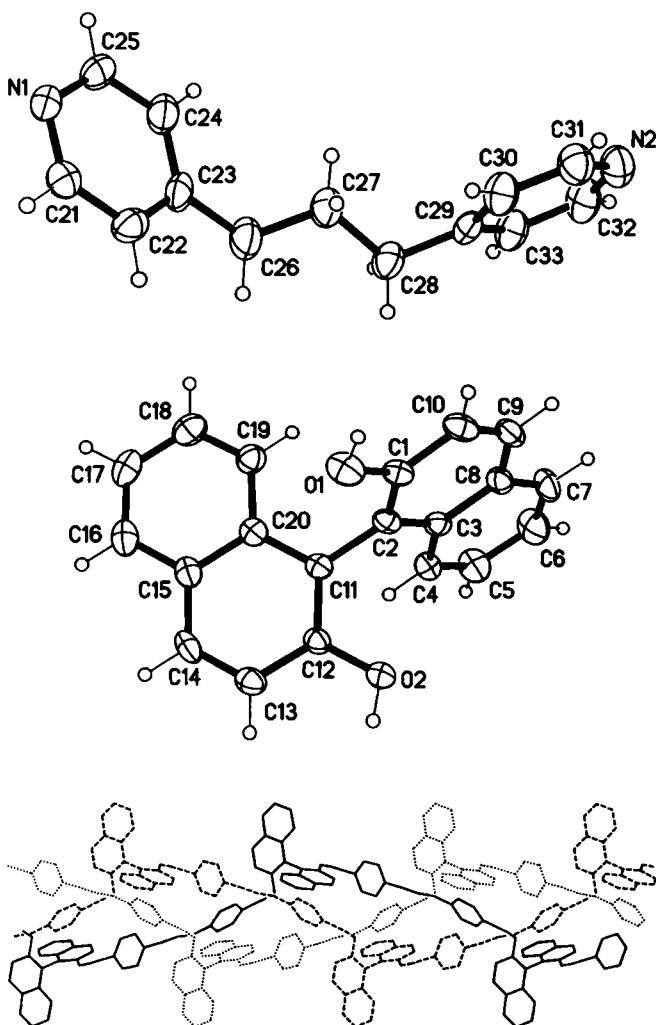


Crystal structure of (*R*)-1,1'-bi-2-naphthol 1,3-bis(pyrid-4-yl)propane, $C_{20}H_{14}O_2 \cdot C_{13}H_{14}N_2$

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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, $V = 2606.4$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.062$, $wR_{\text{ref}}(F^2) = 0.124$, $T = 298$ 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.

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...N1ⁱ [2.789(5) Å] ($i: -x+2, y+1/2, -z+1/2$), 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
Diffractionmeter, scan mode:	Bruker APEX SMART CCD, ϕ/ω
$2\theta_{\text{max}}$:	48°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	12711, 4100
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3380
$N(\text{param})_{\text{refined}}$:	342
Programs:	SHELXS-97 [7], SHELXL-97 [8], ORTEP-II [9]

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Table 2. Atomic coordinates and displacement parameters (in Å²).

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

Table 2. Continued.

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

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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)	4a	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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