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9,10-Bis(3-hydroxy-3,3-diphenylprop-1-ynyl)-anthracene *N*,*N*′-dimethylformamide solvate

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Key indicators

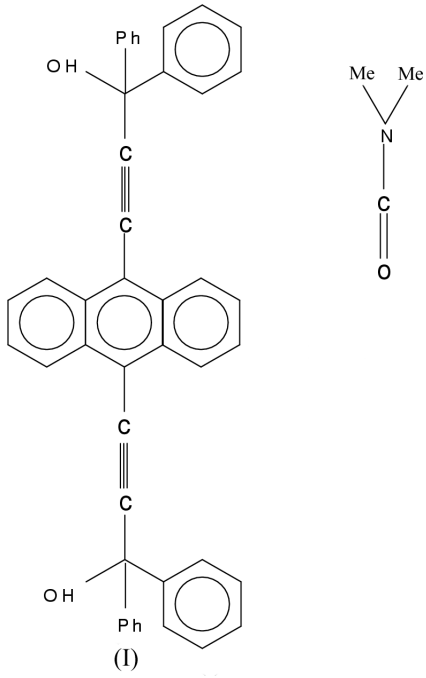
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.176
Data-to-parameter ratio = 14.9

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

The X-ray crystal structure of the title compound, $C_{47}H_{37}NO_3$, has been determined. The zigzag packing of the molecules is stabilized by intermolecular O—H···O hydrogen bonds between the host and guest molecules, and intramolecular C—H···O interactions.

Comment

Knowledge of the host–guest interactions of tertiary acetylenic alcohol containing compounds crystallized with small molecules like ketones, amines and sulfoxides are useful in preparing optically active tertiary acetylenic alcohols (Toda *et al.*, 1981, 1991; Johnson *et al.*, 1992). We have prepared the novel title compound, (I), based on diphenylhydroxypropyne and anthracene units and cocrystallized with DMF, since DMF is known to be a good proton acceptor (Weber *et al.*, 1991).



(I)

The C*sp*—C*sp* bond lengths [Fig. 1; C15—C16 and C30—C31 1.190 (2) and 1.187 (2) Å, respectively] are comparable with the literature values (Venkataraman *et al.*, 1997). The bond angles at C30 and C31 [C9—C30—C31 = 176.5 (2)° and C30—C31—C32 = 175.8 (2)°] are slightly but significantly larger than the angles at C15 and C16 [C10—C15—C16 = 173.3 (2)° and C15—C16—C17 = 170.6 (2)°]. The bond lengths [N1*A*—C1*A* and C1*A*—O1*A* 1.367 (5) and 1.225 (5) Å, respectively] and the sum of bond angles around N1*A* (360.0°) in the guest molecule (DMF) indicate that the N1*A* atom is in

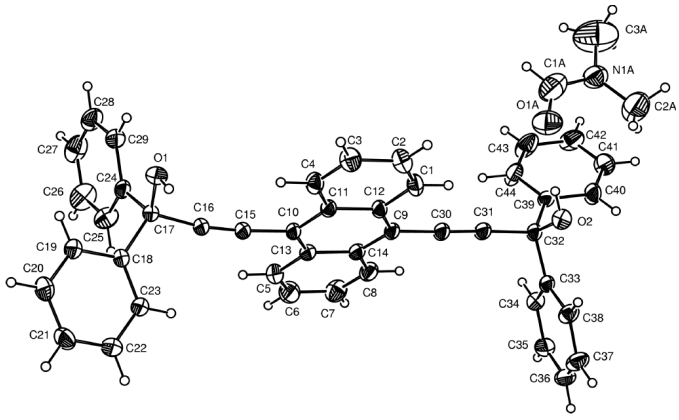


Figure 1
The molecular structure of (I) showing displacement ellipsoids at the 30% probability level.

the sp^2-hybridized state and hence there is a slight delocalization of electrons in the N1*A*—C1*A*—O1*A* unit.

The complete zigzag packing of the crystal is mainly stabilized by O—H···O hydrogen-bonding interactions of the DMF molecule with the two hydroxyl groups of the host molecule. In addition, atom O1 is involved, as acceptor, in a C—H···O-type interaction.

Experimental

Single crystals were obtained by slow evaporation from a solution of the compound in DMF.

Crystal data

$C_{47}H_{37}NO_3$
M_r = 663.78
Monoclinic, $P2_1/c$
a = 16.290 (3) Å
b = 11.837 (2) Å
c = 19.426 (4) Å
β = 104.11 (3)°
V = 3632.8 (12) Å^3
Z = 4
D_x = 1.214 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 5–20°
μ = 0.59 mm^{-1}
T = 293 (2) K
Rectangular, yellow–brown
0.53 × 0.40 × 0.23 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
7151 measured reflections
6910 independent reflections
5927 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 70.1°
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 14$
$l = -23 \rightarrow 22$
3 standard reflections
every 200 reflections
frequency: 60 min
intensity decay: <1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.176
S = 1.08
6910 reflections
465 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0989P)^2 + 1.1549P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.062
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0049 (4)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1*A*	0.82	2.06	2.863 (3)	168
C29—H29···O1	0.93	2.35	2.704 (3)	102
C38—H38···O2	0.93	2.33	2.689 (2)	102
C2A—H2*A*1···O1*A*	0.96	2.36	2.747 (5)	104
O1—H1···O1*A*^{i}	0.82	2.04	2.865 (3)	180
C2—H2*A*···O1^{ii}	0.93	2.46	3.292 (2)	149

Symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

H atoms were fixed geometrically at calculated positions, each riding on the parent atom with an isotropic displacement parameter 1.2 (1.5 for methyl H atoms) times that of the parent atom.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *PARST* (Nardelli, 1995).

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