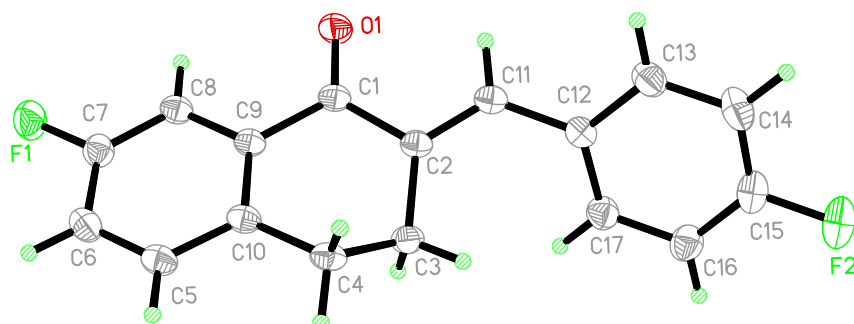


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Crystal structure of (*E*)-7-fluoro-2-(4-fluorobenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one, C₁₇H₁₂F₂O



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Abstract

C₁₇H₁₂F₂O, triclinic, $P\bar{1}$ (no. 2), $a = 7.9570(14)$ Å, $b = 8.9872(19)$ Å, $c = 9.3974(16)$ Å, $\alpha = 94.467(16)^\circ$, $\beta = 96.737(14)^\circ$, $\gamma = 103.162(16)^\circ$, $V = 646.0(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0417$, $wR_{\text{ref}}(F^2) = 0.1112$, $T = 293$ K.

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

Table 1: Data collection and handling.

Crystal:	Yellow block
Size	0.14 × 0.12 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractionmeter, scan mode:	Xcalibur
θ_{max} , completeness:	25.5°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4428, 2400, 0.016
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1652
$N(\text{param})_{\text{refined}}$:	181
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

1 Source of material

7-Fluoro-3,4-dihydronaphthalen-1(2*H*)-one (1.32 g, 8.06 mmol) and 4-fluorobenzaldehyde (1.0 g, 8.06 mmol) were dissolved in 10 mL acetic acid. Then dry hydrogen chloride gas was flowed continuously into the solution for 45 min. After gas insertion, the reaction system was stirred at room temperature for seven days. The response endpoint was detected by thin layer chromatography (TLC). When the reaction was stopped, the precipitate was filtered from the reaction system, then it was dissolved in distilled water and regulated to a neutral pH with saturated aqueous Na₂CO₃ solution. The precipitate was filtered from the system and dissolved with dichloromethane. The organic phase was washed successively with deionized water and brine, dried over anhydrous sodium sulfate and condensed under vacuum. The crude product was purified by silica-gel column chromatography (petroleum ether:ethyl acetate = 10:1, v:v). Single crystal was obtained under ambient conditions via solvent evaporation in the mixed solvents of dichloromethane and methanol (1:1, v:v) and drying under vacuo at 333 K for 3 h.

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2897 (2)	0.86379 (17)	1.08772 (17)	0.0506 (4)
C2	0.2123 (2)	0.74985 (17)	0.95954 (17)	0.0495 (4)
C3	0.3155 (2)	0.63419 (18)	0.92180 (19)	0.0572 (4)
H3A	0.271923	0.584786	0.824813	0.069*
H3B	0.300575	0.555561	0.987450	0.069*
C4	0.5082 (2)	0.71159 (19)	0.93126 (18)	0.0578 (4)
H4A	0.572539	0.634308	0.912205	0.069*
H4B	0.524186	0.782153	0.858237	0.069*
C5	0.7488 (2)	0.80928 (19)	1.1405 (2)	0.0606 (5)
H5	0.821014	0.761959	1.091607	0.073*
C6	0.8127 (2)	0.8891 (2)	1.2743 (2)	0.0641 (5)
H6	0.926527	0.896022	1.315988	0.077*
C7	0.7039 (2)	0.95811 (19)	1.34421 (19)	0.0605 (5)
C8	0.5368 (2)	0.95247 (18)	1.28701 (18)	0.0554 (4)
H8	0.467020	1.001660	1.336998	0.066*
C9	0.47278 (19)	0.87084 (16)	1.15122 (17)	0.0481 (4)
C10	0.5789 (2)	0.79775 (17)	1.07690 (17)	0.0510 (4)
C11	0.0605 (2)	0.75926 (18)	0.88763 (17)	0.0532 (4)
H11	0.018322	0.843308	0.917254	0.064*
C12	−0.0481 (2)	0.65289 (18)	0.76740 (17)	0.0531 (4)
C13	−0.1454 (2)	0.7082 (2)	0.6616 (2)	0.0706 (5)
H13	−0.142088	0.812596	0.667829	0.085*
C14	−0.2477 (3)	0.6113 (3)	0.5467 (2)	0.0832 (6)
H14	−0.310795	0.649882	0.474736	0.100*
C15	−0.2541 (3)	0.4580 (2)	0.5411 (2)	0.0737 (6)
C16	−0.1665 (2)	0.3968 (2)	0.6456 (2)	0.0725 (5)
H16	−0.175645	0.291410	0.640775	0.087*
C17	−0.0640 (2)	0.49485 (19)	0.7584 (2)	0.0648 (5)
H17	−0.003718	0.454490	0.830773	0.078*
F1	0.76687 (14)	1.03479 (13)	1.47806 (12)	0.0861 (4)
F2	−0.35490 (17)	0.36137 (16)	0.42858 (13)	0.1097 (5)
O1	0.20658 (15)	0.94673 (14)	1.14246 (13)	0.0678 (4)

2 Experimental details

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

3 Comment

Some 3,4-dihydronaphthalene-1(2*H*)-one (DHN) derivatives have been used as novel modulators of allergic and inflammatory reactions, possessing anti-tumor and anti-inflammatory activities [4, 5]. Based on the above studies, the target product of (*E*)-7-fluoro-2-(4-fluorobenzylidene)-

3,4-dihydronaphthalene-1(2*H*)-one was synthesized by the Claisen–Schmidt condensation reaction.

The ORTEP diagram is presented in the Figure. The title compound contains one drug molecule in the asymmetric unit (cf. the figure). Bond lengths and angles are all in the expected ranges. The title molecule has an approximately linear structure [6–8]. The parent nucleus is 3,4-dihydronaphthalene-1(2*H*)-one and the seventh position of the aromatic ring is fluorinated. The olefinic double bonds between 3,4 dihydronaphthalene-1(2*H*)-one and 4-fluorobenzaldehyde groups and carbonyl group form alpha and beta unsaturated ketones. With respect to the C(2) = C(11) olefinic bond, 4-fluorobenzaldehyde and carbonyl groups adopt the *E* stereochemistry [9, 10]. Because of the distorting effect of 3,4-dihydronaphthalene-1(2*H*)-one, the 7-fluorophenyl and 4-fluorobenzaldehyde groups are not coplanar with each other, with a dihedral angle of approximately 57.9° [6]. This twisted configuration may increase the likelihood of interactions with bioactive molecules or the purposes of creating more potent biological activity [11].

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

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