NOTE

Synthesis and Supramolecular Structure of 2-Acetyl-1-naphthol

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2-Acetyl-1-naphthol with the molecular formula $C_{12}H_{10}O_2$, is stabilized by intramolecular O2-H2...O1 hydrogen bond forming a six-membered ring, nearly planar with the naphthone ring and the distances from C1 atom of acetyl group to mean plane of the three six-membered ring is 0.112(3) Å. Moreover, the structure is stabilized by intermolecular C-H... π and π - π stacking interactions.

Key Words: Naphthol, Synthesis, Supramolecular structure.

Naphthaldehyde, hydroxy-naphthaldehyde and their derivatives are an important class of intermediates¹⁻³ which condenses with primary amines to afford Schiff bases^{4,5} that are one of most versatile mixed-donor ligands in the field of coordination chemistry⁶⁻⁸. In this paper, we report on the synthesis and the X-ray single-crystal structure of the 2-acetyl-1-naphthol.

A sample of 2-methoxy naphthalene was obtained from Alfa Aesar and used without further purification. The other reagents and solvents were analytical grade reagents from Tianjin Chemical Reagent Factory. C, H and O analyses were carried out with a GmbH VariuoEL V3.00 automatic elemental analyzer. X-Ray single crystal structure was determined on a Bruker Smart 1000 CCD area detector. Melting points were measured by the use of a microscopic melting point apparatus made in Beijing Taike Instrument Limited Company and the thermometer was uncorrected.

Synthesis: A solution of 2-methoxy naphthalene (0.1 mmol), acetyl chloride (0.1 mmol) in 30 mL ethanol in the presence of $AlCl_3$ was refluxed for 5 h and then cooled to room temperature and filtered. Pale-yellow needle-like single crystals suitable for X-ray diffraction studies were obtained after several weeks by slow evaporation from a methanol-ethyl ether (1:3) mixed solution of the title compound.

X-Ray structure determination: The single crystal of the 2-acetyl-1-naphthol, with approximate dimensions of 0.55 mm $\times$ 0.50 mm $\times$ 0.38 mm was placed on a Bruker Smart 1000 diffractometer equipped with Apex CCD area detector. The diffraction data were collected using a graphite monochromated

MoK_{α} radiation ($\lambda = 0.71073$ Å) at 298(2) K. The structure was solved by using the program SHELXS-97 and Fourier difference techniques and refined by full-matrix least-squares method on F^2 using SHELXL-97. Details of the data collection and refinements of the title compound are given in Table-1. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added theoretically. CCDC: 712170.

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT
FOR THE 2-ACETYL-1-NAPHTHOL

Empirical formula	$C_{12}H_{10}O_2$
Formula weight	186.20
Temperature (K)	298(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$P 2_1/n$
Cell dimensions, (Å, deg)	$a = 7.6246(9)$, $b = 7.0312(7)$, $c = 17.4709(16)$, $\beta = 92.432(1)$
Volume (Å ³)	935.77(17)
Z	4
Density (calculated) (mg/m ³)	1.322
Absorption coefficient (mm ⁻¹)	0.089
$F_{(000)}$	392.0
Index ranges	$-9 \leq h \leq 8$, $-8 \leq k \leq 7$, $-20 \leq l \leq 19$
Reflections collected	4497/1643 [$R_{int} = 0.0454$]
Independent reflections	1494
Data/restraints/parameters	1643/0/129
Goodness of fit indicator	1.071
R [$I > 2\sigma(I)$]	$R_1 = 0.0421$, $wR_2 = 0.1332$
Largest diff. peak and hole (e Å ⁻³)	0.152 and -0.154

TABLE-2
SELECTED BOND DISTANCES (Å) AND ANGLES (°) FOR THE TITLE COMPOUND

Bond	Lengths	Bond	Lengths	Bond	Lengths
O1-C2	1.240(3)	C3-C8	1.429(3)	C7-C12	1.413(3)
O2-C3	1.341(2)	C4-C5	1.413(3)	C8-C9	1.413(3)
C1-C2	1.489(3)	C5-C6	1.352(3)	C9-C10	1.367(3)
C2-C4	1.467(3)	C6-C7	1.412(3)	C10-C11	1.379(3)
C3-C4	1.385(3)	C7-C8	1.408(3)	C11-C12	1.358(3)
Bond	Angles	Bond	Angles	Bond	Angles
O1-C2-C4	119.9(2)	C3-C4-C2	120.0(2)	C7-C8-C9	119.29(19)
O1-C2-C1	119.4(2)	C5-C4-C2	121.6(2)	C7-C8-C3	118.51(18)
C4-C2-C1	120.7(2)	C6-C5-C4	121.8(2)	C9-C8-C3	122.19(19)
O2-C3-C4	122.49(18)	C5-C6-C7	120.67(19)	C10-C9-C8	120.4(2)
O2-C3-C8	116.45(19)	C8-C7-C6	119.51(18)	C9-C10-C11	120.5(2)
C4-C3-C8	121.06(17)	C8-C7-C12	117.91(19)	C12-C11-C10	120.5(2)
C3-C4-C5	118.45(17)	C6-C7-C12	122.58(19)	C11-C12-C7	121.4(2)

X-Ray crystallographic analysis revealed the crystal structure of the title compound. And the structure is shown in Fig. 1. Selected bond distances and angles are listed in Table-2. The single crystal structure of the title compound is built up by only the C₁₂H₁₀O₂ molecule. An intramolecular O2-H2...O1 hydrogen bond between the hydroxyl group and the O atom of carbonyl group forms a six-membered ring in each molecule (Table-3), which is nearly coplanar with the naphthone ring and the distances from C1 atom of acetyl group to mean plane of the three six-membered ring is 0.112(3) Å. There are weak intermolecular π - π stacking interactions between neighbouring aromatic rings (C3-C8) with centroid-to-centroid distances of 3.844(2) Å (Fig. 2). Moreover, the structure is stabilized by intermolecular C-H... π stacking interactions.

Fig. 1. Molecule structure of the title compound with atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30 % probability level

TABLE-3
HYDROGEN BOND [Å, °] FOR THE TITLE COMPOUND

D-H...A	d(D-H)	d(H...A)	$\angle$ DHA	d(D...A)
O2-H2...O1	0.82	1.80	146	2.526(3)

Fig. 2. A perspective view of the intramolecular hydrogen-bond and π - π stacking interactions together with the centroid-centroid contacts

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