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Acta Cryst. (1996). C**52**, 674–675

Methyl *trans*-9,10-Dihydro-9,10-ethanoanthracene-11-carboxylate

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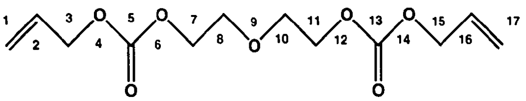
(Received 27 June 1995; accepted 8 August 1995)

Abstract

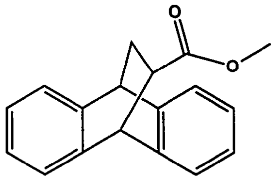
The title compound, $C_{18}H_{16}O_2$, was prepared as an important intermediate in the preparation of deuterium-labelled diethylene glycol bis(allyl carbonate). The bond lengths and angles are similar to those reported for *trans*-9,10-dihydro-9,10-ethanoanthracene-11-carboxylic acid.

Comment

Recent studies of the free radical polymerization of diethylene glycol bis(allyl carbonate), (1), have shown that hydrogen abstraction from position 8 is kinetically favoured, but the thermodynamic product is that derived from allylic abstraction (Qureshi, Solomon & Kelly, 1995; Qureshi, Schiesser & Solomon, 1995). Intramolecular hydrogen transfer from the allylic position 3 to position 8 would explain the formation of the allylic radicals. To test this hypothesis, compound (1), deuterium-labelled at positions 3 and 15, was synthesized. An important intermediate in this preparation was methyl *trans*-9,10-dihydro-9,10-ethanoanthracene-11-carboxylate, (2), which was prepared following the method of Wawzonek & Hallum (1953). While the ^{13}C NMR spectrum could be assigned in accordance with the structure, the aromatic protons in the ^1H NMR spectrum could not. An X-ray structural analysis of (2) was carried out in order to confirm the identity of this compound.



(1)



(2)

The bond lengths and angles found for compound (2) are similar to those found for the corresponding carboxylic acid, *trans*-9,10-dihydro-9,10-ethanoanthracene-11-carboxylic acid (Csoregh, Czugler, Ertan, Weber & Ahrendt, 1990), which crystallizes as a clathrate with dimethyl sulfoxide. Also, the orientation of the carboxylate group, with a torsion angle C(12)—C(11)—C(13)—O(1) of 10.4 (2)°, is similar to that observed for the carboxylic acid group, where the corresponding torsion angle is −5.0°.

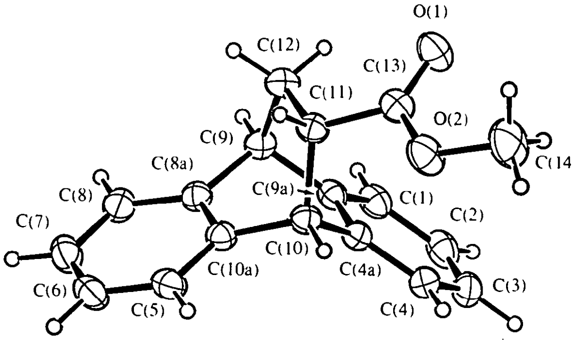


Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of compound (2). Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Crystals of the title compound were obtained from light petroleum.

Crystal data

$C_{18}H_{16}O_2$
$M_r = 264.31$
Monoclinic
$P2_1/n$
$a = 13.1844\ (9)$ Å
$b = 6.2358\ (7)$ Å
$c = 17.562\ (2)$ Å
$\beta = 107.719\ (6)°$

Cu $K\alpha$ (Ni-filtered) radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 21.6–40.6°$
$\mu = 0.650\ \text{mm}^{-1}$
$T = 293\ (1)$ K

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$V = 1375.4\,(2)$ Å^3
$Z = 4$
$D_x = 1.276$ Mg m^{-3}

Wedge
0.25 × 0.25 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 MACHS diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3794 measured reflections
2822 independent reflections
2295 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0177$
$\theta_{\rm max} = 74.89°$
$h = -1 \rightarrow 16$
$k = -7 \rightarrow 1$
$l = -21 \rightarrow 21$
3 standard reflections
frequency: 150 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0359$
$wR(F^2) = 0.0958$
$S = 1.024$
2822 reflections
246 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.2514P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$

$\Delta\rho_{\rm max} = 0.169$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.119$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0029 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
O(1)	0.28961 (9)	−0.0157 (2)	0.64459 (7)	0.0645 (3)
O(2)	0.33628 (10)	0.3196 (2)	0.62674 (6)	0.0664 (3)
C(1)	0.05631 (11)	−0.0672 (3)	0.39314 (8)	0.0491 (3)
C(2)	−0.01914 (11)	0.0808 (3)	0.39970 (8)	0.0571 (4)
C(3)	0.01138 (12)	0.2848 (3)	0.42788 (9)	0.0576 (4)
C(4)	0.11781 (11)	0.3438 (2)	0.45098 (8)	0.0487 (3)
C(4a)	0.19316 (10)	0.1979 (2)	0.44376 (7)	0.0403 (3)
C(5)	0.41210 (11)	0.2981 (3)	0.36443 (8)	0.0493 (3)
C(6)	0.44212 (12)	0.2183 (3)	0.30073 (9)	0.0595 (4)
C(7)	0.41031 (12)	0.0168 (3)	0.27080 (9)	0.0601 (4)
C(8)	0.34835 (11)	−0.1110 (3)	0.30400 (8)	0.0514 (3)
C(8a)	0.31885 (9)	−0.0325 (2)	0.36797 (7)	0.0416 (3)
C(9)	0.25558 (10)	−0.1491 (2)	0.41385 (7)	0.0419 (3)
C(9a)	0.16273 (9)	−0.0088 (2)	0.41480 (7)	0.0408 (3)
C(10)	0.31233 (10)	0.2326 (2)	0.46794 (7)	0.0401 (3)
C(10a)	0.35064 (9)	0.1724 (2)	0.39810 (7)	0.0407 (3)
C(11)	0.36020 (10)	0.0645 (2)	0.53548 (7)	0.0404 (3)
C(12)	0.32958 (11)	−0.1631 (2)	0.50203 (8)	0.0451 (3)
C(13)	0.32422 (10)	0.1116 (2)	0.60785 (8)	0.0458 (3)
C(14)	0.2993 (3)	0.3896 (5)	0.69251 (14)	0.0895 (7)

Table 2. *Selected geometric parameters (Å, °)*

O(1)—C(13)	1.198 (2)	C(9)—C(12)	1.561 (2)
O(2)—C(13)	1.336 (2)	C(10)—C(10a)	1.509 (2)
O(2)—C(14)	1.451 (2)	C(10)—C(11)	1.564 (2)
C(4a)—C(10)	1.513 (2)	C(11)—C(13)	1.514 (2)
C(8a)—C(9)	1.511 (2)	C(11)—C(12)	1.542 (2)
C(9)—C(9a)	1.509 (2)		
C(4)—C(4a)—C(10)	126.49 (12)	C(10a)—C(10)—C(4a)	108.20 (10)
C(9a)—C(4a)—C(10)	113.08 (11)	C(10a)—C(10)—C(11)	106.99 (10)
C(8)—C(8a)—C(9)	126.81 (13)	C(5)—C(10a)—C(10)	126.25 (12)
C(10a)—C(8a)—C(9)	112.87 (11)	C(8a)—C(10a)—C(10)	113.52 (11)
C(9a)—C(9)—C(8a)	107.95 (10)	O(1)—C(13)—O(2)	123.09 (13)
C(9a)—C(9)—C(12)	106.60 (10)	O(1)—C(13)—C(11)	126.26 (13)
C(8a)—C(9)—C(12)	106.04 (10)	O(2)—C(13)—C(11)	110.64 (11)

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1205). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 675–677

5-Methyl-2-thiophenecarboxylic Acid

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(*Received 26 June 1995; accepted 5 September 1995*)

Abstract

The present X-ray diffraction study establishes the molecular structure of 5-methyl-2-thiophenecarboxylic acid, $C_6H_6O_2S$. The crystal structure is stabilized by an O—H···O hydrogen bond and C—H···O and S···S non-bonded intermolecular interactions.

Comment

The crystal and molecular structure of 5-methyl-2-thiophenecarboxylic acid, (I), was first reported by Simonsen, Cordell & Boggs (1980) without atomic

Acta Crystallographica Section C
ISSN 0108-2701 ©1996

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† Contribution No. 1338 of the Instituto de Química, UNAM.