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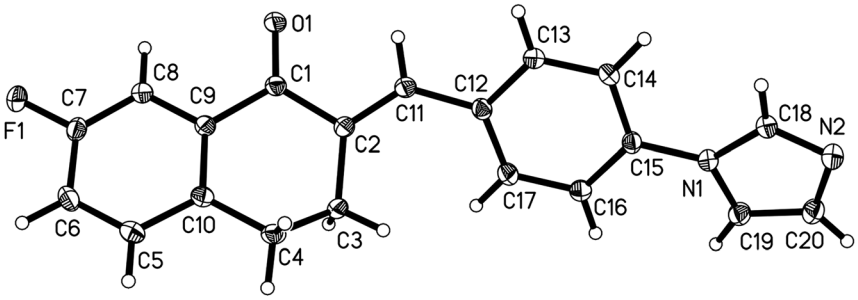
Crystal structure of (*E*)-2-(4-(1*H*-imidazol-1-yl) benzylidene)-7-fluoro-3,4-dihydronaphthalen-1(2*H*)-one, $C_{20}H_{15}FN_2O$



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Abstract

$C_{20}H_{15}FN_2O$, triclinic, $P\bar{1}$ (no. 2), a = 8.0724(1) Å, b = 9.6473(1) Å, c = 9.6635(1) Å, α = 80.903(1)°, β = 82.325(1)°, γ = 84.147(1)°, V = 733.922(14) Å^3, Z = 2, $R_{gt}(F)$ = 0.0366, $wR_{ref}(F^2)$ = 0.0934, T = 293 K.

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The crystal structure is shown in the Figure. 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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.15 × 0.13 × 0.10 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	0.81 mm^{-1}
Diffractometer, scan mode:	XtaLAB AFC12 (RINC),
θ_{max}, completeness:	71.9°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6,303, 2,770, 0.014
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,687
$N(param)_{refined}$:	218
Programs:	CrysAlis^{PRO}[1], SHELX[2,3]

1 Source of material

Synthesis method based on literature in Ref. 4: in a 250 mL spherical backside flask, imidazole (10.88 g, 0.16 mol) and potassium carbonate (27.64 g, 0.2 mol) were reacted with *N*,*N*-dimethylformamide (10.0 mL) at 353 K for 4 h. The *p*-fluorobenzaldehyde (2.48 g, 0.02 mol) is added and the temperature is raised to 393 K for reflux. Thin-Layer Chromatography (TLC, dichloromethane:methyl alcohol = 15:1, *v*:*v*) was used for detection. After the response, the yellow intermediate was purified by extraction, vacuum distillation and silica gel column (dichloromethane:methyl alcohol = 30:1, *v*:*v*). Using 25 % sodium hydroxide (2.0 mL) as catalyst, the intermediate (1.70 g, 10.00 mmol) and 7-fluoro-3,4-dihydronaphthhalen-1(2*H*)-one (2.30 g, 10.00 mmol) were dissolved in methanol (5.0 mL) and stirred at room temperature for 1 h. Filtered and the filter cake dissolved in methylene chloride, the crude product was obtained by vacuum distillation and recrystallized at 293 K to obtain yellow crystals (dichloromethane:methyl alcohol = 1:1, *v*:*v*).

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

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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}
C1	0.82305 (14)	0.64246 (12)	−0.13061 (12)	0.0226 (3)
C2	0.77436 (14)	0.73780 (12)	−0.02250 (12)	0.0223 (3)
C3	0.90416 (15)	0.83353 (12)	−0.00444 (13)	0.0255 (3)
H3A	0.870902	0.874896	0.081287	0.031*
H3B	0.911473	0.909311	−0.083435	0.031*
C4	1.07581 (15)	0.75065 (13)	0.00353 (13)	0.0266 (3)
H4A	1.159296	0.814850	0.007953	0.032*
H4B	1.071606	0.682666	0.089119	0.032*
C5	1.29487 (15)	0.65281 (13)	−0.17719 (13)	0.0271 (3)
H5	1.377630	0.682788	−0.133197	0.032*
C6	1.34109 (15)	0.58753 (13)	−0.29541 (13)	0.0286 (3)
H6	1.453236	0.573879	−0.331600	0.034*
C7	1.21586 (15)	0.54301 (12)	−0.35851 (13)	0.0262 (3)
C8	1.04922 (15)	0.55929 (12)	−0.30723 (12)	0.0247 (3)
H8	0.968042	0.526767	−0.351028	0.030*
C9	1.00428 (14)	0.62582 (12)	−0.18776 (12)	0.0218 (3)
C10	1.12670 (14)	0.67499 (12)	−0.12191 (12)	0.0229 (3)
C11	0.62089 (14)	0.72668 (12)	0.05057 (12)	0.0233 (3)
H11	0.563242	0.655199	0.030039	0.028*
C12	0.52927 (14)	0.80754 (12)	0.15656 (12)	0.0227 (3)
C13	0.39888 (14)	0.74504 (12)	0.24790 (12)	0.0246 (3)
H13	0.374321	0.655116	0.238504	0.030*
C14	0.30593 (14)	0.81330 (13)	0.35142 (13)	0.0256 (3)
H14	0.220059	0.769395	0.410675	0.031*
C15	0.34086 (14)	0.94791 (12)	0.36703 (12)	0.0219 (3)
C16	0.46513 (15)	1.01424 (12)	0.27461 (13)	0.0259 (3)
H16	0.486813	1.105295	0.282638	0.031*
C17	0.55700 (15)	0.94529 (13)	0.17041 (13)	0.0263 (3)
H17	0.638856	0.991496	0.108326	0.032*
C18	0.15526 (14)	0.95435 (13)	0.59372 (12)	0.0250 (3)
H18	0.140833	0.858480	0.611856	0.030*
C19	0.23889 (15)	1.15810 (13)	0.48889 (13)	0.0281 (3)
H19	0.289392	1.229127	0.425915	0.034*
C20	0.14064 (16)	1.17197 (13)	0.61159 (13)	0.0305 (3)
H20	0.112239	1.256746	0.646644	0.037*
F1	1.26047 (9)	0.48216 (8)	−0.47745 (8)	0.0357 (2)
N1	0.24878 (12)	1.01667 (10)	0.47639 (10)	0.0229 (2)
N2	0.08820 (13)	1.04393 (11)	0.67778 (11)	0.0295 (3)
O1	0.72062 (10)	0.58009 (10)	−0.17490 (9)	0.0320 (2)

(methylene), U_{iso}(H) = 1.2U_{eq}(C), and *d*(C–H) = 0.93 Å (aromatic), U_{iso}(H) = 1.2U_{eq}(C).

3 Comment

It has been established that 3,4-dihydronaphthalene-1(2*H*)-one derivatives act as *a*,*b*-unsaturated ketones having proper anti-tumor and anti-inflammatory things to do.[4,5] It frequently inhibits the activation of NF-κB transcription factors *in vivo* to exert therapeutic effects.[6] In order to similarly discover molecules with higher therapeutic effects, we selected to introduce an imidazole ring at the C15 position, it can no longer solely enhance the water solubility of molecules *via* intramolecular hydrogen bonding, and it additionally has incredible contributions in antibacterial and anti-inflammatory aspects.[7] In this study, *p*-fluorobenzaldehyde and imidazole reacted to acquire 4-(1*H*-imidazol-1-yl)benzaldehyde intermediate. Then, the intermediate reacts with 7-fluoro-3,4-dihydronaphthalen-1(2*H*)-one to obtain the title compound through the Claisen–Schmidt reaction.

Single-crystal structure analysis reveals that there is only a molecule in the asymmetric unit of the title crystal structure (*cf*. the Figure). Bond lengths and angles are all in the expected ranges.[8–10] Fluorine atoms with electron withdrawing properties replace the 7 position of 3,4-dihydronaphthalene-1(2*H*)-one, and the introduction of fluorine can decorate organic undertaking and enhance metabolism.[11] In the C(2) position, *a*,*b*-unsaturated ketone was obtained through the Claisen–Schmidt reaction.[8,9,12] The torsion angle of O(1)=C(1)–C(2)=C(11) is about 15.96(17)°. The bond lengths of O(1)=C(1) and C(2)=C(11) are 1.2274(14) Å and 1.3470(16) Å, respectively. In the C(15) position, there's an imidazole group substitution. Nitrogen atoms on imidazole groups have lone pair electrons, which are easy to act as hydrogen bond receptors and form hydrogen bond or electrostatic interaction with bioactive molecules.[7] The dihedral angle between 3,4-dihydronaphthalene-1(2*H*)-one and phenyl group is about 49.2°. The dihedral angle between the phenyl group and the imidazole group is about 16.8°.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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