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1,4,5,8-Tetramethoxyanthracene

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.052
wR factor = 0.140
Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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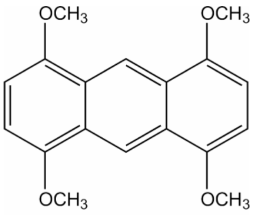
The structure of the title compound, $C_{18}H_{18}O_4$, consists of separate centrosymmetric planar molecules arranged in columns parallel to the monoclinic unique axis. The normal to the mean molecular plane lies at 55.4° to the unique axis, giving adjacent columns with alternating stacking directions. The mean deviation of all non-H atoms from the least-squares molecular plane is 0.064 Å. Bond lengths in the aromatic rings range from 1.347 (2) to 1.440 (2) Å and the two unique Me—O bond distances are 1.418 (2) and 1.419 (2) Å.

Comment

There has been considerable ongoing research involving the electrochemistry of compounds incorporating multiple quinones. Some of these compounds are anthracene derivatives (Jozefiak *et al.*, 1989), while others are based on triptycene architecture (Doerner *et al.*, 1998; Jozefiak *et al.*, 1989; Quast & Fuchsbauer, 1986; Russell & Suleman, 1981). 1,4,5,8-Tetramethoxyanthracene (1,4,5,8-TMA), (I), has been used as an intermediate in the syntheses of some of these compounds. During research involving the synthesis of molecules that can undergo photoinduced electron transfer, 1,4,5,8-TMA was used as a synthetic intermediate.



(I)

The structure of (I) consists of individual molecules having no unusually short intermolecular contacts. The packing is in columns parallel to the *b* axis. Each molecule is planar, as shown in Fig. 1, with an average deviation from the least-squares plane through all non-H atoms of 0.064 Å, and a maximum of 0.164 Å by the methyl atom C3′. The molecule is slightly distorted, since aromatic ring angles deviate from 120°, ranging from 118.44 (13)° at C2—C7—C6 to 122.27 (14)° at C1—C7—C6. Perhaps the most interesting feature of this structure is the tilt of the methoxy groups. The two independent methoxy groups bend toward each other down the length of the molecule, with facing O—C—C angles of 113.85 (13) and 114.16 (13)°. Since the nearest intermolecular distances are typical of graphitic lamellar structures, these distortions are most likely due to intramolecular steric interactions between the methyl H atoms and their nearest-neighbor aromatic H atom. This type of distortion is in agreement with gas-phase molecular mechanics (MM2) calculations.

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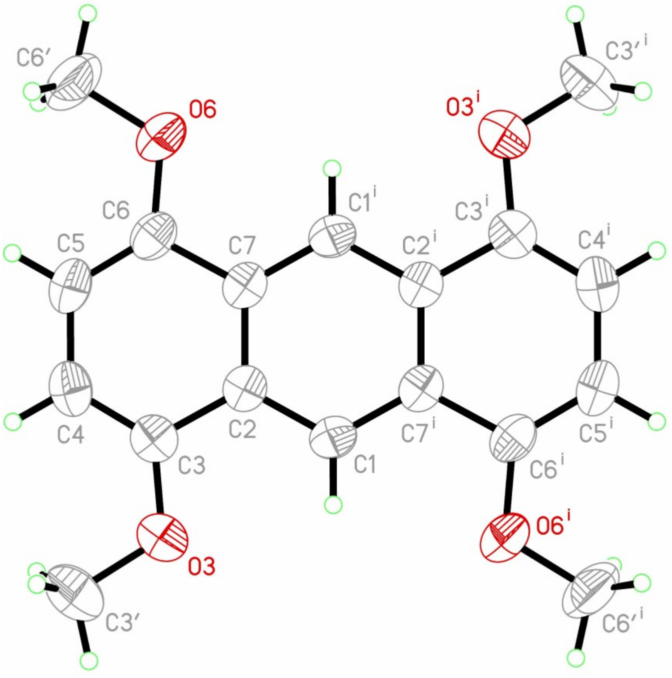

Figure 1
The molecular structure of the title compound, with displacement ellipsoid plot shown at the 50% probability level, viewed perpendicular to the mean plane [symmetry code: (i) $2 - x, -y, 1 - z$].

Experimental

The title compound was prepared from bromo-2,5-dimethoxybenzene, using a synthesis developed by Fleming & Mah (1975) and refined by Fitzgerald *et al.* (1992). Their method is a one-step synthesis, using 2,2,6,6-tetramethylpiperidine in refluxing tetrahydrofuran, and provides moderate yields.

Crystal data

$C_{18}H_{18}O_4$
$M_r = 298.32$
Monoclinic, $P2_1/n$
$a = 11.1715$ (9) Å
$b = 5.9994$ (5) Å
$c = 11.7159$ (10) Å
$\beta = 109.961$ (2)°
$V = 738.05$ (11) Å^3
$Z = 2$
$D_x = 1.342$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2419 reflections
$\theta = 3.1$–27.4°
$\mu = 0.09$ mm^{-1}
$T = 298$ (2) K
Plate, clear pale yellow
0.41 × 0.27 × 0.11 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: none
6951 measured reflections
1696 independent reflections
1159 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\max} = 27.5$°
$h = -14 \rightarrow 14$
$k = -7 \rightarrow 7$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.140$
$S = 1.01$
1696 reflections
100 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}

H atoms were located in difference Fourier maps, then positioned geometrically and allowed to ride on their respective parent atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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