
C7'	0.1325 (3)	0.2225 (3)	0.7364 (6)	0.063
C8'	0.1056 (3)	0.3002 (3)	0.7658 (5)	0.055
C8a'	0.1706 (2)	0.3766 (3)	0.8992 (4)	0.043
C9'	0.1548 (2)	0.4658 (3)	0.9540 (5)	0.046
C9a'	0.1747 (2)	0.4670 (3)	1.1475 (5)	0.044
C10'	0.3255 (2)	0.4660 (3)	1.1446 (4)	0.042
C10a'	0.2626 (2)	0.3766 (3)	1.0020 (4)	0.043
C11'	0.2352 (2)	0.5530 (3)	0.9455 (4)	0.045
C12'	0.3227 (2)	0.5521 (3)	1.0422 (4)	0.044
C13'	0.4264 (3)	0.4681 (3)	1.2550 (5)	0.057
C14'	0.2217 (3)	0.6326 (3)	0.8405 (5)	0.055
C15'	0.1114 (4)	0.7011 (4)	0.6689 (7)	0.097
C16'	0.4147 (3)	0.6212 (3)	1.0478 (5)	0.049
C17'	0.5472 (3)	0.7638 (4)	1.1969 (7)	0.080
O1'	0.1288 (2)	0.6207 (2)	0.7610 (4)	0.067
O2'	0.2846 (2)	0.6975 (2)	0.8298 (4)	0.072
O3'	0.4543 (2)	0.6967 (2)	1.1762 (4)	0.062
O4'	0.4500 (2)	0.6061 (2)	0.9518 (4)	0.064

Table 2. Selected bond lengths (Å) and ring-junction angles (°) with e.s.d.'s in parentheses

C=C	1.331, 1.333(5)	C=O	1.175–1.201(4)
C—C (aromatic)	1.358–1.416(6)	C—OMe	1.329–1.340(5)
C—CO <sub>2</sub> Me	1.488–1.509(6)	O—Me	1.447–1.488(6)
C—C (other)	1.510–1.550(6)		

External	125.7–127.2(3)
Internal (non-aromatic)	112.6–113.9(3)
C=C—CO <sub>2</sub> Me	120.2, 121.0(3) (at C11)
	125.8, 126.0(3) (at C12)
CH <sub>3</sub> —C—C	114.1–114.7(3)

The triclinic cell used was determined by the CAD-4 software; the transformation (001/010/101) produces the type I reduced cell (*International Tables for Crystallography*, Volume A, page 739) with  $a = 8.3073$ ,  $b = 14.747$ ,  $c = 15.038$  Å,  $\alpha = 69.29$ ,  $\beta = 82.21$ ,  $\gamma = 89.14^\circ$ .

Peaks of about  $0.8 \text{ e} \text{ \AA}^{-3}$  in the final difference map indicate minor disorder of one methoxycarbonyl group in each molecule; this disorder could not be satisfactorily modelled and is the reason for the slightly high  $R$  factors.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry together with details of the synthesis and stereo molecular and packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55869 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1025]

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## Structure of Dimethylammonium $N,N,N',N'$ -Tetramethylformamidinium Hexachlorotellurate(IV)

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## Abstract

The structure consists of three ionic species. The anion,  $\text{TeCl}_6^{2-}$ , is a slightly distorted octahedron with average  $\text{Te—Cl} = 2.532$  Å. One of the cations,  $(\text{CH}_3)_2\text{NH}_2^+$ , has  $\text{C—N} = 1.44$  (1) Å and  $\text{C—N—C} = 114.8$  (6)°. The other cation is the formamidinium [dimethyl(dimethylaminomethylene)ammonium] ion,  $(\text{CH}_3)_2\text{NC}(\text{H})\text{N}(\text{CH}_3)_2^+$ , with the central  $\text{C—N} = 1.30$  (1) Å,  $\text{N—C—N} = 129.8$  (8)° and the peripheral  $\text{C—N} = 1.46$  (1) Å. Planes through the terminal  $\text{C—N—C}$  atoms make angles of 14 and  $-14^\circ$ , respectively, relative to the best plane through all the non-H atoms of this ion. There are two short distances between the N atom of  $(\text{CH}_3)_2\text{NH}_2^+$  and Cl atoms of two different  $\text{TeCl}_6^{2-}$  units, directed approximately along the lines  $\text{N—H}\cdots\text{Cl}$  and with  $\text{N}\cdots\text{Cl} = 3.36, 3.44$  Å. There are also two short distances between the central C atom of  $(\text{CH}_3)_2\text{NC}(\text{H})\text{N}(\text{CH}_3)_2^+$  and Cl atoms of two different  $\text{TeCl}_6^{2-}$  units. The vector from one Cl to C atom is nearly perpendicular to the best plane through the N and C atoms of the ion,  $\text{C}\cdots\text{Cl} = 3.34$  Å. The vector from the other Cl to C is directed approximately along the line  $\text{C—H}\cdots\text{Cl}$  with  $\text{C}\cdots\text{Cl} = 3.68$  Å.

## Comment

The preparation of  $\text{C}_7\text{H}_{21}\text{Cl}_6\text{N}_3\text{Te}$  from  $N,N$ -dimethylformamide and  $\text{TeCl}_4$  was reported by Zingaro, Herrera & Meyers (1990). The structure of  $\text{TeCl}_6^{2-}$  in general positions and in the presence of unsymmetrical cations is that of a distorted octahedron (Abriel, 1987). Based on covalent radii taken from Pauling (1960), the expected  $\text{Te—Cl}$  distance is 2.51 Å, compared to the average value found here of 2.532 Å and a typical experimental value of 2.54 Å for  $(\text{NH}_4)_2\text{TeCl}_6$  (Hazell, 1966). The distortions are systematic with long and short  $\text{Te—Cl}$  distances approximately  $180^\circ$  from each other. In addition, the longest  $\text{Te—Cl}$  distances, 2.570 (2) and 2.618 (2) Å,

† Deceased.