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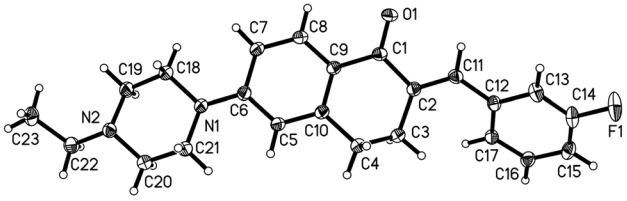
Crystal structure of (*E*)-6-(4-ethylpiperazin-1-yl)-2-(3-fluorobenzylidene)-3,4-dihydronaphthalen-1(*2H*)-one, $C_{23}H_{25}FN_2O$



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Abstract

$C_{23}H_{25}FN_2O$, triclinic, $P\bar{1}$ (no. 2), a = 6.5011(2) Å, b = 11.2794(4) Å, c = 14.0796(6) Å, α = 110.246(4)°, β = 97.236(3)°, γ = 98.335(2)°, V = 941.01(6) Å^3, Z = 2, $R_{gt}(F)$ = 0.0476, $wR_{ref}(F^2)$ = 0.1366, 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:	0.69 mm^{-1}
Diffractometer, scan mode:	SuperNova
θ_{max}, completeness:	73.8°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14,800, 3721, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3395
$N(param)_{refined}$:	245
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3]

1 Source of material

Based on literature synthetic techniques [4, 5], *N*-ethylpiperazine (18.27 g, 0.16 mol) and potassium carbonate (27.64 g, 0.2 mol) have been delivered to *N*,*N*-dimethylformamide (2 mL), and after stirring at 313 K overnight, 6-fluoro-3,4-dihydronaphthalen-1(*2H*)-one (3.28 g, 0.02 mol) and *N*,*N*-dimethylformamide (0.5 mL) had been combined and introduced dropwise to the response gadget and the temperature of mixture was adjusted to 373 K. The reaction lasted for 5.5 h. It used to be monitored *via* thin-layer chromatography (TLC, dichloromethane:methyl alcohol = 10:1, *v*:*v*). The product was filtered and the filtrate used to be washed with dichloromethane. The solvent was once eliminated by using attention underneath decreased pressure. Separation and purification used to be carried out by way of a silica gel column with the use of dichloromethane:methyl alcohol (20:1, *v*:*v*) as eluent. Intermediates have been obtained. Using 25 % sodium hydroxide solution (5.0 mL) as a catalyst, 6-(4-ethylpiperazinyl)-3,4-dihydronaphthalen-1(2*H*)-one (0.26 g, 1.00 mmol) used to be blended with 3-fluorobenzaldehyde (0.274 g, 2 mmol) in 10 mL of methanol for 12 h at room temperature below nitrogen atmosphere. The filter residue was once dissolved using dichloromethane and washed with distilled water. The accumulated natural segment used to be dried over anhydrous sodium sulfate. The solvent used to be eliminated by way of awareness underneath decreased pressure. The received product is soluble in a combination of dichloromethane (2 mL) and methyl alcohol (2 mL), and slowly evaporated at room temperature to achieve appropriate colourless crystals, (*E*)-6-(4-ethylpiperazin-1-yl)-2-(3-fluorobenzylidene)-3,4-dihydronaphthalen-1(*2H*)-one.

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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	1.1192 (2)	0.22187 (14)	0.09765 (11)	0.0367 (3)
C2	0.91358 (19)	0.17804 (12)	0.02154 (10)	0.0333 (3)
C3	0.7120 (2)	0.17249 (14)	0.06413 (10)	0.0377 (3)
H3A	0.596591	0.115287	0.009579	0.045*
H3B	0.678803	0.257850	0.087683	0.045*
C4	0.7299 (2)	0.12527 (14)	0.15274 (10)	0.0391 (3)
H4A	0.603104	0.132011	0.182304	0.047*
H4B	0.739950	0.035066	0.126926	0.047*
C5	0.9158 (2)	0.22839 (12)	0.33858 (10)	0.0332 (3)
H5	0.789688	0.201500	0.356809	0.040*
C6	1.0955 (2)	0.29448 (12)	0.41698 (10)	0.0325 (3)
C7	1.2853 (2)	0.32830 (13)	0.38452 (10)	0.0365 (3)
H7	1.409173	0.368293	0.433309	0.044*
C8	1.2896 (2)	0.30316 (13)	0.28254 (10)	0.0362 (3)
H8	1.416403	0.327773	0.263856	0.043*
C9	1.10892 (19)	0.24154 (12)	0.20552 (10)	0.0314 (3)
C10	0.92115 (19)	0.20228 (12)	0.23548 (10)	0.0315 (3)
C11	0.9237 (2)	0.15400 (13)	−0.07730 (10)	0.0351 (3)
H11	1.058834	0.158119	−0.092789	0.042*
C12	0.7489 (2)	0.12184 (12)	−0.16484 (10)	0.0330 (3)
C13	0.7657 (2)	0.03678 (14)	−0.26175 (11)	0.0414 (3)
H13	0.884262	−0.000233	−0.270254	0.050*
C14	0.6048 (3)	0.00868 (14)	−0.34410 (11)	0.0454 (4)
C15	0.4289 (2)	0.06224 (14)	−0.33743 (11)	0.0436 (3)
H15	0.323100	0.041018	−0.394981	0.052*
C16	0.4133 (2)	0.14883 (14)	−0.24257 (11)	0.0393 (3)
H16	0.296638	0.187975	−0.236045	0.047*
C17	0.5705 (2)	0.17773 (13)	−0.15700 (10)	0.0350 (3)
H17	0.556929	0.235234	−0.093399	0.042*
C18	1.2817 (2)	0.36874 (16)	0.59737 (11)	0.0431 (3)
H18A	1.387071	0.421999	0.578309	0.052*
H18B	1.336297	0.293655	0.598829	0.052*
C19	1.2444 (2)	0.44441 (14)	0.70348 (11)	0.0417 (3)
H19A	1.375352	0.467765	0.753162	0.050*
H19B	1.201972	0.523421	0.703566	0.050*
C20	0.8866 (2)	0.33917 (15)	0.65923 (11)	0.0409 (3)
H20A	0.845014	0.418449	0.659484	0.049*
H20B	0.774742	0.291008	0.678877	0.049*
C21	0.9130 (2)	0.26050 (15)	0.55183 (11)	0.0435 (3)
H21A	0.941764	0.177807	0.550021	0.052*
H21B	0.782283	0.244486	0.503628	0.052*
C22	1.0481 (2)	0.44190 (14)	0.83798 (10)	0.0412 (3)
H22A	0.925817	0.394120	0.851295	0.049*
H22B	1.016742	0.524341	0.840573	0.049*
C23	1.2364 (3)	0.46564 (16)	0.92225 (11)	0.0502 (4)
H23A	1.269932	0.384601	0.919697	0.075*
H23B	1.202438	0.509687	0.988194	0.075*
H23C	1.355975	0.517684	0.912241	0.075*
F1	0.6224 (2)	−0.07580 (10)	−0.43747 (7)	0.0724 (4)
N1	1.08606 (17)	0.32706 (11)	0.52014 (9)	0.0369 (3)
N2	1.08151 (17)	0.37027 (10)	0.73407 (8)	0.0334 (3)
O1	1.28790 (16)	0.24587 (15)	0.07123 (9)	0.0617 (4)

2 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).

3 Comment

3,4-Dihydronaphthalen-1(2*H*)-one is a potential active fragment of anti-tumor and anti-inflammatory compounds [4, 6]. In latest years, some active compounds, such as (2-((2-fluoro-4-(trifluoromethyl)phenyl)(hydroxy)methyl)-7-methoxy-3,4-dihydronaphthalen-1((2*H*))-one) [7], (*E*)-7-bromo-2-(4-(4-methylpiperazin)benzylidene)-3,4-dihydronaphthhalen-1(2*H*)-one [8] and (*E*)-7-bromo-2-(4-methoxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one [9] were reported. Nitrogen-containing heterocyclic compounds have accurate organic activity, are noticeably secure in the human body, and have excessive operational efficiency. They are convenient to have interaction with DNA through hydrogen bonds [10]. So we use *N*-ethylpiperazine for substitution in this study. The substitution of benzylidene can decorate anti-inflammatory activity [11]. The introduction of *α*,*β*-unsaturated ketone can make 3,4-dihydronaphthalene-1(2*H*)-one have two pharmacophores and minimize cytotoxicity, as proven by our group [12, 13]. Some 3,4-dihydronaphthalene-1(2*H*)-one derivatives substituted by way of electron withdrawing businesses have been proven to have anti-neuroinflammatory effects [4]. Therefore, we are changing benzaldehyde with electron withdrawing fluorophenyl group, which can amplify the lipophilicity of drugs [14]. In this study, *N*-ethylpiperazine and 3,4-dihydronaphthalen-1(2*H*)-one were used to shape an intermediate through nucleophilic addition, further to generate the title compound, (*E*)-6-(4-ethylpiperazin)-2-(3-fluorobenzidene)-3,4-dihydronaphthhalen-1(2*H*)-one, through Claisen–Schmidt condensation with 3-fluoro-benzaldehyde.

Single-crystal structure analysis reveals that the title compound crystallizes in the triclinic system $P\bar{1}$, with one molecule in the asymmetric unit. The bond lengths and bond angles of the title compound are within the normal range and are consistent with the compounds reported previously [7–9]. The pharmacophore of the title compound is 3,4-dihydronaphthalene-1(2*H*)-one, which was substituted by *N*-ethylpiperazine at C6 position and by 3-fluorobenzidene

at C2 position. 3-Fluorobenzene is linked to the central 3,4-dihydronaphthalene-1(2*H*)-one ring by a C(2)=C(11) double bond. It adopts the *E* stereochemistry of the alkene double bond [5, 7, 8]. The bond length of C(2)=C(11) is 1.3348(18) Å. The torsion angle of O(1)=C(1)–C(2)–C(11) is about −6.5(2)°, and the torsion angle of C(1)–C(2)–C(11)–C(12) is about −173.92(12)°, respectively. 3,4-Dihydronaphthalene-1(2*H*)-one ketone is now not coplanar with 3-fluorobenzidene, and their dihedral angle is about 49.88(3)°. In addition, *N*-ethylpiperazine ring takes a "chair" configuration [15]. The torsion angle of N(1)–C(21)–C(20)–N(2) is 56.88(17)°, and the torsion angle of N(1)–C(18)–C(19)–N(2) is −56.76(17)°, respectively. The N(2) atom at the terminal of the molecule is replaced by an ethyl group. The bond length of N(2)–C(22) is 1.4674(17) Å. As a whole, the title compound appears as a linear structure.

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

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

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