organic compounds

9-(Dimethoxymethyl)anthracene

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In the title compound, $C_{17}H_{16}O_2$, a dimethyl acetal of 9-anthraldehyde, the anthracene skeleton is planar and the two methoxy groups attached to the C atom at position 9 of the aromatic ring system are located above and below the ring system.

Related literature

For 9-chloro-10-(dimethoxymethyl)anthracene, see: Yuan *et al.* (2004). For anologous compounds, see: Akiba *et al.* (1999); Yamashita *et al.* (2005).
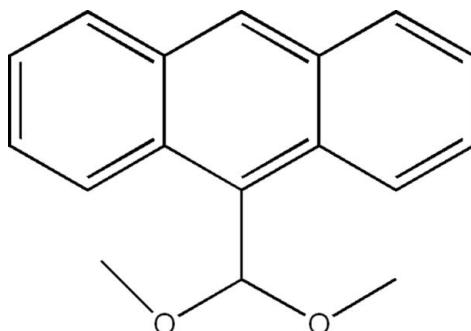
Experimental

Crystal data

$C_{17}H_{16}O_2$	V = 2766.0 (10) Å^3
M_r = 252.30	Z = 8
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
a = 8.2310 (16) Å	μ = 0.08 mm^{-1}
b = 17.446 (4) Å	T = 291 (2) K
c = 19.261 (4) Å	0.16 × 0.12 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer	12827 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	2433 independent reflections
T_{min} = 0.987, T_{max} = 0.990	1215 reflections with $I > 2\sigma(I)$
	R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052	174 parameters
$wR(F^2)$ = 0.166	H-atom parameters constrained
S = 0.90	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
2433 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2672).

References

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supporting information

9-(Dimethoxymethyl)anthracene

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S1. Comment

Anthracene is a very useful compound for supramolecular photochemistry. Several crystal structures of anthracene derivatives have been reported previously (Akiba *et al.*, 1999; Yuan *et al.*, 2004; Yamashita *et al.*, 2005;).

The atom-numbering scheme of the title compound is shown in Fig. 1. The bond lengths and angles are in the normal ranges compared with related compounds. The dihedral angles between the anthracene plane and the C15—O1—C16 and C15—O2—C17 groups are 71.6 (3) and 75.8 (4)°, respectively, and that between the latter two groups is 85.6 (2)°. These dihedral angles are silghtly different from 9-chloro-10-(dimethoxymethyl)anthracene (Yuan *et al.*, 2004)

S2. Experimental

The treatment of 9-anthraldehyde (0.5 mmol, 0.010 g) and 4-aminopyridine (0.5 mmol, 0.041 g) in 20 cm³ me thanol with a few drops of HOAc as catalyst under reflux condition for 2 h produces the yellow compound 9-(dimethoxymethyl)-anthracene as a by-product. Single crystals suitable for X-ray diffraction measurement formed after 1 week by slow evaporation at room temperature in air. Anal. Calcd. for $C_{17}H_{16}O_2$: C, 80.93; H, 6.39; O, 12.68%. Found: C, 81.00; H, 6.33; O, 12.74%. FT—IR (KBr pellets, cm⁻¹): 2932, 1448, 1186, 1105, 1066, 891, 740. ¹H NMR (500 MHz, CDCl₃): d = 3.57 (s, 6H), 6.59 (s, 1H), 7.29–7.56 (m, 4H), 8.01(d, 2H, J = 8.4 Hz), 8.49 (s, 1H), 8.74 (d, 2H, J = 8.9 Hz).

S3. Refinement

H atoms were placed in geometrically idealized positions (C—H = 0.93–0.98 Å) and refined as riding atoms, with U_{iso}(H) = 1.5U_{eq}(for methyl C) or U_{iso}(H) = 1.2U_{eq}(C) for the other C atoms. The methyl groups were allowed to rotate but not to tip.
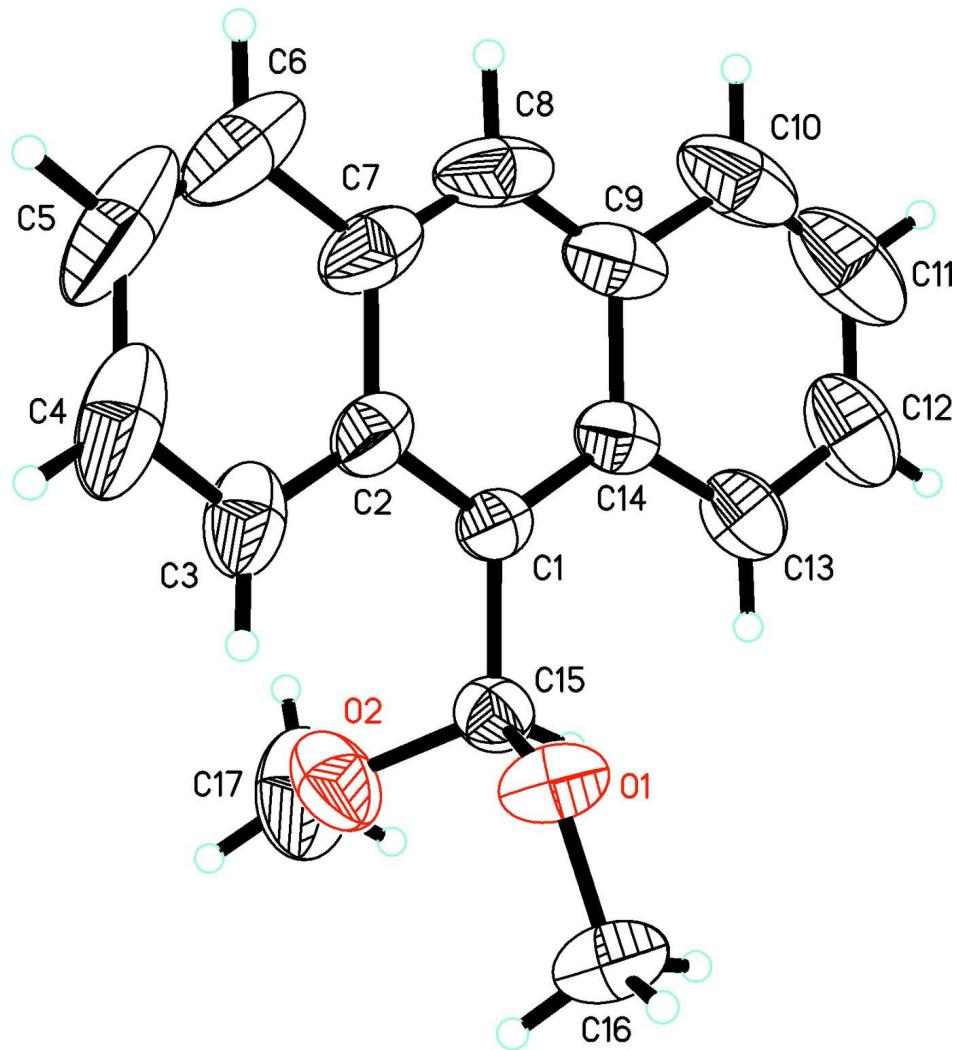
Figure 1

A perspective view of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

9-(dimethoxymethyl)anthracene

Crystal data

$C_{17}H_{16}O_2$	$F(000) = 1072$
$M_r = 252.30$	$D_x = 1.212$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1781 reflections
$a = 8.2310\,(16)$ Å	$\theta = 2.4$–19.5°
$b = 17.446\,(4)$ Å	$\mu = 0.08$ mm^{-1}
$c = 19.261\,(4)$ Å	$T = 291$ K
$V = 2766.0\,(10)$ Å^3	Block, yellow
$Z = 8$	$0.16 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer	12827 measured reflections
Radiation source: fine-focus sealed tube	2433 independent reflections
Graphite monochromator	1215 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.045$
Absorption correction: multi-scan ($SADABS$; Bruker, 2000)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
	$h = -9 \rightarrow 4$
	$k = -20 \rightarrow 20$
$T_{min} = 0.987$, $T_{max} = 0.990$	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.052$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.166$	
$S = 0.90$	H-atom parameters constrained
2433 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0954P)^2]$
174 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.14$ e Å^{-3}
	$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

Experimental. The structure was solved by direct methods (Bruker, 2000) and successive difference Fourier syntheses.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.9017 (2)	0.20745 (12)	0.59464 (10)	0.0592 (6)
C2	1.0164 (3)	0.15624 (15)	0.62298 (12)	0.0719 (7)
C3	1.0584 (3)	0.08576 (16)	0.59060 (17)	0.0944 (9)
H3	1.0052	0.0709	0.5502	0.113*
C4	1.1755 (4)	0.03974 (19)	0.6180 (2)	0.1378 (15)
H4	1.2030	−0.0056	0.5956	0.165*
C5	1.2563 (5)	0.0603 (4)	0.6806 (3)	0.170 (3)
H5	1.3356	0.0282	0.6991	0.204*
C6	1.2182 (5)	0.1251 (3)	0.7122 (2)	0.1453 (19)
H6	1.2704	0.1376	0.7535	0.174*
C7	1.0996 (3)	0.1764 (2)	0.68494 (16)	0.0975 (9)
C8	1.0683 (4)	0.2472 (3)	0.71548 (15)	0.1118 (13)
H8	1.1193	0.2591	0.7572	0.134*
C9	0.9649 (4)	0.3003 (2)	0.68643 (14)	0.0918 (9)
C10	0.9437 (5)	0.3758 (3)	0.71550 (19)	0.1275 (14)

H10	0.9967	0.3890	0.7564	0.153*
C11	0.8487 (6)	0.4268 (3)	0.6839 (3)	0.1449 (18)
H11	0.8379	0.4755	0.7029	0.174*
C12	0.7655 (4)	0.40904 (18)	0.6236 (2)	0.1184 (11)
H12	0.7001	0.4458	0.6027	0.142*
C13	0.7787 (3)	0.33873 (15)	0.59484 (15)	0.0859 (8)
H13	0.7208	0.3279	0.5545	0.103*
C14	0.8787 (3)	0.28072 (14)	0.62451 (11)	0.0683 (6)
C15	0.8077 (3)	0.18624 (13)	0.53064 (11)	0.0665 (6)
H15	0.7224	0.2244	0.5228	0.080*
C16	0.8383 (4)	0.19124 (18)	0.40849 (13)	0.1082 (10)
H16A	0.7751	0.1457	0.4013	0.162*
H16B	0.9189	0.1954	0.3727	0.162*
H16C	0.7683	0.2352	0.4071	0.162*
C17	0.5853 (4)	0.11064 (19)	0.5615 (2)	0.1447 (14)
H17A	0.5903	0.1194	0.6106	0.217*
H17B	0.5388	0.0611	0.5528	0.217*
H17C	0.5191	0.1494	0.5402	0.217*
O1	0.91609 (19)	0.18736 (10)	0.47445 (8)	0.0856 (6)
O2	0.7373 (2)	0.11349 (10)	0.53439 (10)	0.1011 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0515 (12)	0.0712 (14)	0.0550 (12)	−0.0066 (10)	−0.0002 (10)	0.0067 (11)
C2	0.0531 (13)	0.0901 (18)	0.0726 (15)	−0.0153 (12)	−0.0023 (12)	0.0273 (13)
C3	0.0662 (16)	0.0813 (18)	0.136 (2)	0.0036 (14)	0.0014 (15)	0.0344 (18)
C4	0.085 (2)	0.116 (3)	0.212 (4)	0.0196 (19)	0.016 (3)	0.085 (3)
C5	0.076 (3)	0.217 (6)	0.217 (6)	0.015 (3)	−0.002 (3)	0.159 (5)
C6	0.075 (2)	0.228 (5)	0.133 (3)	−0.030 (3)	−0.028 (2)	0.112 (4)
C7	0.0644 (17)	0.152 (3)	0.0764 (19)	−0.0268 (18)	−0.0093 (15)	0.047 (2)
C8	0.088 (2)	0.197 (4)	0.0512 (17)	−0.063 (2)	−0.0075 (16)	0.017 (2)
C9	0.0807 (18)	0.134 (3)	0.0610 (16)	−0.0455 (18)	0.0157 (15)	−0.0198 (17)
C10	0.122 (3)	0.168 (4)	0.093 (2)	−0.070 (3)	0.036 (2)	−0.057 (3)
C11	0.133 (4)	0.138 (4)	0.164 (4)	−0.051 (3)	0.069 (3)	−0.078 (3)
C12	0.109 (2)	0.084 (2)	0.162 (3)	−0.0046 (17)	0.035 (2)	−0.034 (2)
C13	0.0760 (17)	0.0774 (18)	0.104 (2)	−0.0029 (13)	0.0122 (15)	−0.0166 (15)
C14	0.0594 (13)	0.0861 (17)	0.0592 (14)	−0.0165 (12)	0.0092 (11)	−0.0070 (13)
C15	0.0599 (13)	0.0661 (15)	0.0734 (15)	0.0031 (11)	−0.0072 (12)	−0.0050 (11)
C16	0.116 (2)	0.140 (3)	0.0684 (17)	0.0100 (19)	−0.0208 (17)	−0.0019 (16)
C17	0.090 (2)	0.130 (3)	0.214 (4)	−0.0208 (19)	0.020 (2)	0.042 (3)
O1	0.0743 (11)	0.1222 (15)	0.0603 (10)	0.0161 (9)	−0.0063 (9)	−0.0078 (8)
O2	0.0775 (12)	0.0773 (12)	0.1486 (18)	−0.0096 (9)	−0.0174 (11)	−0.0122 (10)

Geometric parameters (Å, º)

C1—C2	1.410 (3)	C10—H10	0.9300
C1—C14	1.415 (3)	C11—C12	1.384 (6)

C1—C15	1.501 (3)	C11—H11	0.9300
C2—C7	1.420 (4)	C12—C13	1.350 (4)
C2—C3	1.421 (4)	C12—H12	0.9300
C3—C4	1.361 (4)	C13—C14	1.424 (3)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.422 (7)	C15—O2	1.397 (3)
C4—H4	0.9300	C15—O1	1.403 (2)
C5—C6	1.324 (6)	C15—H15	0.9800
C5—H5	0.9300	C16—O1	1.424 (3)
C6—C7	1.425 (5)	C16—H16A	0.9600
C6—H6	0.9300	C16—H16B	0.9600
C7—C8	1.392 (4)	C16—H16C	0.9600
C8—C9	1.377 (4)	C17—O2	1.357 (3)
C8—H8	0.9300	C17—H17A	0.9600
C9—C14	1.429 (3)	C17—H17B	0.9600
C9—C10	1.443 (5)	C17—H17C	0.9600
C10—C11	1.331 (5)		
C2—C1—C14	120.3 (2)	C10—C11—H11	119.2
C2—C1—C15	120.4 (2)	C12—C11—H11	119.2
C14—C1—C15	119.20 (19)	C13—C12—C11	120.5 (4)
C1—C2—C7	119.4 (3)	C13—C12—H12	119.7
C1—C2—C3	122.8 (2)	C11—C12—H12	119.7
C7—C2—C3	117.8 (3)	C12—C13—C14	121.8 (3)
C4—C3—C2	120.8 (3)	C12—C13—H13	119.1
C4—C3—H3	119.6	C14—C13—H13	119.1
C2—C3—H3	119.6	C1—C14—C13	123.8 (2)
C3—C4—C5	120.8 (4)	C1—C14—C9	119.3 (2)
C3—C4—H4	119.6	C13—C14—C9	116.9 (2)
C5—C4—H4	119.6	O2—C15—O1	108.44 (18)
C6—C5—C4	119.7 (4)	O2—C15—C1	113.28 (18)
C6—C5—H5	120.2	O1—C15—C1	107.61 (17)
C4—C5—H5	120.2	O2—C15—H15	109.1
C5—C6—C7	122.0 (5)	O1—C15—H15	109.1
C5—C6—H6	119.0	C1—C15—H15	109.1
C7—C6—H6	119.0	O1—C16—H16A	109.5
C8—C7—C2	119.1 (3)	O1—C16—H16B	109.5
C8—C7—C6	121.9 (4)	H16A—C16—H16B	109.5
C2—C7—C6	119.0 (4)	O1—C16—H16C	109.5
C9—C8—C7	122.7 (3)	H16A—C16—H16C	109.5
C9—C8—H8	118.7	H16B—C16—H16C	109.5
C7—C8—H8	118.7	O2—C17—H17A	109.5
C8—C9—C14	119.0 (3)	O2—C17—H17B	109.5
C8—C9—C10	122.1 (4)	H17A—C17—H17B	109.5
C14—C9—C10	118.8 (3)	O2—C17—H17C	109.5
C11—C10—C9	120.2 (4)	H17A—C17—H17C	109.5
C11—C10—H10	119.9	H17B—C17—H17C	109.5
C9—C10—H10	119.9	C15—O1—C16	113.76 (19)

| C10—C11—C12 | 121.7 (4) | C17—O2—C15 | 115.8 (2) |