lution occurred in the crystallization process. The actual crystal studied has the absolute configuration shown in Figs. 1 and 2 (the opposite enantiomer had $R = 0.055$).

In (3), minor disorder is indicated by difference map peaks of up to 0.5 e Å^{-3}, particularly near the Cl atoms, but these could not be interpreted in terms of any meaningful geometry; this is the likely reason for the high R factor.

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 bond lengths and angles, details of synthesis and photolysis, UV-vis. and ESR spectra, and molecular and packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71297 (79 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1036]

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

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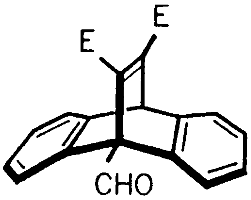
Abstract

The structure of the title compound has been determined. The molecular structure and dimensions are normal, with the carboxyl group adjacent to the formyl substituent not conjugated with the C11=C12 double bond and the remote carboxyl group fully conjugated.

Comment

The general molecular structure and dimensions of (1) (Fig. 1 and Table 2) are similar to those of related materials (Pokkuluri, Scheffer & Trotter, 1993). The carboxyl group adjacent to the CHO substituent on C9 is rotated out of the plane of the C11=C12 double bond [C12—C11—C13—O2 = 96.9 (3)°, cos^2(angle) = 0.01], presumably as a result of steric repulsions [C11—C9—C17 = 115.6 (2)°], and hence is not conjugated with the C=C bond; the remote carboxyl group is fully conjugated [C11—C12—C15—O4 = −179.0 (3)°, cos^2(angle) = 1.00]. The differences are reflected in the C—CO_2Me bond lengths of 1.489 (3) (non-conjugated) and 1.477 (3) Å (conjugated).



(1)

Photolysis of (1) yields a normal di-π-methane semibullvalene-type photoproduct (Chen, Pokkuluri, Scheffer & Trotter, 1990).

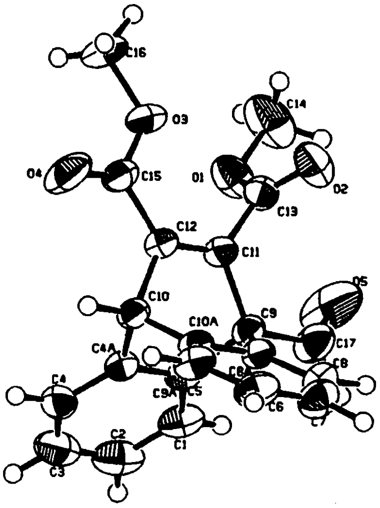


Fig. 1. View of the molecule (50% probability ellipsoids; stereo version in the supplementary material).

Experimental

Crystal data

$C_{21}H_{16}O_5$
$M_r = 348.35$

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å

Triclinic
$P\bar{1}$
$a = 10.176$ (1) Å
$b = 12.075$ (2) Å
$c = 8.077$ (1) Å
$\alpha = 108.88$ (1)°
$\beta = 112.79$ (1)°
$\gamma = 83.86$ (1)°
$V = 865.6$ (2) Å^3
$Z = 2$
$D_x = 1.34$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 25$–42°
$\mu = 0.75$ mm^{-1}
$T = 294$ K
Plate
0.3 × 0.2 × 0.08 mm
Colourless
Crystal source: synthesis (Chen, 1991)

Data collection

Rigaku AFC-6*S* diffractometer
Absorption correction: empirical (ψ scans)
$T_{\min} = 0.89$, $T_{\max} = 1.00$
3789 measured reflections
3520 independent reflections
2045 observed reflections [$3\sigma(I)$]

$R_{\text{int}} = 0.022$
$\theta_{\max} = 77.7°$
$h = -12 \rightarrow 11$
$k = -15 \rightarrow 14$
$l = 0 \rightarrow 9$
3 standard reflections monitored every 150 reflections intensity variation: 1.7%

Refinement

Refinement on F
Final $R = 0.040$
$wR = 0.052$
$S = 1.42$
2045 reflections
300 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

Extinction correction: *TEXSAN* (Molecular Structure Corporation, 1985)
Extinction coefficient: 1.15×10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: *TEXSAN* (Molecular Structure Corporation, 1985). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *TEXSAN*. The structure of (1) was determined by direct methods (*TEXSAN*).

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

$$B_{\text{eq}} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C1	0.1955 (3)	0.1664 (3)	0.7298 (4)	0.052 (2)
C2	0.2588 (4)	0.2185 (3)	0.9235 (5)	0.066 (2)
C3	0.3583 (4)	0.3064 (3)	0.9974 (4)	0.067 (2)
C4	0.3998 (3)	0.3443 (2)	0.8796 (4)	0.051 (1)
C4*A*	0.3391 (2)	0.2927 (2)	0.6873 (3)	0.039 (1)
C5	0.5464 (3)	0.1897 (2)	0.4027 (4)	0.047 (1)
C6	0.5722 (3)	0.0802 (2)	0.2964 (4)	0.056 (2)
C7	0.4724 (3)	−0.0089 (2)	0.2181 (4)	0.057 (2)
C8	0.3428 (3)	0.0092 (2)	0.2424 (4)	0.048 (1)
C8*A*	0.3168 (2)	0.1184 (2)	0.3489 (3)	0.037 (1)
C9	0.1823 (2)	0.1564 (2)	0.3940 (3)	0.038 (1)
C9*A*	0.2370 (2)	0.2034 (2)	0.6122 (3)	0.039 (1)
C10	0.3710 (2)	0.3221 (2)	0.5377 (3)	0.038 (1)
C10*A*	0.4182 (2)	0.2084 (2)	0.4274 (3)	0.038 (1)
C11	0.1287 (2)	0.2652 (2)	0.3291 (3)	0.037 (1)
C12	0.2274 (2)	0.3502 (2)	0.4037 (3)	0.038 (1)
C13	−0.0185 (2)	0.2655 (2)	0.1885 (3)	0.042 (1)
C14	−0.2546 (3)	0.3226 (5)	0.1423 (6)	0.090 (3)
C15	0.2111 (3)	0.4657 (2)	0.3725 (3)	0.044 (1)
C16	0.0515 (5)	0.5986 (3)	0.2388 (7)	0.082 (3)
C17	0.0736 (3)	0.0590 (2)	0.3183 (4)	0.058 (2)
O1	−0.1080 (2)	0.3205 (2)	0.2685 (2)	0.061 (1)
O2	−0.0504 (2)	0.2194 (2)	0.0216 (3)	0.064 (1)
O3	0.0811 (2)	0.4833 (1)	0.2615 (3)	0.063 (1)
O4	0.3063 (2)	0.5364 (2)	0.4437 (3)	0.078 (1)
O5	−0.0499 (2)	0.0713 (2)	0.2678 (5)	0.117 (2)

Table 2. *Geometric parameters* (Å, °)

C1—C2	1.388 (4)	C9—C11	1.539 (3)
C1—C9*A*	1.381 (3)	C9—C17	1.507 (3)
C2—C3	1.364 (5)	C10—C10*A*	1.519 (3)
C3—C4	1.388 (4)	C10—C12	1.526 (3)
C4—C4*A*	1.377 (3)	C11—C12	1.333 (3)
C4*A*—C9*A*	1.392 (3)	C11—C13	1.489 (3)
C4*A*—C10	1.516 (3)	C12—C15	1.477 (3)
C5—C6	1.386 (4)	C13—O1	1.318 (3)
C5—C10*A*	1.379 (3)	C13—O2	1.203 (3)
C6—C7	1.373 (4)	C14—O1	1.448 (3)
C7—C8	1.392 (4)	C15—O3	1.320 (3)
C8—C8*A*	1.384 (3)	C15—O4	1.194 (3)
C8*A*—C9	1.536 (3)	C16—O3	1.448 (4)
C8*A*—C10*A*	1.391 (3)	C17—O5	1.170 (3)
C9—C9*A*	1.549 (3)		
C2—C1—C9*A*	118.9 (3)	C4*A*—C9*A*—C9	112.4 (2)
C1—C2—C3	121.0 (3)	C4*A*—C10—C10*A*	105.6 (2)
C2—C3—C4	120.4 (3)	C4*A*—C10—C12	105.5 (2)
C3—C4—C4*A*	119.4 (3)	C10*A*—C10—C12	106.5 (2)
C4—C4*A*—C9*A*	120.1 (2)	C5—C10*A*—C8*A*	120.6 (2)
C4—C4*A*—C10	126.8 (2)	C5—C10*A*—C10	126.5 (2)
C9*A*—C4*A*—C10	113.1 (2)	C8*A*—C10*A*—C10	113.0 (2)
C6—C5—C10*A*	119.0 (2)	C9—C11—C12	113.4 (2)
C5—C6—C7	120.7 (3)	C9—C11—C13	120.0 (2)
C6—C7—C8	120.7 (2)	C12—C11—C13	126.5 (2)
C7—C8—C8*A*	118.8 (2)	C10—C12—C11	114.4 (2)
C8—C8*A*—C9	127.1 (2)	C10—C12—C15	118.3 (2)
C8—C8*A*—C10*A*	120.2 (2)	C11—C12—C15	127.3 (2)
C9—C8*A*—C10*A*	112.7 (2)	C11—C13—O1	112.3 (2)
C8*A*—C9—C9*A*	104.9 (2)	C11—C13—O2	123.0 (2)
C8*A*—C9—C11	106.2 (2)	O1—C13—O2	124.7 (2)
C8*A*—C9—C17	113.7 (2)	C12—C15—O3	113.4 (2)
C9*A*—C9—C11	104.7 (2)	C12—C15—O4	122.8 (2)
C9*A*—C9—C17	110.8 (2)	O3—C15—O4	123.7 (3)
C11—C9—C17	115.6 (2)	C9—C17—O5	124.8 (3)
C1—C9*A*—C4*A*	120.3 (2)	C13—O1—C14	116.0 (2)
C1—C9*A*—C9	127.4 (2)	C15—O3—C16	117.2 (3)

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 and packing and stereo molecular diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71390 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1045]

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