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Hydrogen Bonding in the 1:1 Adduct of 1,10-Phenanthroline and 1,1′-Binaphthyl-2,2′-diol

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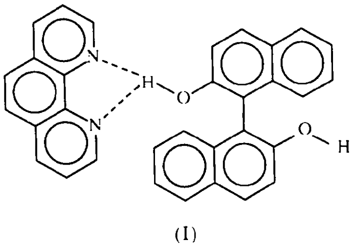
Abstract

The title compound, $C_{12}H_8N_2.C_{20}H_{14}O_2$, was obtained as a 1:1 adduct from non-aqueous solutions of the two components. The lattice contains centrosymmetric dimers in which O—H···O and O—H···N hydrogen bonding is important. Rotation about the C—C′ bond of the binaphthyl moiety produces a torsion angle of −85.5 (2)°.

Comment

The title compound, (I), was identified initially during work on the direct electrochemical synthesis of metal derivatives of 1,1′-binaphthyl-2,2′-diol (binaphthol) and related enolic compounds (García-Martínez & Tuck, 1997). It was subsequently shown that the same material could be produced by the evaporation of equimolar solutions in various non-aqueous solvents.



(I)

The structure of the adduct is shown in Fig. 1. Selected geometric parameters are given in Table 1 and hydrogen-bonding details are given in Table 2. Crystals were obtained from three different solvents (CH_3OH, C_2H_5OH and CH_2Cl_2) and, in each case, the cell parameters were measured, showing that the three sets of crystals were identical within experimental error.

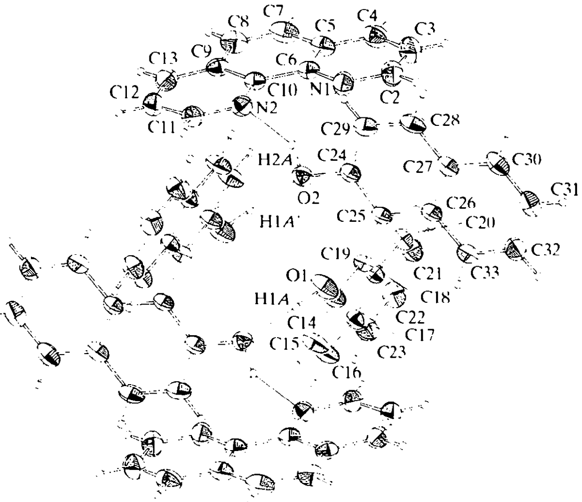


Fig. 1. *ZORTEP* (Zsolnai, 1994) diagram of $C_{12}H_8N_2.C_{20}H_{14}O_2$ showing the numbering system. Atoms are represented as displacement ellipsoids drawn at the 30% probability level.

The structures of binaphthol and its derivatives have been of interest because of the use of such molecules in the optical resolution of asymmetric species (Cram & Cram, 1979). The structures of both the (+)-(*R*)- and racemic forms have been reported by Mori *et al.* (1993). Immediate points of comparison with the title compound are the (O)C—C—C—C(O) torsion angles, which are −76.731 and −88.266°, respectively, in the (+)-(*R*)- and racemic molecules [data obtained from the Cambridge Structural Database (Allen & Kennard, 1993)], and −85.5 (2)° (C14—C19—C25—C24) for the binaphthol moiety in the present case, but in the parent binaphthol structures, the OH groups are *cis*, in contrast to the quasi-*trans* mode in the title structure. This is undoubtedly related to the change in the type of hydrogen bonding, which in both forms of the parent binaphthol is intermolecular (O1—H···O2). In the title compound, we observe two different hydrogen-bonding interactions, both intermolecular. In one mode, illustrated in Fig. 1, binaphthol molecules are linked by O—H···O2′ bonds of length 2.765 (2) Å, compared with values of 2.961 (4) and 2.852 (3) Å in the two forms of the parent substance.

There is also evidence of hydrogen bonding between O2—H and the N atoms of 1,10-phenanthroline, with O2···N1 3.144 (2) and O2···N2 2.719 (2) Å, so that the latter interaction is clearly the stronger. This important O—H···N bonding is in contrast to the case of the 1:1:1 complex of (*R*)-(+)-binaphthol, *N*,*N*,*N*′,*N*′-tetrakis(2-butyl)terephthalimide and methanol, where the three components are alternatively linked by O—H···O distances of 2.68 (1), 2.67 (1) and 2.59 (1) Å (Toda *et al.*, 1986).

It is clear that the crystal structure of the title compound, and the conformation of the binaphthol entity in particular, is largely governed by these hydrogen-bonding interactions.

Experimental

1,10-Phenanthroline (50 mg, 0.28 mmol) and binaphthol (40 mg, 0.14 mmol) were dissolved in methanol, ethanol or dichloromethane (25 ml) with warming. Slow evaporation gave colourless crystals suitable for X-ray analysis.

Crystal data

$C_{12}H_8N_2.C_{20}H_{14}O_2$
$M_r = 466.52$
Triclinic
$P\overline{1}$
$a = 10.8768 (15)$ Å
$b = 11.238 (2)$ Å
$c = 11.6786 (8)$ Å
$\alpha = 97.929 (8)^\circ$
$\beta = 110.895 (7)^\circ$
$\gamma = 109.635 (9)^\circ$
$V = 1201.0 (3)$ Å^3
$Z = 2$
$D_x = 1.290$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 9–42^\circ$
$\mu = 0.640$ mm^{-1}
$T = 293 (2)$ K
Prism
0.25 × 0.25 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5148 measured reflections
4896 independent reflections
2916 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.026$
$\theta_{\rm max} = 74.30^\circ$
$h = -12 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = -14 \rightarrow 0$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.129$
$S = 1.016$
4896 reflections
354 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.1363P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.177$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.139$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0064 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C2	1.319 (3)	O1—C14	1.365 (2)
N1—C6	1.356 (3)	O2—C24	1.372 (2)
N2—C11	1.324 (3)	C14—C19	1.372 (2)
N2—C10	1.358 (2)	C14—C15	1.409 (3)
C2—C3	1.394 (3)	C18—C19	1.427 (2)
C5—C6	1.407 (3)	C19—C25	1.492 (2)
C6—C10	1.446 (3)	C24—C25	1.376 (3)
C9—C10	1.407 (3)	C24—C29	1.407 (3)
C11—C12	1.387 (3)	C25—C26	1.426 (3)
C2—N1—C6	117.2 (2)	N2—C10—C6	118.49 (18)
C11—N2—C10	117.57 (19)	N2—C11—C12	124.3 (2)
N1—C2—C3	124.0 (3)	O1—C14—C19	118.01 (16)
N1—C6—C5	123.4 (2)	O1—C14—C15	120.69 (17)
N1—C6—C10	117.73 (18)	O2—C24—C25	118.64 (16)
N2—C10—C9	122.05 (19)	O2—C24—C29	119.91 (19)
C18—C19—C25—C26	−86.5 (2)	C14—C19—C25—C26	95.1 (2)
C18—C19—C25—C24	92.8 (2)	C14—C19—C25—C24	−85.5 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2′	0.96 (3)	1.81 (3)	2.765 (2)	174 (2)
O2—H2*A*···N1	0.94 (3)	2.48 (3)	3.144 (2)	128 (2)
O2—H2*A*···N2	0.94 (3)	1.87 (3)	2.719 (2)	149 (2)

Symmetry codes: (i) $1 - x, 2 - y, 2 - z$.

H atoms bound to atoms O1 and O2 (H1*A* and H2*A*) were refined isotropically, while all other H atoms were refined as riding (*SHELXL*97 *AFIX*43 label; Sheldrick, 1997).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1995). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *HELENA* (Spek, 1996). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97. Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1399). Services for accessing these data are described at the back of the journal.

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8-Chloro-5-(4-chlorophenyl)-5*H*-indeno[1,2-*d*]pyrimidine

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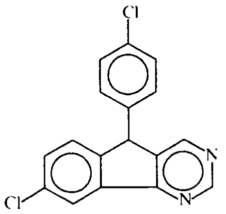
Abstract

The title compound, $C_{17}H_{10}Cl_2N_2$, crystallizes with two independent molecules in the asymmetric unit, with little conformational difference between them. The molecules have two planar components and the angle between the two planes is 95.5° in molecule I and 97.3° in molecule II. The crystal structure exhibits only van der Waals interactions. The three-dimensional structure and folding of the two independent molecules in the asymmetric unit are reminiscent of other P450 aromatase inhibitors.

Comment

The conversion of steroidal androgens (C_{19} compounds containing a methyl group at the C-19 position) to steroidal estrogens (C_{18} compounds with an aromatic *A* ring) in humans is catalyzed in three steps by aromatase (P450 arom, CYP19). Steroidal and non-steroidal inhibitors of these reactions are used clinically to treat estrogen-dependent disorders. Among the most potent non-steroidal inhibitors are aminogluthemide (Mason *et al.*, 1987), fadrozole (Furet *et al.*, 1993), vorozole (De Coster *et al.*, 1990; Peeters *et al.*, 1993), letrozole (Bhatnagar *et al.*, 1990) and anastrozole (Dukes *et al.*, 1996). The title compound, LY113174, is part of a family of substituted pyrimidines (Taylor *et al.*, 1987; Jones *et al.*, 1990) that show varying degrees of inhibitory activity towards aromatase (Hirsch *et al.*, 1987). The structure determination of LY113174 was carried out in order to characterize this aromatase inhibitor of known activity.



LY113174

Perspective views showing the atomic numbering schemes for the two independent molecules, both with the *S* configuration at the chiral center, are given in Fig. 1. The bond lengths and angles of the two molecules are similar and a search of the April 1997 version of the Cambridge Structural Database (Allen *et al.*, 1979) for the three-ring moiety produced no hits. A least-squares superposition of the two molecules shows only a slight variation in the two conformations. The angle between the two planar components of the twisted molecules is 95.5° for molecule I and 97.3° for molecule II (this is illustrated in a figure deposited with the supplementary material). Relevant torsion angles which help to illuminate the three-dimensional twist in the title compound are given in Table 1, the first four values referring to the first molecule and second four giving the corresponding parameters for the second independent molecule.

The two principal molecular planes, *i.e.* the tricyclic plane and the phenyl plane, contain essentially all the atoms. In molecule I, the atom that is furthest from the tricyclic plane (containing atoms C7–C17, N1 and N2) is C17 at a distance of −0.021 (3) Å. The Cl2 atom is in the tricyclic plane and the Cl1 atom is in the plane defined by atoms C1–C6. For molecule II, the two atoms showing the greatest deviation from the tricyclic plane are C107 and N102 at distances of 0.033 (3) and −0.023 (3) Å, respectively. The Cl atoms, Cl101 and Cl102, do not deviate from their respective planes. The *p*-Cl atoms cause the expected increase in the ring angle in which the central atom is that bonded to the chlorine and a decrease in the two adjacent ring angles (Brisse & Sygusch, 1974; Domenicano & Murray-Rust, 1979) from 120°.

There is only one intermolecular contact per molecule that is less than the sum of the van der Waals radii of the atoms. In molecule I, atoms Cl1 and Cl2($x, y, z+1$) are 3.660 (1) Å apart, while in molecule II, atoms Cl101 and Cl102($x, y, z+1$) are 3.840 (1) Å apart.