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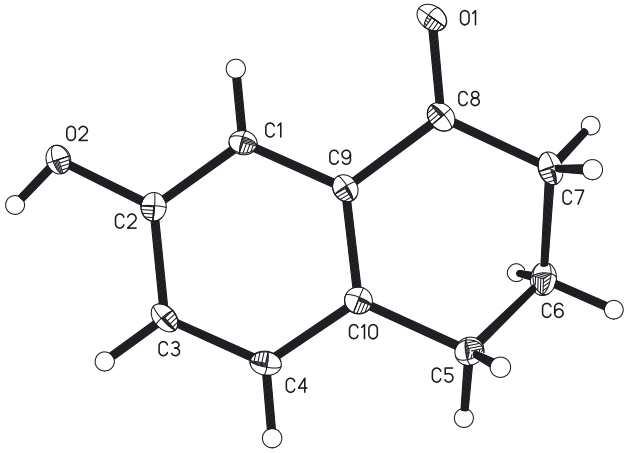
Crystal structure of 7-hydroxy-3,4-dihydronaphthalen-1(2*H*)-one, $C_{10}H_{10}O_2$



https://doi.org/10.1515/ncrs-2023-0353
Received July 30, 2023; accepted August 17, 2023;
published online September 5, 2023

Abstract

$C_{10}H_{10}O_2$, monoclinic, *C*2/*c* (no. 15), a = 16.6341(14) Å, b = 7.4319(4) Å, c = 14.8943(10) Å, β = 123.243(8)°, V = 1540.0(2) Å^3, Z = 8, $R_{gt}(F)$ = 0.0371, $wR_{ref}(F^2)$ = 0.0997, T = 293 K.

CCDC no.: 2275510

The crystal structure is shown in the Figure 1. Displacement ellipsoids are drawn at the 30 % probability level.

Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.13 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	SuperNova
θ_{max}, completeness:	25.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4935, 1427, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1246
$N(param)_{refined}$:	110
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3]

1 Source of material

The title compound was prepared according to a literature protocol [4, 5]. 7-Hydroxy-3,4-dihydronaphthalen-1(2*H*)-ones were prepared from hydroxy-substituted benzene and succinic anhydride in three steps: the Friedel–Crafts reaction, the Wolff–Kishner–Huang Minglong reduction reaction and the cyclization reaction. Firstly, to a stirred slurry of anhydrous $AlCl_3$ (6.65 g, 50 mmol) in 1,2-dichloroethane (10 mL), both succinic anhydride (0.50 g, 5 mmol) and phenol (0.47 g, 5 mmol) was added portionwise under nitrogen with stirring, while keeping the temperature at room temperature. The reaction solution was concentrated, and the residue was recrystallized from ethanol to obtain 4-(4-hydroxyphenyl)-4-oxobutanoic acid. Secondly, the resulting compound (0.75 g, 3.75 mmol) was added to a round bottom flask and DEG (10 mL), 85 % hydrazine hydrate (5 mL) and potassium hydroxide were added sequentially and the reaction mixture was refluxed at 453 K for 3 h to give 4-(4-hydroxyphenyl) butanoic acid. Lastly, the resulting compound (0.40 g, 2.25 mmol) was added to PPA (7.50 g, 22.5 mmol), and the mixture was stirred using a mechanical stirrer at 373 K for 30 min. The reaction product was extracted with chloroform, dried with anhydrous sodium sulfate, and concentrated to dryness to obtain 7-hydroxy-3,4-dihydronaphthalen-1(2*H*)-ones.

2 Experimental details

The H atoms were placed in idealized positions and treated as riding on their parent atoms, with d(C–H) = 0.97 Å (methylene), U_{iso}(H) = 1.2U_{eq}(C), and d(C–H) = 0.93 Å (aromatic), U_{iso}(H) = 1.2U_{eq}(C).

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.11021 (10)	0.55147 (19)	0.32568 (11)	0.0176 (3)
H1	0.094016	0.451346	0.350025	0.021*
C2	0.11924 (10)	0.5356 (2)	0.23934 (12)	0.0182 (3)
C3	0.14628 (10)	0.6856 (2)	0.20546 (12)	0.0215 (4)
H3	0.153846	0.675683	0.148246	0.026*
C4	0.16181 (10)	0.8489 (2)	0.25692 (12)	0.0209 (4)
H4	0.180212	0.947540	0.233797	0.025*
C5	0.16413 (11)	1.0501 (2)	0.39453 (13)	0.0237 (4)
H5A	0.149608	1.144834	0.342942	0.028*
H5B	0.230602	1.063499	0.453720	0.028*
C6	0.09867 (11)	1.0683 (2)	0.43607 (13)	0.0254 (4)
H6A	0.110433	1.182494	0.472837	0.031*
H6B	0.031951	1.065266	0.376376	0.031*
C7	0.11841 (11)	0.9144 (2)	0.51303 (12)	0.0224 (4)
H7A	0.182161	0.929830	0.577274	0.027*
H7B	0.072613	0.920891	0.533996	0.027*
C8	0.11217 (10)	0.7311 (2)	0.46702 (11)	0.0184 (3)
C9	0.12528 (9)	0.71732 (19)	0.37669 (11)	0.0168 (3)
C10	0.15071 (10)	0.8699 (2)	0.34218 (12)	0.0183 (3)
O1	0.09886 (8)	0.59653 (14)	0.50503 (9)	0.0250 (3)
O2	0.10025 (8)	0.37319 (14)	0.18943 (9)	0.0249 (3)
H2	0.100350	0.383576	0.134682	0.037*

3 Comment

1-Tetralones are aromatic bicyclic compounds containing 3,4-dihydronaphthalen-1(2*H*)-one (DHN) molecules, which are of increasing interest due to their chemical properties and potential applications in the pharmaceutical industry [6]. Tetrahydronaphthones are organic compounds containing a benzene ring with special aromatic properties. In addition to the benzene ring, it contains a reactive carbonyl functional group, which plays an important role in the properties and reactions of the compound. In addition, methylene groups are present in tetralone, and the presence of methylene groups gives the compound a certain space configuration. The benzene ring in the molecule is aromatic, and the ring in which the carbonyl group is located is not aromatic, resulting in different behaviors of the benzene ring and the carbonyl ring in terms of reactivity and reaction selectivity. Therefore, tetralone can be further synthesized into numerous derivatives by adding different substituents, altering their interactions and bioactivities towards biomolecules [7, 8].

The bond length of C(8)=O(1) is 1.2276 (18) Å, which represents a typical C=O double bond (see the figure). There are also three methylene groups in the molecule, the C(5)–C(6) bond distance is 1.525(2) Å, the C(6)–C(7) bond distance is 1.5223(4) Å, and the angle of C(5)–C(6)–C(7) is 109.37(12)°. In addition, there is a phenolic hydroxyl group with a bond length of 1.3608(17) Å (C(2)–O(2)), which can act as an electron donor or acceptor and link to other molecules in the form of hydrogen bonds. Other bond lengths and bond angles are all in the normal ranges [9, 11]. Due to the distortion effect of 3,4-dihydronaphthalen-1(2*H*)-one, 7-hydroxyphenyl is not coplanar with its aromatic substituents. This distorted conformation may increase the likelihood of interaction with biologically active molecules, resulting in enhanced biological activity [10].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: Science and Technology Innovation Development Plan of Yantai (No.2020XDRH105) and the National Natural Science Foundation of China (No. 81473104).
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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