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Methyl 2,2′,7′-trihydroxy-1,1′-binaphthalene-3-carboxylate acetone solvate

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In the title compound, $C_{22}H_{16}O_5$, the two naphthyl units are approximately perpendicular. The 3-naphthylcarboxyl group and the *ortho*-hydroxyl group are involved in an intermolecular O—H···O hydrogen bond with O···O = 2.606 (2) Å. Molecules related by a glide plane are linked by an intermolecular O—H···O hydrogen bond with O···O = 2.747 (2) Å, creating a spiral. A solvent molecule is an acceptor in an O—H···O hydrogen bond with O···O = 2.700 (2) Å.

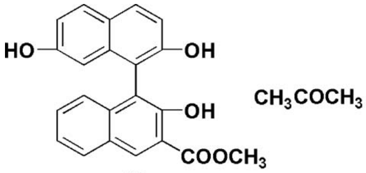
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.119
Data-to-parameter ratio = 14.6

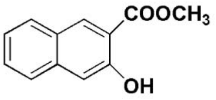
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

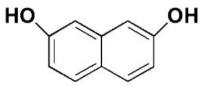
1,1′-Binaphthol and its derivatives are used as chiral auxiliaries in stereoselective organic synthesis (Nishizawa *et al.*, 1981; Noyori *et al.*, 1979; Naruse *et al.*,1988; Hesemann & Moreau, 2003; Teo *et al.*, 2005; Guo *et al.*, 2006). These compounds can be prepared by various methods (Feringa & Wynberg, 1977; Toda *et al.*, 1989; Nakajima *et al.*, 1999; Xin *et al.*, 2002). However, oxidative coupling of 2-naphthols is the most direct procedure.



(I)



(II)



(III)

In the present work, compound (I) was obtained from cross-coupling compounds (II) and (III) under catalysis of the $CuCl_2·2H_2O$ complex of tetramethylethylenediamine (TMEDA) (1:2) in methanol. The cross-coupling reaction took place at room temperature. The molecule of (I) in the crystal structure reveals intramolecular hydrogen bonding (Fig. 1 and Table 1). Details of intermolecular hydrogen bonds between the hydroxyl groups at O4 and O5, and between the O5 hydroxyl group and acetone (Table 1 and Fig. 2) illustrate their effect on the crystal packing. The relative orientation of the naphthyl ring systems is defined by the torsion angle C9—C10—C12—C21 of −102.9 (2)°. This overall conformation reduces repulsion between the O3 and O5 hydroxyl groups.

Experimental

Under the catalysis of the complex of $CuCl_2·2H_2O$-TMEDA (1:2) in methanol (15 ml), the cross-coupling of (II) (0.202 g) and (III) (0.160 g) afforded compound (I), which was purified through a short column of gel silica (eluted with petroleum ether–EtOAc, 4:1). It was crystallized from acetone (yield 88.4%; m.p. 519–521 K). Analysis

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calculated for $C_{22}H_{16}O_5$: C 73.33, H 4.48%; found: C 72.63, H 4.54.

Crystal data

$C_{22}H_{16}O_5 \cdot C_3H_6O$
$M_r = 418.43$
Monoclinic, $P2_1/c$
$a = 13.477$ (4) Å
$b = 12.062$ (4) Å
$c = 14.049$ (4) Å
$\beta = 109.223$ (6)°
$V = 2156.5$ (12) Å^3
$Z = 4$
$D_x = 1.289$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.40 × 0.30 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.957$, $T_{\max} = 0.971$
11034 measured reflections
4184 independent reflections
2707 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.119$
$S = 1.02$
4184 reflections
286 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.4103P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O5—H5A⋯O6	0.82	1.88	2.700 (2)	176
O3—H3A⋯O1	0.82	1.88	2.606 (2)	148
O4—H4⋯O5$^{\rm i}$	0.82	1.93	2.7466 (19)	171

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

H atoms were located in a difference map and constrained to ride on their parent atoms [distances 0.82 (OH), 0.93 (CH) and 0.96 Å (CH_3)], with $U_{\rm iso}$(H) = 1.2 (1.5 for methyl and hydroxyl) times $U_{\rm eq}$(C,O).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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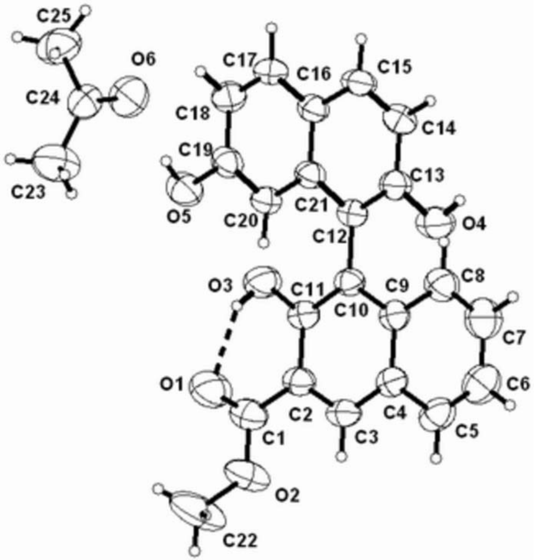

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

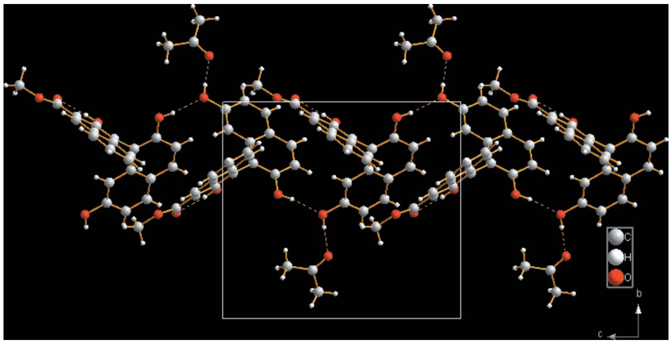

Figure 2
Packing diagram of (I), with hydrogen bonds shown as dashed lines.

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