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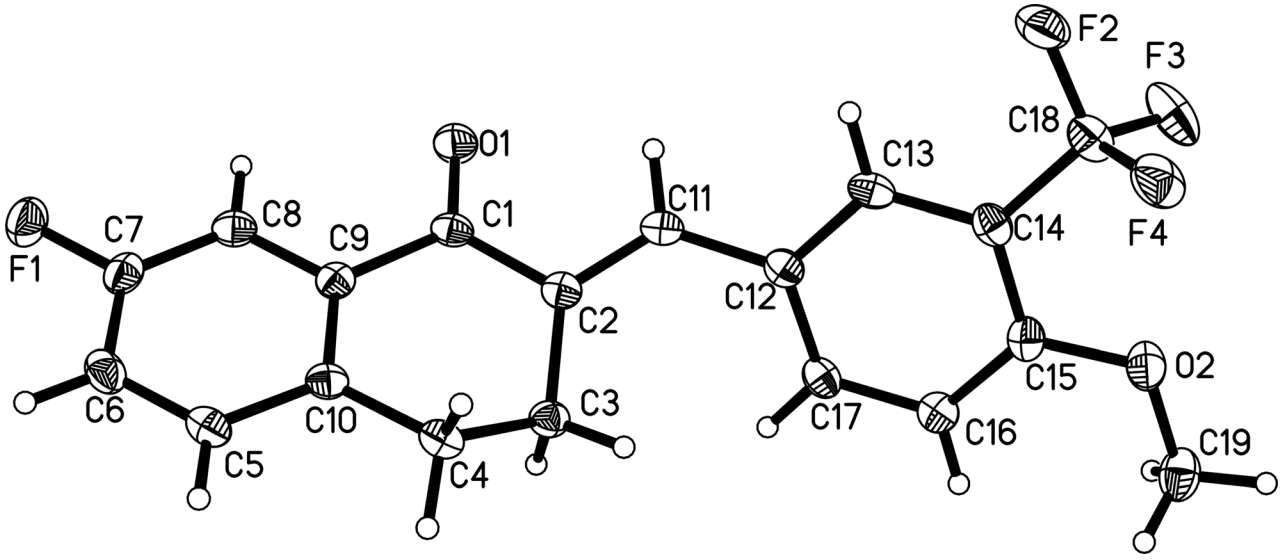
Crystal structure of *E*-7-fluoro-2-(4-methoxy-3-(trifluoromethyl)benzylidene)-3,4-dihydronaphthalen-1(2*H*)-one, $C_{19}H_{14}F_4O_2$



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Abstract

$C_{19}H_{14}F_4O_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.3798(6) Å, b = 9.2019(7) Å, c = 10.4504(7) Å, α = 77.941(6)°, β = 85.712(6) , γ = 79.078(6)°, V = 773.25(10) Å^3, Z = 2, $R_{gt}(F)$ = 0.0458, $wR_{ref}(F^2)$ = 0.1165, T = 100 K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.13 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	SuperNova
θ_{max}, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5133, 2883, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2295
$N(param)_{refined}$:	227
Programs:	Rigaku [1], SHELX [2, 3]

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The molecular structure is shown in the figure. Displacement ellipsoids are drawn at the 40% probability level. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

7-Fluoro-3,4-dihydronaphthalen-1(2*H*)-one (0.7 g, 4.26 mmol) and 4-methoxy-3-(trifluoromethyl)benzaldehyde (0.9 g, 4.41 mmol) were dissolved in 10 mL acetic acid. Then dry hydrogen chloride gas was flowed continuously into the

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1799 (2)	0.71520 (19)	0.54160 (17)	0.0236 (4)
C2	0.2767 (2)	0.66612 (18)	0.42792 (17)	0.0232 (4)
C3	0.4107 (2)	0.75193 (19)	0.37178 (17)	0.0250 (4)
H3A	0.448956	0.726761	0.287832	0.030*
H3B	0.501186	0.722321	0.429965	0.030*
C4	0.3504 (2)	0.92302 (19)	0.35392 (17)	0.0265 (4)
H4A	0.442375	0.974487	0.329762	0.032*
H4B	0.275737	0.955141	0.282688	0.032*
C5	0.2627 (2)	1.11316 (19)	0.49984 (18)	0.0281 (4)
H5	0.316336	1.180502	0.441458	0.034*
C6	0.1812 (2)	1.1580 (2)	0.60875 (19)	0.0304 (4)
H6	0.179257	1.254379	0.623963	0.036*
C7	0.1029 (2)	1.0566 (2)	0.69437 (18)	0.0288 (4)
C8	0.1018 (2)	0.9142 (2)	0.67446 (18)	0.0268 (4)
H8	0.046577	0.848716	0.733359	0.032*
C9	0.1846 (2)	0.86908 (18)	0.56486 (16)	0.0226 (4)
C10	0.2665 (2)	0.96876 (19)	0.47514 (17)	0.0232 (4)
C11	0.2350 (2)	0.55238 (19)	0.38274 (17)	0.0246 (4)
H11	0.142170	0.518023	0.422143	0.030*
C12	0.3159 (2)	0.47545 (18)	0.27996 (17)	0.0239 (4)
C13	0.2232 (2)	0.42274 (18)	0.19880 (17)	0.0263 (4)
H13	0.110562	0.439204	0.210267	0.032*
C14	0.2951 (2)	0.34704 (18)	0.10235 (17)	0.0255 (4)
C15	0.4663 (2)	0.31625 (18)	0.08632 (17)	0.0255 (4)
C16	0.5598 (2)	0.36569 (18)	0.16784 (17)	0.0263 (4)
H16	0.672644	0.345677	0.159092	0.032*
C17	0.4851 (2)	0.44432 (18)	0.26157 (17)	0.0249 (4)
H17	0.549463	0.477673	0.314208	0.030*
C18	0.1932 (2)	0.2952 (2)	0.01478 (19)	0.0326 (5)
C19	0.7015 (2)	0.1987 (2)	−0.02040 (19)	0.0357 (5)
H19A	0.748516	0.288537	−0.040339	0.054*
H19B	0.730044	0.142993	−0.089386	0.054*
H19C	0.742119	0.137356	0.060604	0.054*
F1	0.02173 (14)	1.10027 (12)	0.80187 (11)	0.0402 (3)
F2	0.03357 (14)	0.33078 (13)	0.04423 (11)	0.0433 (3)
F3	0.22496 (17)	0.14519 (12)	0.02381 (12)	0.0514 (4)
F4	0.21277 (15)	0.35684 (13)	−0.11192 (10)	0.0426 (3)
O1	0.09904 (16)	0.63236 (14)	0.61598 (12)	0.0332 (3)
O2	0.52819 (16)	0.23916 (14)	−0.00859 (13)	0.0332 (3)

solution lasting 45 min. After gas insertion, the reaction system was stirred at room temperature for five 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 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 = 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.

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).

Comment

Previous studies suggested that 3,4-dihydronaphthalen-1(2*H*)-ones (DHNs) might be developed as anti-neuroinflammatory drugs [4, 5]. Therefore, we structurally modified this class of compounds in the hope of improving their activity. In our study, a series of novel DHNs were synthesized by Claisen–Schmidt condensation reaction and we obtained a new single crystal by introducing trifluoromethyl.

Single-crystal structure analysis revealed that the title compound crystallized in the triclinic space group $P\bar{1}$. 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. The parent nucleus is 3,4-dihydronaphthalen-1(2*H*)-one. The third and fourth positions of the parent nucleus are two methylene saturated carbons. The olefinic double bonds between 3,4-dihydronaphthalen-1(2*H*)-one and 4-methoxy-3-(trifluoromethyl) benzaldehyde groups and carbonyl group form alpha and beta unsaturated ketones. With respect to the C(12) = C(11) olefinic bonds, 4-methoxy-3-(trifluoromethyl)benzaldehyde and carbonyl groups adopt the *E* stereochemistry [6, 7]. Because of the distorting effect of 3,4-dihydronaphthalen-1(2*H*)-one, the 7-fluorophenyl and 4-methoxy-3-(trifluoromethyl)benzaldehyde groups are not coplanar with each other, with a dihedral angle of approximately 62° [8]. This twisted configuration may increase the likelihood of interactions with bioactive molecules or the purposes of creating more potent biological activity [9, 10]. All geometric parameters are in the expected ranges.

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

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