

Miao-Chang Liu, Xiao-Bo Huang and Deng-Ze Wu*

The crystal structure of 6-(benzofuran-2-yl)-2-oxo-4,5-diphenyl-3,4-dihydro-2H-pyran-3-carbonitrile, $C_{26}H_{17}NO_3$

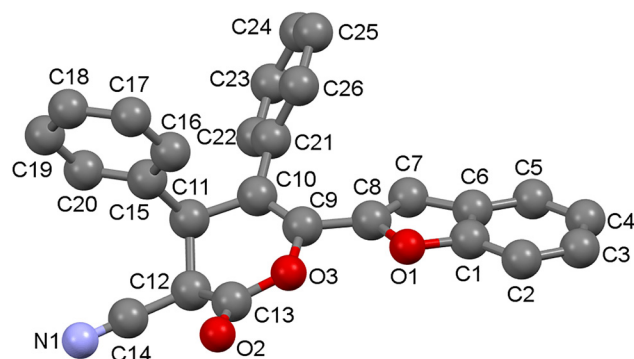


Table 1: Data collection and handling.

Crystal:	Colorless prismatic
Size:	0.11 × 0.08 × 0.04 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.0°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14,894, 3949, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3193
$N(\text{param})_{\text{refined}}$:	272
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

$C_{26}H_{17}NO_3$, monoclinic, Cc (no. 9), $a = 20.044(2)$ Å, $b = 10.7294(9)$ Å, $c = 9.4232(8)$ Å, $\beta = 91.689(3)^\circ$, $V = 2025.6(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0360$, $wR_{\text{ref}}(F^2) = 0.0814$, $T = 293(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

The title compound was prepared from the reaction of 8-(hexyloxy)-3,6-dimethyl-1-(piperidin-1-yl)isoquinoline-7-carbonitrile, which was obtained according to the published method [5], and selenium dioxide. The mixture of the

aforementioned carbonitrile (1.0 mmol), selenium dioxide (3.0 mmol), and xylene (15.0 mL) was stirred for 12 h at 100 °C. After the reaction solution was cooled to room temperature, it was extracted with dichloromethane. After removing the solvent under reduced pressure, the crude product was purified by column chromatography using petroleum ether and ethyl acetate to obtain the target product in 62 % yield as light yellow solid. Crystals were obtained by a slow evaporation from a mixture of ethyl acetate and petroleum ether.

2 Experimental details

H atoms bonded to C were positioned geometrically and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2 U_{\text{eq}}(\text{C})$ for all other H atoms.

3 Comment

In recent years, the design and synthesis of 4H-pyran derivatives substituted with multiple aromatic rings have received widespread attention because some of these compounds exhibited interesting solid-state photochromic property [6], mechanofluorochemical property [7, 8], and room – temperature phosphorescence activity [9]. The compounds usually have twisted molecular conformations and loose stacking arrangements in crystalline state, capable of emitting solid-state fluorescence. More importantly, their morphology is easily affected by external stimuli, exhibiting

*Corresponding author: **Deng-Ze Wu**, College of Chemistry and Materials Engineering, Wenzhou University, Chashan University Town, Wenzhou, Zhejiang Province, 325035, People's Republic of China, E-mail: wudengze@wzu.edu.cn. <https://orcid.org/0000-0003-1429-8197>

Miao-Chang Liu, Key Laboratory of Functional Small Organic Molecules, Ministry of Education, Jiangxi Normal University, 99 Ziyang Road, Nanchang, Jiangxi Province, 330022, People's Republic of China

Xiao-Bo Huang, College of Chemistry and Materials Engineering, Wenzhou University, Chashan University Town, Wenzhou, Zhejiang Province, 325035, People's Republic of China

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.40708 (10)	0.60784 (17)	0.41561 (18)	0.0474 (5)
O2	0.60827 (11)	0.60281 (18)	0.6462 (2)	0.0553 (6)
O3	0.52096 (10)	0.54054 (16)	0.51944 (18)	0.0448 (5)
N1	0.74342 (18)	0.4200 (4)	0.5970 (5)	0.1075 (14)
C1	0.35427 (15)	0.6411 (3)	0.3272 (3)	0.0468 (7)
C2	0.31607 (18)	0.7472 (3)	0.3414 (4)	0.0655 (9)
H2	0.3234	0.8038	0.4151	0.079*
C3	0.26651 (19)	0.7636 (3)	0.2391 (4)	0.0724 (10)
H3	0.2393	0.8336	0.2438	0.087*
C4	0.25574 (17)	0.6801 (4)	0.1301 (4)	0.0720 (10)
H4	0.2217	0.6953	0.0632	0.086*
C5	0.29429 (18)	0.5741 (3)	0.1176 (4)	0.0626 (9)
H5	0.2867	0.5180	0.0436	0.075*
C6	0.34529 (15)	0.5541 (3)	0.2206 (3)	0.0465 (7)
C7	0.39534 (15)	0.4612 (3)	0.2460 (3)	0.0467 (7)
H7	0.4021	0.3898	0.1924	0.056*
C8	0.43075 (15)	0.4967 (2)	0.3620 (3)	0.0405 (6)
C9	0.49056 (13)	0.4464 (2)	0.4339 (2)	0.0377 (6)
C10	0.51634 (13)	0.3328 (2)	0.4236 (2)	0.0367 (6)
C11	0.58616 (13)	0.3073 (2)	0.4798 (3)	0.0395 (6)
H11	0.6079	0.2540	0.4105	0.047*
C12	0.62443 (14)	0.4332 (3)	0.4843 (3)	0.0459 (7)
H12	0.6290	0.4607	0.3859	0.055*
C13	0.58575 (15)	0.5321 (2)	0.5591 (3)	0.0417 (6)
C14	0.69158 (18)	0.4246 (3)	0.5477 (4)	0.0633 (9)
C15	0.58871 (15)	0.2388 (2)	0.6216 (3)	0.0430 (7)
C16	0.54184 (16)	0.2597 (2)	0.7234 (3)	0.0499 (7)
H16	0.5073	0.3158	0.7050	0.060*
C17	0.54550 (19)	0.1981 (3)	0.8529 (3)	0.0606 (9)
H17	0.5138	0.2132	0.9209	0.073*
C18	0.5963 (2)	0.1147 (3)	0.8799 (4)	0.0689 (10)
H18	0.5991	0.0734	0.9667	0.083*
C19	0.6426 (2)	0.0925 (3)	0.7797 (4)	0.0750 (11)
H19	0.6768	0.0358	0.7987	0.090*
C20	0.63926 (17)	0.1533 (3)	0.6504 (3)	0.0600 (9)
H20	0.6709	0.1369	0.5827	0.072*
C21	0.47815 (14)	0.2280 (2)	0.3577 (3)	0.0398 (6)
C22	0.50224 (16)	0.1628 (2)	0.2439 (3)	0.0485 (7)
H22	0.5437	0.1830	0.2085	0.058*
C23	0.46498 (19)	0.0674 (3)	0.1820 (3)	0.0603 (9)
H23	0.4812	0.0248	0.1044	0.072*
C24	0.40431 (19)	0.0357 (3)	0.2349 (4)	0.0650 (9)
H24	0.3792	−0.0277	0.1922	0.078*
C25	0.38059 (19)	0.0970 (3)	0.3503 (4)	0.0647 (9)
H25	0.3399	0.0741	0.3873	0.078*
C26	0.41717 (15)	0.1932 (3)	0.4120 (3)	0.0513 (8)
H26	0.4008	0.2348	0.4903	0.062*

solid-state fluorescence response properties. Obviously, the crystalline structural characteristics of these compounds warrant further research to obtain more diverse 4H-pyran-based organic materials. In the title compound, the 4-position

benzene ring is perpendicular to the 5-position benzene ring, and the dihedral angle between the 5-position benzene ring and the 6-position benzofuran unit is 66°. Due to incomplete conjugation, the atoms of the central 3,4-dihydro-2H-pyran-2-one ring are not in the same plane. The results indicate that the molecule has a twisted molecular conformation, which is similar to previous reports in the literature [6–9]. As a result, no obvious π – π stacking is observed even though in the presence of multiple aromatic rings. In the same column, the molecules in the upper and lower layers are connected to each other through C12–H12···O2 (2.384 Å), C7–H7···O1 (2.613 Å), and C22–H22···O2 (2.710 Å) hydrogen bonds. The molecules in different columns are linked by C2–H2···N1 (2.688 Å) hydrogen bond.

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