Tableau 2. *Paramètres géométriques* (Å, °)

| | | | |
|---|---|---|---|
| O1—C1 | 1,210 (8) | C20—C21 | 1,374 (13) |
| O2—N1 | 1,434 (8) | C21—C22 | 1,340 (11) |
| O2—C10 | 1,502 (8) | C22—C23 | 1,359 (15) |
| O31—C31 | 1,215 (5) | C23—C24 | 1,401 (11) |
| O32—N31 | 1,416 (5) | C31—C32 | 1,450 (6) |
| O32—C40 | 1,500 (6) | C31—C40 | 1,549 (6) |
| N1—C11 | 1,273 (7) | C32—C33 | 1,376 (6) |
| N31—C41 | 1,275 (5) | C32—C37 | 1,376 (6) |
| C1—C2 | 1,464 (8) | C33—C34 | 1,385 (6) |
| C1—C10 | 1,542 (9) | C34—C35 | 1,404 (7) |
| C2—C3 | 1,386 (9) | C35—C36 | 1,392 (7) |
| C2—C7 | 1,370 (10) | C36—C37 | 1,372 (6) |
| C3—C4 | 1,378 (10) | C37—C38 | 1,522 (6) |
| C4—C5 | 1,383 (14) | C38—C39 | 1,558 (8) |
| C5—C6 | 1,395 (10) | C38—C40 | 1,535 (7) |
| C6—C7 | 1,416 (9) | C40—C42 | 1,527 (5) |
| C7—C8 | 1,522 (8) | C41—C42 | 1,535 (8) |
| C8—C9 | 1,569 (12) | C41—C49 | 1,460 (6) |
| C8—C10 | 1,539 (10) | C42—C43 | 1,504 (6) |
| C10—C12 | 1,522 (7) | C43—C44 | 1,403 (7) |
| C11—C12 | 1,523 (12) | C43—C48 | 1,395 (6) |
| C11—C19 | 1,478 (9) | C44—C45 | 1,401 (7) |
| C12—C13 | 1,509 (9) | C45—C46 | 1,364 (7) |
| C13—C14 | 1,389 (9) | C46—C47 | 1,388 (7) |
| C13—C18 | 1,395 (10) | C47—C48 | 1,369 (7) |
| C14—C15 | 1,382 (11) | C49—C50 | 1,402 (9) |
| C15—C16 | 1,399 (12) | C49—C54 | 1,379 (6) |
| C16—C17 | 1,365 (11) | C50—C51 | 1,379 (7) |
| C17—C18 | 1,374 (11) | C51—C52 | 1,383 (7) |
| C19—C20 | 1,387 (13) | C52—C53 | 1,368 (10) |
| C19—C24 | 1,361 (9) | C53—C54 | 1,382 (7) |

| | | | |
|---|---|---|---|
| N1—O2—C10 | 106,1 (4) | C36—C37—C38 | 127,6 (4) |
| O2—N1—C11 | 108,7 (6) | C37—C38—C39 | 109,8 (4) |
| O1—C1—C2 | 129,5 (6) | C37—C38—C40 | 102,6 (4) |
| O1—C1—C10 | 124,4 (5) | C39—C38—C40 | 113,4 (3) |
| C2—C1—C10 | 106,1 (5) | O32—C40—C31 | 101,6 (4) |
| C1—C2—C3 | 128,5 (7) | O32—C40—C38 | 105,3 (3) |
| C1—C2—C7 | 108,7 (5) | o32—C40—C42 | 103,8 (4) |
| C3—C2—C7 | 122,8 (6) | C31—C40—C38 | 104,4 (4) |
| C2—C3—C4 | 117,0 (8) | C31—C40—C42 | 113,9 (3) |
| C3—C4—C5 | 121,5 (7) | C38—C40—C42 | 125,1 (4) |
| C4—C5—C6 | 121,8 (7) | N31—C41—C42 | 113,8 (4) |
| C5—C6—C7 | 116,5 (8) | N31—C41—C49 | 120,5 (5) |
| C2—C7—C6 | 120,4 (6) | C42—C41—C49 | 125,5 (4) |
| C2—C7—C8 | 113,2 (5) | C40—C42—C41 | 99,0 (4) |
| C6—C7—C8 | 126,3 (7) | C40—C42—C43 | 117,1 (3) |
| C7—C8—C9 | 109,4 (6) | C41—C42—C43 | 109,9 (4) |
| C7—C8—C10 | 100,9 (6) | C42—C43—C44 | 120,2 (4) |
| C9—C8—C10 | 112,3 (5) | C42—C43—C48 | 120,9 (4) |
| O2—C10—C1 | 101,7 (5) | C14—C13—C18 | 118,0 (7) |
| O2—C10—C8 | 105,2 (5) | C13—C14—C15 | 121,8 (7) |
| O2—C10—C12 | 103,5 (5) | C14—C15—C16 | 119,1 (7) |
| C1—C10—C8 | 104,7 (5) | C15—C16—C17 | 119,1 (8) |
| C1—C10—C12 | 113,8 (5) | C16—C17—C18 | 122,0 (7) |
| C8—C10—C12 | 125,0 (6) | C13—C18—C17 | 120,0 (7) |
| N1—C11—C12 | 114,9 (6) | C11—C19—C20 | 120,8 (6) |
| N1—C11—C19 | 120,6 (7) | C11—C19—C24 | 120,8 (8) |
| C12—C11—C19 | 124,5 (5) | C20—C19—C24 | 118,3 (7) |
| C10—C12—C11 | 98,2 (5) | C19—C20—C21 | 119,1 (7) |
| C10—C12—C13 | 117,5 (6) | C20—C21—C22 | 122 (1) |
| C11—C12—C13 | 109,3 (6) | C21—C22—C23 | 120,0 (8) |
| C12—C13—C14 | 121,8 (6) | C22—C23—C24 | 118,6 (7) |
| C12—C13—C18 | 120,2 (6) | C19—C24—C23 | 121,6 (8) |
| N31—O32—C40 | 107,4 (3) | C44—C43—C48 | 118,9 (4) |
| O32—N31—C41 | 110,0 (4) | C43—C44—C45 | 119,0 (4) |
| O31—C31—C32 | 130,4 (4) | C44—C45—C46 | 121,1 (5) |
| O31—C31—C40 | 123,2 (4) | C45—C46—C47 | 119,9 (5) |
| C32—C31—C40 | 106,4 (3) | C46—C47—C48 | 120,2 (4) |
| C31—C32—C33 | 128,0 (4) | C43—C48—C47 | 120,9 (4) |
| C31—C32—C37 | 110,2 (4) | C41—C49—C50 | 119,8 (4) |
| C33—C32—C37 | 121,7 (4) | C41—C49—C54 | 121,7 (5) |
| C32—C33—C34 | 119,3 (4) | C50—C49—C54 | 118,4 (4) |
| C33—C34—C35 | 118,4 (4) | C49—C50—C51 | 119,7 (5) |
| C34—C35—C36 | 122,2 (4) | C50—C51—C52 | 121,4 (6) |
| C35—C36—C37 | 117,7 (4) | C51—C52—C53 | 118,6 (5) |
| C32—C37—C36 | 120,8 (4) | C52—C53—C54 | 120,9 (5) |
| C32—C37—C38 | 111,5 (3) | C49—C54—C53 | 121,0 (6) |

L'ensemble des calculs ont été effectués à l'aide d'un ordinateur Digital PDP 11/60 et de l'ensemble de programmes *SDP* (Frenz, 1985).

Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances et angles des atomes d'hydrogène, ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 55786: 11 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: PA1018]

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*Acta Cryst.* (1993). C**49**, 846–848

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


JAMES TROTTER AND VIVIEN C. YEE

*Department of Chemistry, University of British Columbia, Vancouver, BC, Canada V6T 1Z1*


(*Received* 23 *July* 1992; *accepted* 11 *November* 1992)


## Abstract

The two ester groups have different orientations, with C=C—C=O torsion angles of −165.7(3) and −89.4(4)° respectively for the groups remote from and adjacent to the Cl substituent. The remote ester group is therefore fully conjugated with the C=C double bond [$\cos^2$(angle) = 0.94] and the adjacent group non-conjugated [$\cos^2$(angle) = 0], presumably as a result of steric effects.


## Comment

The structure of the title compound was determined as part of a structural and photochemical study of dibenzo-

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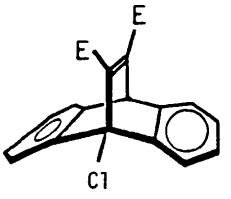
barrelene diesters (Garcia-Garibay, Scheffer, Trotter & Wireko, 1990; Pokkuluri, Scheffer & Trotter, 1993).




E = $CO_2Me$

## Experimental

*Crystal data*

$C_{20}H_{15}ClO_4$
$M_r$ = 354.79
Monoclinic
$P2_1/a$
$a$ = 11.865 (1) Å
$b$ = 13.800 (1) Å
$c$ = 10.568 (1) Å
$\beta$ = 104.25 (1)°
$V$ = 1677.1 (1) Å$^3$
$Z$ = 4
$D_x$ = 1.405 Mg m$^{-3}$
$D_m$ = 1.400 Mg m$^{-3}$
Density measured by flotation

Cu $K\alpha$ radiation
$\lambda$ = 1.54056 Å
Cell parameters from 25 reflections
$\theta$ = 31-46°
$\mu$ = 2.22 mm$^{-1}$
$T$ = 294 K
Prism
0.4 × 0.3 × 0.2 mm
Colourless
Crystal source: Yokohama (1987)

*Data collection*

Nonius CAD-4F diffractometer
Absorption correction: analytical
$T_{min}$ = 0.49, $T_{max}$ = 0.79
3438 measured reflections
3438 independent reflections
2291 observed reflections [$I > 3\sigma(I)$]

$\theta_{max}$ = 75°
$h = -14 \rightarrow 0$
$k = 0 \rightarrow 17$
$l = -12 \rightarrow 13$
3 standard reflections monitored every 150 reflections
intensity variation: none

*Refinement*

Refinement on $F$
Final $R$ = 0.046
$wR$ = 0.058
$S$ = 2.2
2291 reflections
287 parameters
H-atom coordinates and thermal parameters refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = 0.31 (H-atom parameter)

$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$
Extinction correction: Coppens
Extinction coefficient: 1.08 (4) × 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: Patterson and Fourier. Program(s) used to refine structure: *ORFLS* (Busing, Martin & Levy, 1962). Molecular graphics: *ORTEP*II (Johnson, 1976).

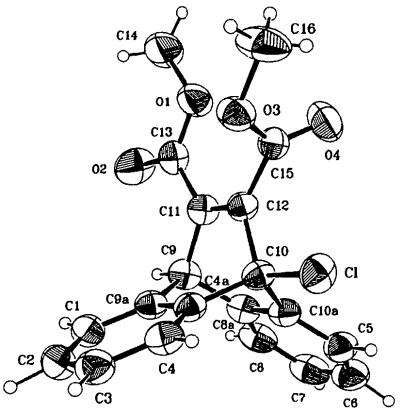



Fig. 1. View of the molecule (50% probability ellipsoids); the chemical-numbering system is used, except that Cl is bonded to C10.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2$)

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.7172 (3) | 0.2652 (3) | 0.4032 (3) | 0.050 |
| C2 | 0.7971 (3) | 0.2980 (3) | 0.3359 (4) | 0.055 |
| C3 | 0.8798 (3) | 0.3658 (3) | 0.3915 (4) | 0.053 |
| C4 | 0.8845 (3) | 0.4029 (2) | 0.5153 (3) | 0.046 |
| C4a | 0.8059 (3) | 0.3708 (2) | 0.5821 (3) | 0.040 |
| C5 | 0.6386 (3) | 0.5316 (3) | 0.7278 (3) | 0.052 |
| C6 | 0.5197 (4) | 0.5525 (3) | 0.7008 (4) | 0.064 |
| C7 | 0.4385 (3) | 0.4845 (4) | 0.6474 (4) | 0.065 |
| C8 | 0.4717 (3) | 0.3918 (3) | 0.6189 (4) | 0.057 |
| C8a | 0.5899 (3) | 0.3693 (2) | 0.6450 (3) | 0.045 |
| C9 | 0.6425 (3) | 0.2758 (2) | 0.6137 (3) | 0.046 |
| C9a | 0.7218 (3) | 0.3020 (2) | 0.5262 (3) | 0.041 |
| C10 | 0.7959 (2) | 0.4013 (2) | 0.7180 (3) | 0.040 |
| C10a | 0.6720 (3) | 0.4396 (2) | 0.7001 (3) | 0.042 |
| C11 | 0.7245 (3) | 0.2414 (2) | 0.7406 (3) | 0.043 |
| C12 | 0.8045 (3) | 0.3065 (2) | 0.7973 (3) | 0.040 |
| C13 | 0.7163 (3) | 0.1383 (2) | 0.7785 (3) | 0.047 |
| C14 | 0.7883 (8) | 0.0155 (3) | 0.9288 (6) | 0.084 |
| C15 | 0.8929 (3) | 0.2950 (2) | 0.9235 (3) | 0.042 |
| C16 | 1.0831 (4) | 0.2454 (6) | 1.0227 (5) | 0.078 |
| O1 | 0.7857 (3) | 0.1167 (2) | 0.8929 (2) | 0.064 |
| O2 | 0.6558 (2) | 0.0815 (2) | 0.7087 (3) | 0.074 |
| O3 | 0.9916 (2) | 0.2603 (2) | 0.9054 (2) | 0.053 |
| O4 | 0.8764 (2) | 0.3170 (2) | 1.0267 (2) | 0.064 |
| Cl | 0.9033 (1) | 0.4871 (1) | 0.7941 (1) | 0.053 |

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

| | | | |
|---|---|---|---|
| C=C | 1.338 (4) | C=O | 1.189, 1.193 (4) |
| C—C (aromatic) | 1.364-1.399 (5) | C—OMe | 1.319, 1.322 (4) |
| C—$CO_2$Me | 1.487, 1.489 (4) | O—Me | 1.446, 1.447 (5) |
| C—C (other) | 1.505-1.543 (4) | C—Cl | 1.780 (3) |

| | |
|---|---|
| Ring-junction angles | |
| External | 126.8-127.9 (3) |
| Internal (non-aromatic) | 111.2-113.7 (3) |
| C=C—$CO_2$Me | 127.4, 126.6 (3) |
| Cl—C—C | 111.9-113.3 (2) |

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 a packing diagram, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55830 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1019]

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