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Crystal structure of 2-(prop-2-yn-1-yloxy)-1-naphthaldehyde, $C_{14}H_{10}O_2$

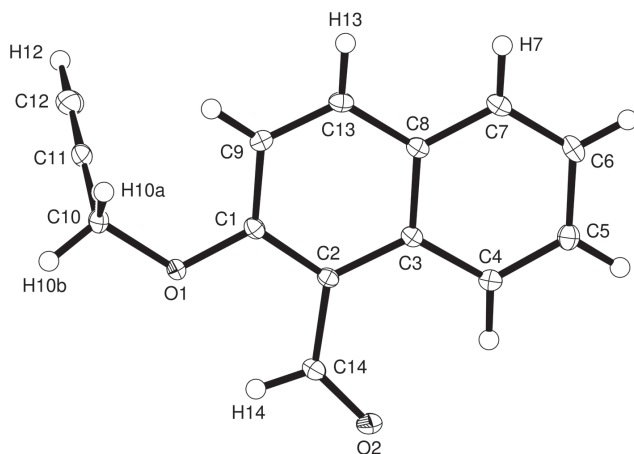


Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.27 × 0.23 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker D8, φ and ω -scans
θ_{\max} , completeness:	29.2°, >98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5714, 2698, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2331
$N(\text{param})_{\text{refined}}$:	146
Programs:	Bruker programs [1], SHELX [2, 3], ORTEP [4, 5]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.15277(10)	0.11219(10)	0.85144(8)	0.01354(18)
O2	0.00241(11)	0.64280(11)	0.81017(8)	0.01726(19)
C1	0.19716(13)	0.22884(14)	0.71450(11)	0.0107(2)
C2	0.15425(13)	0.42228(14)	0.69049(11)	0.0100(2)
C3	0.20416(13)	0.55085(14)	0.55046(10)	0.0100(2)
C4	0.17094(14)	0.74880(14)	0.51611(11)	0.0125(2)
H4	0.110732	0.802214	0.586957	0.015*
C5	0.22518(14)	0.86365(14)	0.38127(12)	0.0142(2)
H5	0.201613	0.995178	0.360677	0.017*
C6	0.31491(14)	0.78950(15)	0.27349(11)	0.0145(2)
H6	0.352464	0.870173	0.181493	0.017*
C7	0.34764(14)	0.59988(15)	0.30240(11)	0.0134(2)
H7	0.407337	0.549735	0.229621	0.016*
C8	0.29314(13)	0.47775(14)	0.44014(11)	0.0108(2)
C9	0.28052(13)	0.15940(14)	0.60232(11)	0.0120(2)
H9	0.304213	0.028900	0.619079	0.014*
C10	0.20014(15)	-0.08884(14)	0.88394(12)	0.0145(2)
H10A	0.148802	-0.108438	0.802586	0.017*
H10B	0.141261	-0.150300	0.978711	0.017*
C11	0.40391(15)	-0.17982(14)	0.89820(11)	0.0154(2)
C12	0.56916(16)	-0.25015(16)	0.90931(12)	0.0209(2)
H12	0.701089	-0.306298	0.918185	0.025*
C13	0.32691(13)	0.28170(14)	0.46922(11)	0.0122(2)
H13	0.383215	0.234142	0.394264	0.015*
C14	0.05512(14)	0.48134(14)	0.80993(11)	0.0128(2)
H14	0.028782	0.383891	0.896693	0.015*

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Abstract

$C_{14}H_{10}O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 7.498(3)$ Å, $b = 7.973(3)$ Å, $c = 9.660(4)$ Å, $\alpha = 67.211(13)^\circ$, $\beta = 84.489(14)^\circ$, $\gamma = 72.224(14)^\circ$, $V = 506.8(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0394$, $wR_{\text{ref}}(F^2) = 0.1135$, $T = 100(2)$ K.

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The figure above shows the title structure, indicating the atom numbering scheme. Thermal ellipsoids are drawn at the 50% probability level. Table 1 shows details of the crystal and the data collection, and Table 2 lists the atomic coordinates and displacement parameters.

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Source of materials

2-Hydroxy-1-naphthaldehyde (4 g) was mixed with 1.5 equivalent of anhydrous potassium carbonate in 60 mL of

N,N-dimethylformamide as aprotic solvent. The mixture was stirred for 15 min at room temperature before propargyl bromide (1.4 equivalent) was added. The activated mixture was heated to 40 °C using ultrasound sonication for 30 min. (Yield 77%, m.p. 113–115 °C). Colorless crystals were attained from cold ethanol. ¹H NMR (400 MHz, CDCl₃) 10.92 (1H, s, CHO), 9.30 (1H, d, *J* = 8.8 Hz, H-8), 8.08 (1H, d, *J* = 9.0 Hz, H-4), 7.81 (1H, d, *J* = 8.0 Hz, H-5), 7.65 (1H, m, *J* = 8.4, 5.6, 1.2 Hz, H-7), 7.47 (1H, dd, *J* = 8.0, 0.8 Hz, H-6), 7.40 (1H, d, *J* = 9.0 Hz, H-3), 4.96 (2H, d, *J* = 2.4 Hz, OCH₂), 2.61 (1H, t, *J* = 2.4 Hz). ¹³C NMR (100 MHz, CDCl₃) δ 192.00, 161.90, 137.31, 131.44, 129.91, 129.11, 128.26, 125.22, 125.11, 117.99, 113.99, 77.68, 76.82, 57.38. IR (Neat, cm⁻¹): 3250, 2894, 2117, 1651. **Elemental analysis** – Anal. Calcd. For C₁₄H₁₀O₂: C, 79.98%; H, 4.79%. Found: C, 79.74%; H, 4.56%.

Experimental details

Hydrogen atoms were included in idealized positions and ride on the parent atoms.

Comment

Naphthalene derivatives have attracted much attention as precursors for medicinal agents and dye-intermediates [6, 7]. 2-Hydroxy-1-naphthaldehyde is often used as an excellent functionalized fluorescent backbone for the synthesis of a variety of fluorescent chemosensors [8, 9]. Application of the Williamson reaction affords a very useful transformation in organic synthesis since the products have valuable industrial applications [10, 11]. Over decades, the increasing requirement for reaction acceleration and energy saving has developed modern process technologies and safer procedures for organic synthesis. For example, ultrasound technology has been applied in the alkylation reaction leading to a high degree of utilization of raw materials: it is practically wasteless [12]. Thus, the title compound reported in this study was prepared *via* a Williamson ether reaction using sonication in short time and good yield.

Our product, in which all bond lengths and angles, determined from an X-ray diffraction study, are within the normal ranges and in agreement with the data obtained by spectroscopic methods, including ¹H and ¹³C NMR, and IR and elemental analyses. The C10–C11≡C12 group is directed out of the aromatic ring mean-plane; the atoms C10, C11, and C12 are displaced 0.175 Å, 1.483 Å, and 2.512 Å respectively from that mean plane, and O1 and O2 are displaced 0.09 and 0.12 Å. The π–π stacking distance, measured as the perpendicular distance between the overlapping planes of the naphthalene groups is 3.415 Å [13]; the distance between the centroids of (C6, C5, C4, C3, C8, C7) and the neighbouring (C1, C9, C13,

C8, C3, C2) is 3.688 Å. The torsion angle around C1–O1–C10–C11 is 68.16° indicating a *gauche* conformation. The oxygen atoms, O1 and O2, differ in their intermolecular contacts; O2 is the acceptor of weak C–H···O hydrogen bonds from two neighboring molecules whereas O1 has no close contacts. Other short intermolecular contacts are at van der Waals' distances.

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