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Racemic 1,1′-bi-2-naphthol dimethylformamide sesquisolvate: a redetermination

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.099
wR factor = 0.254
Data-to-parameter ratio = 13.3

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

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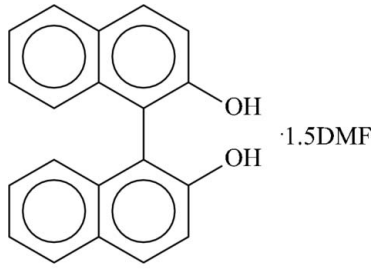
In the crystal structure of the title compound, $C_{20}H_{14}O_2\cdot1.5C_3H_7NO$, one of the hydroxyl groups forms a hydrogen bond to the dimethylformamide (DMF) molecule that lies on a general position and the other a hydrogen bond to the DMF molecule that lies on a twofold rotation axis. The naphthyl residues are almost perpendicular to each other.

Comment

A number of solvates and clathrates of 1,1′-bi-2-naphthol have been structurally verified, as noted from a search of the Cambridge Structural Database (Version 5.26; Allen, 2002). The reagent itself exists in the racemic and optically active (+) and (−) forms. The title 1.5DMF solvate, (I), was originally reported in space group *Cc* (Hirano *et al.*, 2003), but *PLATON* (Spek, 2003) suggests a centre of inversion in the crystal structure; this is confirmed in the present re-investigation of (I) (Fig. 1). In the higher-symmetric space group *C*2/*c*, one of the two DMF solvates is disordered over a twofold rotation axis.



(I)

The two aromatic systems of (I) enclose a dihedral angle of 88.4 (1)°. One of the hydroxy groups forms a hydrogen bond to the ordered DMF (which lies on a general position) and the other to the disordered DMF (which lies on a twofold rotation axis). Apart from these two hydrogen bonds (Table 1), there are no other important intermolecular interactions in the crystal structure. Racemic 1,1′-bi-2-naphthol crystallizes in the non-centrosymmetric space group *Iba*2 (Gridunova *et al.*, 1982; Mori *et al.*, 1993; Toda *et al.*, 1997, Nieger, 1999) and also has the two naphthol residues in an almost perpendicular orientation.

Experimental

Racemic 1,1′-bi-2-naphthol (0.29 g, 1 mmol) was dissolved in a mixed-solvent system of water (5 ml) and dimethylformamide (5 ml). This solution was added to an ethanol solution (10 ml) of 2-amino-pyrimidine (0.19 g, 2 mmol). Evaporation of the solvent over several

weeks gave colourless block-shaped crystals of (I). The diffraction measurements represent the best of six sets of measurements; the crystals were not strongly diffracting in any of the attempts.

Crystal data

$C_{20}H_{14}O_2\cdot1.5C_3H_7NO$	D_x = 1.222 Mg m^{-3}
M_r = 395.96	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 2122 reflections
a = 14.250 (1) Å	θ = 2.4–20.7°
b = 10.806 (1) Å	μ = 0.08 mm^{-1}
c = 28.126 (2) Å	T = 295 (2) K
β = 96.374 (1)°	Block, colourless
V = 4304.1 (6) Å^3	0.42 × 0.18 × 0.15 mm
Z = 8	

Data collection

Bruker APEX CCD area-detector diffractometer	3000 reflections with $I > 2\sigma(I)$
ω and φ scans	R_{int} = 0.029
Absorption correction: none	θ_{max} = 25.0°
11051 measured reflections	$h = -16 \rightarrow 16$
3790 independent reflections	$k = -12 \rightarrow 8$
	$l = -33 \rightarrow 33$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1082P)^2 + 6.5323P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.099	
$wR(F^2)$ = 0.254	
S = 1.18	$(\Delta/\sigma)_{max}$ = 0.001
3790 reflections	$\Delta\rho_{max}$ = 0.46 e Å^{-3}
286 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3	0.82	1.86	2.674 (5)	172
O2—H2···O4	0.82	1.96	2.781 (4)	174

One DMF molecule is disordered over a twofold rotation axis. A number of restraints were imposed on this molecule, as follows: C24—O4 = 1.25 (1), C24—N2 = 1.35 (1), N2—C25 = N2—C26 = 1.45 (1), O4···C25 = 2.71 (1), C24···C25 = C24···C26 = 2.43 (2) and C25···C26 = 2.51 (2) Å. The displacement parameters of these atoms were restrained to an approximate isotropic behaviour; furthermore, these atoms were restrained to lie in a common plane. H atoms were positioned geometrically (O—H = 0.82, C—$H_{aromatic}$ = 0.93 and C—H_{methyl} = 0.96 Å), and were included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C,O), or 1.5U_{eq}(C) for methyl groups.

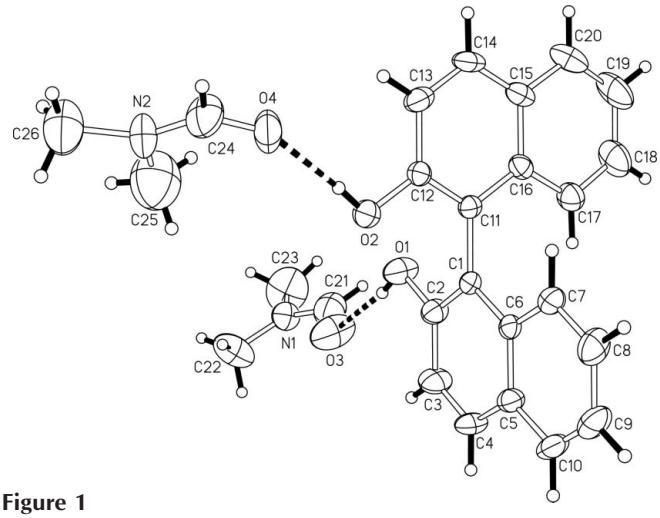


Figure 1
A plot of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii. Dashed lines indicate hydrogen bonds. Only one disorder component is shown.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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