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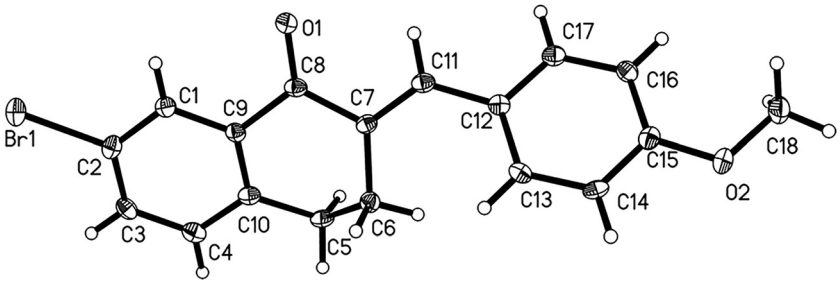
Crystal structure of (*E*)-7-bromo-2-(4-methoxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one, $C_{18}H_{15}BrO_2$



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Abstract

$C_{18}H_{15}BrO_2$, monoclinic, $P2_1/n$ (no. 14), a = 9.1704(9) Å, b = 16.5309(13) Å, c = 10.3528(12) Å, β = 112.368(12)°, V = 1451.3(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0405, $wR_{ref}(F^2)$ = 0.0677, T = 149.99(10) K.

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The molecular structure is shown in the figure. Displacement ellipsoids are drawn at the 40% probability level. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.12 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.834 mm^{-1}
Diffractometer, scan mode:	SuperNova
θ_{max}, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6642, 2694, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1924
$N(param)_{refined}$:	191
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3]

Source of material

7-Bromo-3,4-dihydronaphthalene-1(2*H*)-one (BDHA) was prepared according to the procedures delineated in existing literatures [4, 5]. In the next step, BDHA (0.50 g, 2.20 mmol) and 4-methoxybenzaldehyde (0.30 g, 2.20 mmol) were dissolved in 5 mL of methanol. 25% NaOH solution (2 mL) was added to the above solution and the mixture was stirred for 3.5 h at room temperature (monitored by TLC). The solution was poured to 20 mL water and extracted three times with 10 mL ethyl acetate. The organic layer was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The crude product was purified to obtain the title compound by silica gel column chromatography (Eluent: Petroleum ether/Ethyl acetate/Methanol = 10:10:1, v/v/v). The product was dissolved in methanol and light yellow crystals suitable for single crystal X-ray diffraction were obtained by slow evaporation.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.14469 (3)	0.10658 (2)	1.01994 (4)	0.02932 (13)
C1	0.8313 (3)	0.09750 (18)	0.8161 (4)	0.0187 (8)
H1	0.807693	0.073498	0.887069	0.022*
C2	0.9838 (3)	0.12313 (19)	0.8398 (4)	0.0200 (8)
C3	1.0195 (3)	0.15912 (19)	0.7355 (4)	0.0208 (8)
H3	1.122135	0.175817	0.753186	0.025*
C4	0.9027 (3)	0.17049 (18)	0.6043 (4)	0.0219 (9)
H4	0.927398	0.195144	0.534404	0.026*
C5	0.6201 (3)	0.1537 (2)	0.4332 (4)	0.0215 (8)
H5A	0.611318	0.103741	0.381547	0.026*
H5B	0.647639	0.196599	0.382756	0.026*
C6	0.4605 (3)	0.17306 (19)	0.4431 (4)	0.0201 (8)
H6A	0.465240	0.226296	0.483907	0.024*
H6B	0.378756	0.173909	0.350024	0.024*
C7	0.4194 (3)	0.11158 (18)	0.5304 (4)	0.0175 (8)
C8	0.5500 (3)	0.07868 (19)	0.6569 (4)	0.0190 (8)
C9	0.7139 (3)	0.10863 (18)	0.6828 (4)	0.0175 (8)
C10	0.7478 (3)	0.14516 (18)	0.5760 (4)	0.0185 (8)
C11	0.2729 (3)	0.08307 (19)	0.5047 (4)	0.0197 (8)
H11	0.266369	0.042066	0.563761	0.024*
C12	0.1223 (3)	0.10820 (18)	0.3957 (4)	0.0168 (8)
C13	0.0890 (3)	0.18799 (18)	0.3462 (4)	0.0189 (8)
H13	0.166563	0.227525	0.379100	0.023*
C14	−0.0590 (3)	0.20823 (19)	0.2486 (4)	0.0200 (8)
H14	−0.080132	0.261313	0.217325	0.024*
C15	−0.1757 (3)	0.1495 (2)	0.1974 (4)	0.0197 (8)
C16	−0.1452 (3)	0.07011 (19)	0.2444 (4)	0.0194 (8)
H16	−0.221899	0.030364	0.208992	0.023*
C17	0.0011 (3)	0.05112 (19)	0.3448 (4)	0.0201 (8)
H17	0.019526	−0.001388	0.379460	0.024*
C18	−0.4403 (3)	0.1179 (2)	0.0440 (4)	0.0307 (10)
H18A	−0.406666	0.076397	−0.003329	0.046*
H18B	−0.532917	0.143859	−0.020774	0.046*
H18C	−0.464033	0.094194	0.118449	0.046*
O1	0.5271 (2)	0.03091 (14)	0.7381 (3)	0.0270 (6)
O2	−0.3167 (2)	0.17663 (13)	0.1006 (3)	0.0279 (6)

Experimental details

The H atoms were placed in idealized positions and treated as riding on their parent atoms, with d(C–H) = 0.96 Å (methyl), U_{iso}(H) = 1.5U_{eq}(C), and 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).

Comment

1-Tetralones, a wide collection of aromatic bicyclic compounds containing a moiety of 3,4-dihydronaphthalen-1(2*H*)-one (DHN), are gaining more attention due to their chemical characteristics and potential application in pharmaceutical industry. Recent researches demonstrated that 1-tetralones had a broad spectrum of biological activity, and acted as anticancer drugs [6], antifungal agents [7], and protein inhibitors [8]. In order to obtain new drugs, it is necessary to modify the structure of existing biologically active matrices. A set of aryliden-1-tetralone derivatives have been designed and investigated [9–12]. 6-Methoxy-2-phenyl-3,4-dihydronaphthalen-1(2*H*)-one was found to possess antineoplastic and antiviral properties [13]. As adenosine receptor antagonists, (*E*)-7-hydroxy-2-(4-hydroxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one was proved to be an optimal drug candidate to treat neurodegenerative disorders [14]. As a part of our aim to design new anti-neuroinflammatory agents, aromatic-1-tetralones with chloro, fluoro, bromo, methyl and methoxy groups were synthesized by the Claisen–Schmidt condensation reaction, the crystals of (*E*)-7-bromo-2-(4-methoxy benzylidene)-3,4-dihydronaphthalen-1(2*H*)-one were obtained and its structure is reported here.

X-ray crystallographic analysis reveals that the title compound crystallizies in the monoclinic space group P $2_1/n$ with one molecule in the asymmetric unit (*cf*. the figure). The torsion angle of C8–C7–C11–C12 is 175.2(3)°, meaning that the title compound adopts the *E* stereochemistry. The length of C7=C8 olifinic bond is 1.351(4) Å, which is similar to those reported in tetralone-containing compounds [15–18]. Because of the sp^3 hybridization of C5 and C6 atoms, the cyclohexanone ring of 3,4-dihydro naphthalen-1(2*H*)-one displays a chair conformation. Simultaneously, the two benzene rings are turned to each other with a dihedral angle of 51.7(3)°. Though no classic hydrogen bonds were found in the crystal structure, adjacent molecules are connected to form one dimensional structure *via* the C18–H18A–O1 weak hydrogen bond, wich is further linked to form one layer in the ac plane by the C4–H···4pi interaction (pi represents the center of the methoxylbenzene ring, C4···pi = 3.548(4) Å). Because of the existence of dihydronaphthalenone and methoxy groups, the title compound may establish effectively hydrophobic interactions with some proteins. Additionally, the bromine atom will be able to enhance cell permeability and metabolic stability of the title compound. It is of great importance in the design and optimization of halogenated drugs.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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