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Crystal structure of (*R*)-1,1′-bi-2-naphthol 1,3-bis(pyrid-4-yl)propane, $C_{20}H_{14}O_2 \cdot C_{13}H_{14}N_2$

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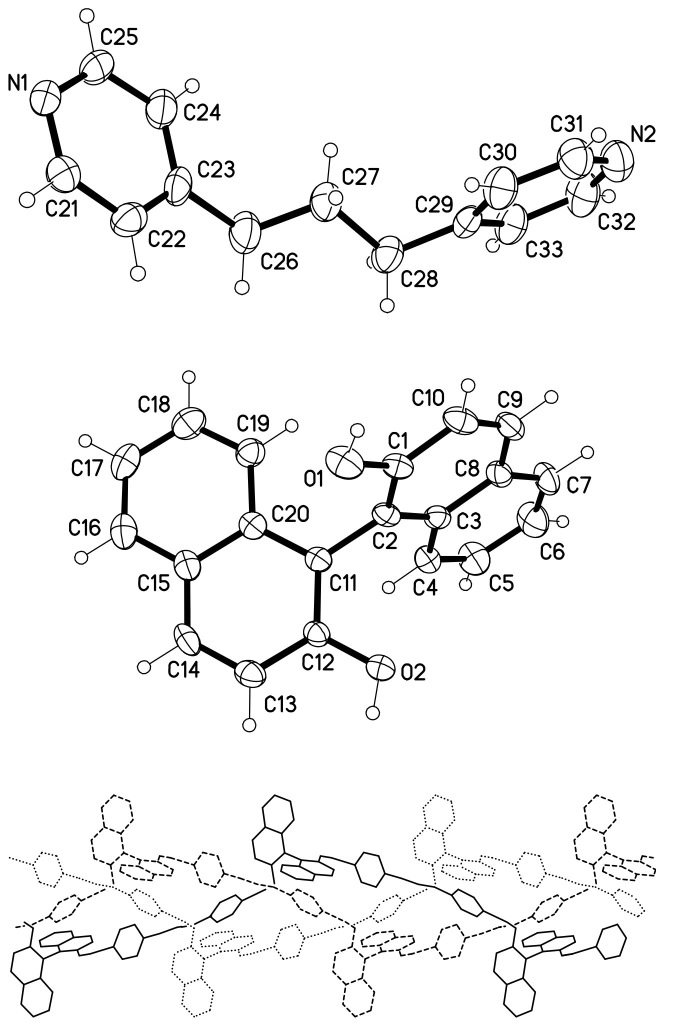

Abstract

$C_{33}H_{28}N_2O_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 10.106(1) Å, b = 11.501(1) Å, c = 22.425(2) Å, V = 2606.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.062, $wR_{ref}(F^2)$ = 0.124, T = 298 K.

Source of material

(*R*)-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 1,3-bis(paired-4-yl)propane (0.20 g, 1 mmol). Evaporation of the solvent over six weeks gave colorless block-shaped crystals of the title compound.

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Experimental details

While the hydrogen atoms attached to carbon atoms were placed on calculated positions, the hydroxy H atoms were refined freely due to their importance in intermolecular hydrogen bonding.

Discussion

1,3-bis(paired-4-yl)propane, a spacer molecule, not only acts as a bridged ligand in the preparation of metal complexes [1,2], but also as a kind of proton receptor in the construction of anionic networks [3]. Consequently, much interest has been focused on its complexes or ion-pair compounds. On the other hand, 1,1′-bi-2-naphthol (binaphthol) is usually used as the precursor for asymmetric catalysis, hosts for molecular recognition and enantiomer separation, and also as intermediates for the synthesis of chiral materials [4-6]. However, to our knowledge, the crystal structure of the 1:1 complex of 1,1′-bi-2-naphthol and 1,3-bis(paired-4-yl)-propane has never been reported so far.

The crystal structure of the title compound (figure, top & middle; 50 % probability displacement ellipsoids) consists of one 1,1′-bi-2-naphthol molecule and 1,3-bis(paired-4-yl)propane molecule. The C1—O1 and C12—O2 bond lengths of 1.358(4) Å and 1.355(4) Å, respectively, are nearly equal to each other, and the two naphthyl rings are not in the same plane with the dihedral angle of 79.17(5)°. Moreover, the two pyridyl conjugated rings can be confirmed by the distances of N1—C21, C25—N1, C21—C22, C22—C23, C23—C24, C24—C25 and C29—C30, C29—C33, C30—C31, C33—C32, C31—N2, C32—N2, respectively, with 1.313(4) Å – 1.386(5) Å which are between single bond and double bond. Furthermore, the two neutral pyridyl rings are not in the same plane with the dihedral angle of 82.6(1)°. The 1,1′-bi-2-naphthol and 1,3-bis(paired-4-yl)propane molecules are interlinked by hydrogen bonds of O1–H1···N2 [2.691(4) Å] and O2–H2···N1^{i} [2.789(5) Å] (i: –x+2,y+½,–z+½), forming helical triple-chains by three entwined strings along [010] direction (figure, bottom; viewing direction [101]).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.12 × 0.17 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.77 cm^{-1}
Diffractometer, scan mode:	Bruker APEX SMART CCD, φ/ω
$2\theta_{max}$:	48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12711, 4100
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3380
$N(param)_{refined}$:	342
Programs:	SHELXS-97 [7], SHELXL-97 [8], ORTEP-II [9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.949(4)	0.331(3)	0.170(2)	0.08(1)
H(2)	4*a*	0.694(4)	0.757(3)	0.182(2)	0.09(1)
H(4)	4*a*	0.5626	0.5849	0.0454	0.062
H(5)	4*a*	0.3531	0.5470	0.0151	0.080
H(6)	4*a*	0.2392	0.3865	0.0493	0.089
H(7)	4*a*	0.3400	0.2589	0.1125	0.078
H(9)	4*a*	0.5421	0.1939	0.1656	0.066
H(10)	4*a*	0.7559	0.2222	0.1916	0.066
H(13)	4*a*	0.8530	0.8380	0.1252	0.063
H(14)	4*a*	1.0051	0.8184	0.0511	0.067
H(16)	4*a*	1.1181	0.6987	–0.0250	0.076
H(17)	4*a*	1.1443	0.5247	–0.0714	0.084
H(18)	4*a*	1.0157	0.3673	–0.0441	0.080
H(19)	4*a*	0.8618	0.3824	0.0294	0.064

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(21)	4*a*	1.4818	–0.5745	0.2791	0.077
H(22)	4*a*	1.4982	–0.4207	0.2162	0.078
H(24)	4*a*	1.1110	–0.4462	0.1889	0.086
H(25)	4*a*	1.1065	–0.5976	0.2551	0.082
H(26A)	4*a*	1.4078	–0.2976	0.1433	0.105
H(26B)	4*a*	1.2774	–0.3407	0.1128	0.105
H(27A)	4*a*	1.2791	–0.1864	0.2090	0.082
H(27B)	4*a*	1.1530	–0.2235	0.1730	0.082
H(28A)	4*a*	1.3635	–0.0874	0.1263	0.079
H(28B)	4*a*	1.2476	–0.1331	0.0862	0.079
H(30)	4*a*	1.2223	0.0021	0.2296	0.084
H(31)	4*a*	1.1008	0.1633	0.2480	0.092
H(32)	4*a*	1.0110	0.1955	0.0818	0.100
H(33)	4*a*	1.1328	0.0351	0.0578	0.085

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*a*	0.9077(3)	0.3981(2)	0.1686(1)	0.062(2)	0.052(2)	0.079(2)	0.003(1)	–0.021(2)	0.004(1)
O(2)	4*a*	0.6940(2)	0.6842(2)	0.1679(1)	0.063(2)	0.043(1)	0.068(2)	–0.008(1)	0.017(1)	–0.014(1)
C(1)	4*a*	0.7808(3)	0.3792(3)	0.1512(1)	0.051(2)	0.040(2)	0.047(2)	–0.003(2)	–0.006(2)	–0.003(2)
C(2)	4*a*	0.7203(3)	0.4611(2)	0.1161(1)	0.052(2)	0.035(2)	0.034(2)	0.000(2)	–0.003(2)	–0.004(1)
C(3)	4*a*	0.5882(3)	0.4423(3)	0.0980(1)	0.050(2)	0.036(2)	0.040(2)	–0.001(2)	0.001(2)	–0.004(2)
C(4)	4*a*	0.5202(3)	0.5182(3)	0.0591(2)	0.052(2)	0.047(2)	0.057(2)	–0.005(2)	0.002(2)	0.004(2)
C(5)	4*a*	0.3944(4)	0.4960(3)	0.0413(2)	0.058(2)	0.070(2)	0.072(3)	0.000(2)	–0.014(2)	0.013(2)
C(6)	4*a*	0.3260(4)	0.3993(4)	0.0614(2)	0.049(2)	0.077(3)	0.097(3)	–0.015(2)	–0.014(2)	0.001(3)
C(7)	4*a*	0.3863(4)	0.3233(3)	0.0987(2)	0.059(2)	0.059(2)	0.078(3)	–0.023(2)	0.007(2)	–0.002(2)
C(8)	4*a*	0.5190(3)	0.3408(3)	0.1170(1)	0.057(2)	0.042(2)	0.046(2)	–0.010(2)	0.004(2)	–0.008(2)
C(9)	4*a*	0.5859(4)	0.2605(3)	0.1527(2)	0.069(2)	0.042(2)	0.054(2)	–0.014(2)	0.006(2)	–0.000(2)
C(10)	4*a*	0.7126(4)	0.2778(3)	0.1687(2)	0.078(3)	0.042(2)	0.045(2)	–0.001(2)	–0.009(2)	0.007(2)
C(11)	4*a*	0.7973(3)	0.5652(2)	0.0962(1)	0.039(2)	0.037(2)	0.043(2)	0.000(2)	–0.007(2)	0.002(2)
C(12)	4*a*	0.7831(3)	0.6723(3)	0.1231(1)	0.039(2)	0.036(2)	0.050(2)	–0.004(2)	–0.005(2)	–0.000(2)
C(13)	4*a*	0.8626(3)	0.7667(3)	0.1061(2)	0.051(2)	0.041(2)	0.065(2)	–0.001(2)	–0.006(2)	0.001(2)
C(14)	4*a*	0.9537(3)	0.7547(3)	0.0619(2)	0.052(2)	0.043(2)	0.072(2)	–0.014(2)	–0.001(2)	0.014(2)
C(15)	4*a*	0.9718(3)	0.6487(3)	0.0322(2)	0.043(2)	0.049(2)	0.055(2)	–0.004(2)	–0.003(2)	0.010(2)
C(16)	4*a*	1.0662(3)	0.6356(4)	–0.0138(2)	0.053(2)	0.075(3)	0.062(2)	–0.006(2)	0.006(2)	0.013(2)
C(17)	4*a*	1.0817(4)	0.5327(4)	–0.0412(2)	0.058(2)	0.091(3)	0.061(2)	0.007(2)	0.014(2)	0.001(2)
C(18)	4*a*	1.0043(4)	0.4381(4)	–0.0247(2)	0.067(2)	0.074(3)	0.059(2)	0.005(2)	0.001(2)	–0.011(2)
C(19)	4*a*	0.9122(3)	0.4471(3)	0.0192(2)	0.051(2)	0.056(2)	0.053(2)	–0.000(2)	0.002(2)	–0.005(2)
C(20)	4*a*	0.8920(3)	0.5530(3)	0.0496(1)	0.041(2)	0.044(2)	0.045(2)	0.002(2)	–0.009(2)	0.004(2)
N(1)	4*a*	1.2943(3)	–0.6022(2)	0.2735(1)	0.058(2)	0.047(2)	0.081(2)	0.001(2)	0.004(2)	0.008(2)
N(2)	4*a*	1.0424(3)	0.1963(3)	0.1676(2)	0.074(2)	0.064(2)	0.096(3)	0.009(2)	–0.013(2)	–0.003(2)
C(21)	4*a*	1.4050(4)	–0.5480(3)	0.2608(2)	0.057(2)	0.065(2)	0.071(3)	0.009(2)	–0.003(2)	0.003(2)
C(22)	4*a*	1.4164(4)	–0.4556(3)	0.2226(2)	0.071(3)	0.058(2)	0.066(2)	–0.011(2)	0.007(2)	–0.004(2)
C(23)	4*a*	1.3075(4)	–0.4156(3)	0.1943(2)	0.086(3)	0.034(2)	0.060(2)	0.001(2)	0.006(2)	–0.005(2)
C(24)	4*a*	1.1889(4)	–0.4708(3)	0.2070(2)	0.072(3)	0.050(2)	0.094(3)	0.001(2)	–0.008(2)	0.008(2)
C(25)	4*a*	1.1872(4)	–0.5622(3)	0.2467(2)	0.057(2)	0.050(2)	0.098(3)	–0.014(2)	0.001(2)	0.003(2)
C(26)	4*a*	1.3154(5)	–0.3157(3)	0.1504(2)	0.123(3)	0.056(2)	0.083(3)	0.019(3)	0.022(3)	0.019(2)
C(27)	4*a*	1.2472(4)	–0.2084(3)	0.1699(2)	0.086(3)	0.055(2)	0.065(2)	0.007(2)	–0.001(2)	0.008(2)
C(28)	4*a*	1.2703(4)	–0.1076(3)	0.1261(2)	0.082(3)	0.053(2)	0.064(2)	–0.002(2)	0.000(2)	0.013(2)
C(29)	4*a*	1.1911(3)	–0.0021(3)	0.1410(2)	0.058(2)	0.041(2)	0.057(2)	–0.004(2)	–0.004(2)	0.009(2)
C(30)	4*a*	1.1798(4)	0.0399(3)	0.1983(2)	0.083(3)	0.060(3)	0.067(3)	0.009(2)	–0.015(2)	0.013(2)
C(31)	4*a*	1.1063(4)	0.1370(4)	0.2089(2)	0.086(3)	0.072(3)	0.073(3)	0.009(3)	–0.004(2)	0.008(2)
C(32)	4*a*	1.0543(4)	0.1559(4)	0.1122(2)	0.092(3)	0.067(3)	0.091(3)	0.010(3)	–0.035(3)	0.013(3)
C(33)	4*a*	1.1271(4)	0.0589(3)	0.0973(2)	0.086(3)	0.060(2)	0.066(3)	0.002(2)	–0.017(2)	0.005(2)

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