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Crystal structure of (*E*)-2-((3-fluoropyridin-4-yl)methylene)-7-methoxy-3,4-dihydronaphthalen-1(2*H*)-one, C₁₇H₁₄FNO₂

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Abstract

C₁₇H₁₄FNO₂, monoclinic, *P*2₁/*n* (no. 14), *a* = 8.0937(8) Å, *b* = 7.1326(6) Å, *c* = 22.367(2) Å, β = 90.863(9)°, *V* = 1291.1(2) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0404, *wR*_{ref}(*F*²) = 0.0974, *T* = 99.97(16) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared according to a literature protocol [4]. 7-Methoxy-3,4-dihydronaphthalen-1(2*H*)-one

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.13 × 0.12 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.11 mm ⁻¹
Diffractometer, scan mode:	SuperNova,
θ _{max} , completeness:	25.5°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	5014, 2397, 0.028
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2042
<i>N</i> (<i>param</i>) _{refined} :	192
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

(0.52 g, 3.0 mmol) and 3-fluoro-4-pyridinecarbaldehyde (0.38 g, 3.0 mmol) were dissolved in 10 mL of methanol. Then NaOH solution (5 M, 3 mL) was added to the above solution. The reaction mixture was stirred for 5 h at room temperature (monitored by thin-layer chromatography). The solvent was removed by filtration and the residues were purified by silica gel column chromatography (petroleum ether/ethyl acetate = 2:1, v/v) to afford light yellow powders. Crystals suitable for X-ray diffraction were obtained by slow evaporation from the solution of dichloromethane and methanol (1:1, v/v) at room temperature.

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) and *U*_{iso}(H) = 1.5*U*_{eq}(C); *d*(C–H) = 0.97 Å (methylene) and *U*_{iso}(H) = 1.2*U*_{eq}(C); *d*(C–H) = 0.93 Å (aromatic) and *U*_{iso}(H) = 1.2*U*_{eq}(C).

Comment

Arylidene-1-tetralone and its derivatives have been investigated for their antitumor, antifungal and antimicrobial properties [5–7]. As one kind of chalcone analogs, arylidene-1-tetralones belong to bicyclic benzofused ring systems, which contain the 3,4-dihydronaphthalen-1(2*H*)-one and the α,β-unsaturated keto group. The

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.69427 (18)	0.0157 (2)	0.92053 (7)	0.0196 (4)
C2	0.76224 (18)	0.2018 (2)	0.90201 (7)	0.0203 (4)
C3	0.79283 (19)	0.3431 (2)	0.95076 (7)	0.0221 (4)
H3A	0.688203	0.397014	0.962515	0.027*
H3B	0.861035	0.443536	0.935453	0.027*
C4	0.87811 (19)	0.2571 (2)	1.00559 (7)	0.0221 (4)
H4A	0.992980	0.233051	0.996489	0.026*
H4B	0.875548	0.346523	1.038225	0.026*
C5	0.81557 (19)	0.0132 (2)	1.08382 (7)	0.0233 (4)
H5	0.870872	0.087974	1.111700	0.028*
C6	0.75292 (19)	−0.1577 (2)	1.10211 (7)	0.0232 (4)
H6	0.766726	−0.196803	1.141535	0.028*
C7	0.66910 (18)	−0.2705 (2)	1.06092 (7)	0.0195 (4)
C8	0.64734 (18)	−0.2087 (2)	1.00270 (7)	0.0187 (4)
H8	0.588971	−0.282454	0.975362	0.022*
C9	0.71196 (18)	−0.0374 (2)	0.98463 (7)	0.0180 (3)
C10	0.79889 (18)	0.0772 (2)	1.02539 (7)	0.0196 (4)
C11	0.79571 (19)	0.2204 (2)	0.84386 (7)	0.0212 (4)
H11	0.763562	0.120936	0.819433	0.025*
C12	0.87676 (19)	0.3778 (2)	0.81373 (7)	0.0217 (4)
C13	0.9930 (2)	0.3404 (2)	0.77042 (7)	0.0236 (4)
C14	1.0772 (2)	0.4790 (3)	0.74124 (7)	0.0296 (4)
H14	1.155374	0.445150	0.713161	0.036*
C15	0.9359 (2)	0.7008 (2)	0.79215 (7)	0.0287 (4)
H15	0.913909	0.826469	0.799674	0.034*
C16	0.8477 (2)	0.5681 (2)	0.82344 (7)	0.0261 (4)
H16	0.769167	0.605597	0.850914	0.031*
C17	0.6258 (2)	−0.5127 (2)	1.13331 (7)	0.0288 (4)
H17A	0.741671	−0.524486	1.142403	0.043*
H17B	0.573892	−0.633239	1.136535	0.043*
H17C	0.576493	−0.427248	1.160973	0.043*
F1	1.02908 (12)	0.15933 (13)	0.75778 (4)	0.0322 (3)
N1	1.05085 (18)	0.6602 (2)	0.75170 (6)	0.0306 (4)
O1	0.60413 (13)	−0.44294 (15)	1.07363 (4)	0.0228 (3)
O2	0.63303 (14)	−0.09245 (15)	0.88405 (5)	0.0273 (3)

pharmacophore of α,β-unsaturated keto moiety can establish the primary binding interaction with bio-thiols from susceptible neoplasms with lower toxicity [8]. Thus, arylidene-1-tetralones demonstrate greater antiproliferative activities against tumor cell lines [9, 10]. Simultaneously, the two aryl rings presented in the arylidene-1-tetralones were easily modified to obtain novel drugs by suitable substituents and functional groups. 2-Benzylidene-1-tetralones with the appropriate substituent (OH, F, Cl, Br, CH₃, N(CH₃)₂, OCH₃) were found to show good inhibitory activity for monoamine oxidase enzymes [11]. Recent results showed that 2-arylidene-1-tetralones containing pyridine groups demonstrated modest biological activity against cancer, bacteria and fungi [12, 13]. In addition, a fluorine

substitution was found to play an important role in bioactivities. As a part of our continuing research on the inhibition of neurogenic inflammation, one new 2-arylidene-1-tetralone that combines a fluorinated aryl moiety and the pyridine group was synthesized by the Claisen–Schmidt condensation reaction.

The title compound crystallizes in the triclinic space group *P*2₁/*n* with one molecule in the asymmetric unit. All bond lengths and angles are normal and comparable with those previously reported for the related structure [14–16]. As shown in the figure, the benzofused rings and the pyridine ring are arranged around the olefinic double bond. The arrangement is in favor for an *E* stereochemical structure. No classic hydrogen bonds are found in the crystal structure, but weak C–H⋯F hydrogen bonds link molecules to form a chain along the crystallographic *c* axis. Because of the high electronegativity, the fluorine atom in the title compound will have higher molecular lipophilicity, which can enhance the membrane permeability of drug, increase binding affinity of target protein and improve metabolic stability [17–19].

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