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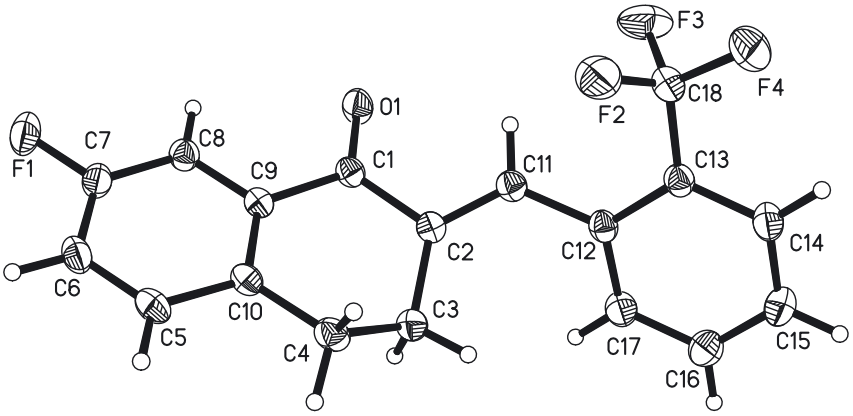
Crystal structure of *E*-7-fluoro-2-(2-(trifluoromethyl)benzylidene)-3,4-dihydronaphthalen-1(2*H*)-one, $C_{18}H_{12}F_4O$



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Abstract

$C_{18}H_{12}F_4O$, triclinic, $P\bar{1}$ (no. 2), a = 7.9400(8) Å, b = 8.1071(9) Å, c = 13.3238(15) Å, α = 89.394(9)°, β = 76.343(9)°, γ = 63.411(11)°, V = 740.79(15) Å^3, Z = 2, $R_{gt}(F)$ = 0.0472, $wR_{ref}(F^2)$ = 0.1244, T = 293 K.

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The crystal structure is shown in figure. Displacement ellipsoids are drawn at the 20% 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.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.16 × 0.13 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.12 mm^{-1}
Diffractometer, scan mode:	Xcalibur
θ_{max}, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5090, 2757, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1886
$N(param)_{refined}$:	208
Programs:	CrysAlis^{PRO} [1], SHELX [2,3]

1 Source of material

7-Methoxy-3,4-dihydronaphthalen-1(2*H*)-one (0.70 g, 4.26 mmol) and 2-(trifluoromethyl)benzaldehyde (0.74 g, 4.26 mmol) were dissolved in 10 mL acetic acid. Then dry hydrogen chloride gas was flowed continuously into the solution lasting 45 min. After gas insertion, the reaction system was stirred at room temperature for 5 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 Na_2CO_3 solution. The precipitate was filtered and dissolved with dichloromethane. The organic phase was washed successively with deionized water and brine, dried over anhydrous sodium sulfate and condensed under vacuum.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3021 (3)	0.1159 (3)	0.44630 (15)	0.0467 (5)
C2	0.4080 (3)	0.0627 (3)	0.33376 (14)	0.0454 (5)
C3	0.5253 (3)	0.1620 (3)	0.29075 (16)	0.0566 (6)
H3A	0.567905	0.136452	0.215629	0.068*
H3B	0.640379	0.116239	0.317107	0.068*
C4	0.4058 (4)	0.3704 (3)	0.32088 (16)	0.0599 (6)
H4A	0.489801	0.428872	0.299560	0.072*
H4B	0.304894	0.418905	0.283863	0.072*
C5	0.2672 (3)	0.5915 (3)	0.48469 (18)	0.0593 (6)
H5	0.297861	0.675030	0.446001	0.071*
C6	0.1780 (3)	0.6394 (3)	0.58907 (19)	0.0614 (6)
H6	0.146742	0.754411	0.621083	0.074*
C7	0.1361 (3)	0.5131 (3)	0.64492 (17)	0.0587 (6)
C8	0.1763 (3)	0.3443 (3)	0.60074 (16)	0.0527 (5)
H8	0.144977	0.262291	0.640638	0.063*
C9	0.2651 (3)	0.2971 (3)	0.49480 (15)	0.0439 (5)
C10	0.3127 (3)	0.4202 (3)	0.43546 (15)	0.0482 (5)
C11	0.3828 (3)	−0.0611 (3)	0.28211 (15)	0.0503 (5)
H11	0.294817	−0.100206	0.319308	0.060*
C12	0.4775 (3)	−0.1433 (3)	0.17318 (15)	0.0488 (5)
C13	0.3769 (3)	−0.1758 (3)	0.10799 (16)	0.0513 (5)
C14	0.4727 (4)	−0.2554 (3)	0.00690 (18)	0.0677 (7)
H14	0.404360	−0.275303	−0.035509	0.081*
C15	0.6667 (4)	−0.3055 (4)	−0.03190 (19)	0.0770 (7)
H15	0.729358	−0.358716	−0.100242	0.092*
C16	0.7680 (4)	−0.2771 (4)	0.0303 (2)	0.0738 (7)
H16	0.899727	−0.310549	0.004040	0.089*
C17	0.6757 (3)	−0.1991 (3)	0.13151 (18)	0.0617 (6)
H17	0.747248	−0.183081	0.173210	0.074*
C18	0.1659 (4)	−0.1226 (4)	0.14540 (18)	0.0653 (6)
F1	0.0486 (2)	0.5596 (2)	0.74883 (11)	0.0901 (5)
F2	0.0566 (2)	0.0534 (2)	0.18312 (14)	0.1006 (6)
F3	0.1271 (3)	−0.2185 (3)	0.22106 (14)	0.1131 (7)
F4	0.0901 (2)	−0.1483 (3)	0.07227 (13)	0.1153 (7)
O1	0.2475 (3)	0.0147 (2)	0.49754 (11)	0.0672 (5)

The crude product was purified by silica-gel column chromatography (petroleum ether: ethyl acetate = 8:1, *v*:*v*). Crystals were obtained under ambient conditions via solvent evaporation in the mixed solvents of dichloromethane and methanol (2:1, *v*:*v*) and drying under vacuo at 333 K for 3 h.

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 (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 (DHN) is a ketone derivative that is often used as an intermediate in molecular synthesis blocks. Many compounds contain this structure [4–6]. In this work, we have synthesized novel DHN derivative *E*-7-fluoro-2-(2-(trifluoromethyl)benzylidene)-3,4-dihydronaphthalen-1(2*H*)-one, using the Claisen-schmidt condensation reaction and report their single crystal structures. This provides an important basis for the study of DHNs derivatives.

The ORTEP diagram is presented in the Figure. The title compound contains one drug molecule in the asymmetric unit (cf. the figure). The 7 position of the aromatic ring in the parent nucleus of 3,4-dihydronaphthalen-1(2*H*)-one is fluorinated [7,8]. The trifluoromethyl group replaces the hydrogen in the benzene ring at position 13. And the carbons at positions 3 and 4 are saturated carbons containing two hydrogens. The carbonyl group at position 11 is an alpha and beta unsaturated ketone formed by the Claisen-schmidt condensation of the parent nucleus of 3,4-dihydronaphthalen-1(2*H*)-one with 4-methoxy-3-(trifluoromethyl)benzaldehyde. In terms of the C(12)=C(11) olefin bond, the *E* stereochemical structure is formed [9]. Due to the distortion of 3,4-dihydronaphthalen-1(2*H*)-one, the 7-methoxy and 2-(trifluoromethyl)benzaldehyde groups are not coplanar with each other and their dihedral angle is about 69.5°. Bond lengths and angles are all in the expected ranges. The title molecular has an approximately linear structure [10,11].

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

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