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Crystal structure of (*E*)-7-methoxy-2-((2-methoxypyridin-3-yl)methylene)-3,4-dihydronaphthalen-1 (2*H*)-one, C₁₈H₁₇NO₃

Figure 1:

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Abstract

C₁₈H₁₇NO₃, triclinic, $P\bar{1}$ (no. 2), $a = 7.4638(8)$ Å, $b = 8.0392(8)$ Å, $c = 12.6767(11)$ Å, $\alpha = 108.187(8)^\circ$, $\beta = 94.029(7)^\circ$, $\gamma = 95.071(8)^\circ$, $V = 715.95(13)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0573$, $wR_{ref}(F^2) = 0.1460$, $T = 100.0(2)$ K.

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The crystal structure is shown in Figure 1. Displacement ellipsoids are drawn at the 40% 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:	Block, colourless
Size:	0.14 × 0.12 × 0.11 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	4744, 2669, 0.037
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2005
$N(param)_{refined}$:	201
Programs:	CyrsAlis ^{PRO} [1], SHELX [2, 3]

Source of material

Five milliliters of sodium hydroxide aqueous solution (25%) was added dropwise to the mixture of 7-methoxy-1-tetralone and 2-methoxynicotinaldehyde in 10 mL methanol and stirred at room temperature for 3 h. The in process-control was monitored by silica gel thin layer chromatography (TLC, 254 nm). When the reaction was finished, the precipitate was filtered from the reaction mixture and dissolved with ethyl acetone. The organic phase was washed with water and brine in turn, and dried over anhydrous sodium sulfate. After filtration and concentration *in vacuo* to yield a white solid, which was purified by silica-gel column chromatography (petroleum ether: ethyl acetate = 1:2, *v/v*). Suitable crystals of the title compound were obtained by recrystallization in dichloromethane and methanol (1:2, *v/v*) system and dried under *vacuo* at 75 °C for 3 h.

Experimental details

The H atoms were placed in idealized positions and treated as riding on their parent atoms, with $d(C-H) = 0.97$ 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

At present, 3,4-dihydronaphthalen-1(2*H*)-one (DHN) derivatives with anti-tumor and anti-inflammatory activities

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.4910 (3)	0.1203 (2)	0.86985 (15)	0.0217 (5)
C2	0.6695 (3)	0.1701 (2)	0.83660 (14)	0.0206 (5)
C3	0.8186 (3)	0.2572 (2)	0.92903 (14)	0.0230 (5)
H3A	0.916215	0.311075	0.899584	0.028*
H3B	0.866031	0.168400	0.956701	0.028*
C4	0.7525 (3)	0.3980 (2)	1.02558 (15)	0.0248 (5)
H4A	0.844833	0.433154	1.088648	0.030*
H4B	0.736184	0.500820	1.003028	0.030*
C5	0.5290 (3)	0.4166 (2)	1.16872 (15)	0.0252 (5)
H5	0.610263	0.503446	1.220032	0.030*
C6	0.3648 (3)	0.3714 (2)	1.20108 (15)	0.0242 (5)
H6	0.336584	0.427547	1.272925	0.029*
C7	0.2402 (3)	0.2406 (2)	1.12574 (14)	0.0223 (5)
C8	0.2868 (3)	0.1579 (2)	1.01896 (14)	0.0200 (5)
H8	0.205669	0.069967	0.968295	0.024*
C9	0.4529 (3)	0.2054 (2)	0.98746 (14)	0.0199 (5)
C10	0.5788 (3)	0.3375 (2)	1.06188 (14)	0.0219 (5)
C11	0.6814 (3)	0.1399 (2)	0.72671 (15)	0.0228 (5)
H11	0.576011	0.088912	0.679913	0.027*
C12	0.8379 (3)	0.1768 (2)	0.67181 (15)	0.0244 (5)
C13	0.8157 (3)	0.2338 (2)	0.57687 (15)	0.0255 (5)
C14	1.1129 (3)	0.2358 (3)	0.55114 (16)	0.0329 (5)
H14	1.206928	0.255827	0.510601	0.039*
C15	1.1523 (3)	0.1801 (3)	0.64078 (16)	0.0327 (6)
H15	1.269939	0.162890	0.660508	0.039*
C16	1.0129 (3)	0.1501 (2)	0.70131 (16)	0.0274 (5)
H16	1.036791	0.111721	0.762329	0.033*
C17	0.0170 (3)	0.2766 (3)	1.25498 (15)	0.0336 (6)
H17A	0.093695	0.256182	1.312615	0.050*
H17B	−0.105858	0.233022	1.258028	0.050*
H17C	0.025292	0.400834	1.265831	0.050*
C18	0.6192 (4)	0.3032 (3)	0.44516 (16)	0.0382 (6)
H18A	0.694018	0.410383	0.452263	0.057*
H18B	0.494655	0.318511	0.431122	0.057*
H18C	0.651981	0.209287	0.384248	0.057*
N1	0.9453 (3)	0.2634 (2)	0.51818 (13)	0.0315 (5)
O1	0.3738 (2)	0.01520 (17)	0.80264 (10)	0.0309 (4)
O2	0.0733 (2)	0.18705 (16)	1.14835 (10)	0.0259 (4)
O3	0.6447 (2)	0.26042 (17)	0.54699 (10)	0.0305 (4)

have been used as novel allergic and inflammatory responses modifiers [4, 5] and as a potential tyrosinase inhibitor for the treatment of pigmentation disorders [6]. However, DHN derivatives are rarely developed as anti-neuroinflammatory drugs. Thus the synthesis and crystal structure of novel benzylidene-substituted DHN derivatives with anti-neuroinflammatory activities are of great significance. Our group also synthesized some of these compounds in the early stage, and studied their anti-neuroinflammatory activity [7–9]. In this study, we designed and synthesized a new DHN derivative through Claisen–Schmidt condensation reaction.

Single-crystal structure analysis revealed that the title compound crystallized in the triclinic space group *P* $\bar{1}$. The ORTEP diagram is presented in Figure 1. Bond lengths and angles are all in the expected ranges [9–11]. There is only a drug molecule in the asymmetric unit. The C(2)=C(11) olefinic bond adopts the (*E*) stereochemistry [12]. Because of the distorting effect of 3,4-dihydronaphthalen-1(2*H*)-one, 7-methoxy and 2-methoxyphenyl groups they are not coplanar with each other. This twisted configuration may increase the likelihood of interactions with bioactive molecules, for the purposes of creating a more potent biological activity [13].

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