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The crystal structure of 5-(benzofuran-2-carbonyl)-*N*-cyclohexyl-5,6-dihydrophenanthridine-6-carboxamide, C₂₉H₂₆N₂O₃

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.47 × 0.20 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Rigaku RAXIS-RAPID/ZJUG,
$\theta_{\max}$, completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10225, 4589, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2289
$N(\text{param})_{\text{refined}}$:	308
Programs:	Rigaku [1, 2], Olex2 [3], SHELX [4]

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Abstract

C₂₉H₂₆N₂O₃, triclinic, $P\bar{1}$ (no. 2), $a = 9.6533(8)$ Å, $b = 11.3185(9)$ Å, $c = 11.4469(10)$ Å, $\alpha = 83.747(3)^\circ$, $\beta = 85.810(2)^\circ$, $\gamma = 71.445(2)^\circ$, $V = 1177.65(17)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0464$, $wR_{\text{ref}}(F^2) = 0.1621$, $T = 296(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of palladium acetate (10 mol%), tricyclohexylphosphonium tetrafluoroborate (20 mol%), pivalic acid (30 mol%), potassium carbonate (0.40 mmol), and the benzofuran-2-carboxamide (0.2 mmol) [5] in degassed anhydrous dimethylacetamide (4 mL) was heated at 383 K for 30 min. After cooling, filtrating, and evaporation of the

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7193(3)	0.3691(2)	0.4273(2)	0.0528(6)
H1	0.620574	0.397972	0.463464	0.063*
C2	0.7379(3)	0.2449(2)	0.3798(2)	0.0540(6)
C3	0.6188(3)	0.2226(3)	0.3376(2)	0.0666(8)
H3	0.528150	0.284316	0.337126	0.080*
C4	0.6342(4)	0.1081(3)	0.2957(3)	0.0761(9)
H4	0.554542	0.093278	0.266344	0.091*
C5	0.7680(4)	0.0172(3)	0.2982(3)	0.0780(9)
H5	0.777854	−0.059982	0.271657	0.094*
C6	0.8875(4)	0.0381(2)	0.3390(2)	0.0723(9)
H6	0.977341	−0.024702	0.339781	0.087*
C7	0.8750(3)	0.1534(2)	0.3795(2)	0.0589(7)
C8	0.9990(3)	0.1822(2)	0.4235(2)	0.0604(7)
C9	1.1450(4)	0.1172(3)	0.3950(3)	0.0774(9)
H9	1.166010	0.050703	0.348473	0.093*
C10	1.2577(4)	0.1496(4)	0.4344(3)	0.0875(10)
H10	1.353965	0.104071	0.415911	0.105*
C11	1.2283(4)	0.2498(3)	0.5013(3)	0.0824(9)
H11	1.305134	0.272139	0.526742	0.099*
C12	1.0857(3)	0.3172(3)	0.5309(2)	0.0682(8)
H12	1.066101	0.385245	0.575148	0.082*
C13	0.9721(3)	0.2818(2)	0.4935(2)	0.0561(7)
C14	0.7347(3)	0.4714(2)	0.3319(2)	0.0549(7)
C15	0.5933(3)	0.6434(2)	0.1937(3)	0.0703(8)
H15	0.689438	0.654453	0.175620	0.084*
C16	0.4903(4)	0.7640(3)	0.2369(3)	0.0906(10)
H16A	0.396789	0.752428	0.261437	0.109*
H16B	0.530488	0.784018	0.304572	0.109*
C17	0.4671(5)	0.8720(3)	0.1416(3)	0.1013(12)
H17A	0.397412	0.946568	0.170504	0.122*

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H17B	0.558879	0.889053	0.122237	0.122*
C18	0.4114(4)	0.8418(3)	0.0332(3)	0.0993(12)
H18A	0.400693	0.910656	−0.027471	0.119*
H18B	0.315907	0.831478	0.051086	0.119*
C19	0.5160(5)	0.7224(3)	−0.0119(3)	0.1071(13)
H19A	0.475968	0.702483	−0.079744	0.128*
H19B	0.608873	0.735241	−0.036371	0.128*
C20	0.5400(4)	0.6144(3)	0.0830(3)	0.0821(9)
H20A	0.448914	0.595889	0.101041	0.099*
H20B	0.611192	0.540682	0.054054	0.099*
C21	0.7737(3)	0.4173(2)	0.6149(2)	0.0535(6)
C22	0.8462(3)	0.3807(2)	0.7271(2)	0.0538(6)
C23	0.8344(3)	0.4501(2)	0.8174(2)	0.0630(7)
H23	0.786349	0.535456	0.817775	0.076*
C24	0.9094(3)	0.3686(2)	0.9131(2)	0.0594(7)
C25	0.9331(4)	0.3802(3)	1.0295(3)	0.0771(9)
H25	0.897533	0.457302	1.060550	0.093*
C26	1.0098(4)	0.2755(3)	1.0972(3)	0.0866(10)
H26	1.027877	0.282163	1.174547	0.104*
C27	1.0608(4)	0.1601(3)	1.0523(3)	0.0932(11)
H27	1.111638	0.090570	1.100647	0.112*
C28	1.0385(4)	0.1451(3)	0.9378(3)	0.0838(10)
H28	1.072830	0.067549	0.907514	0.101*
C29	0.9625(3)	0.2515(3)	0.8714(2)	0.0616(7)
N1	0.8233(2)	0.35037(17)	0.51976(17)	0.0524(5)
N2	0.6093(3)	0.5406(2)	0.2851(2)	0.0734(7)
H2	0.531434	0.523142	0.310794	0.088*
O1	0.8528(2)	0.48586(17)	0.30337(17)	0.0725(6)
O2	0.6576(2)	0.50567(15)	0.61117(16)	0.0658(5)
O3	0.9256(2)	0.25668(15)	0.75639(15)	0.0637(5)

solvent, the residue was purified by column chromatography on silica gel eluted with ethyl acetate and petroleum ether (333–363 K) to afford the title compound in 73% yield. The needle crystals were obtained by slow evaporation of the solution at room temperature.

Experimental details

H atoms were placed in calculated positions with C–H = 0.93–0.98 Å and N–H = 0.86 Å and refined as riding with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom.

Comment

Dihydrophenanthridines are prevalent structural units in many natural products and biologically active molecules [6], and their syntheses have attracted much attention [7, 8]. As a continuation of our work on post cyclization of the Ugi/Rap-Stoermer reaction product [5, 9], here a novel dihydrophenanthridine was designed and synthesized. Similar to the related dihydrophenanthridine [10], the titled compound substituted

with benzofuran-2-carbonyl moiety was synthesized via a Pd-catalyzed intramolecular direct arylation [11].

The crystal structure analysis revealed that geometric parameters of the title compound (*cf.* the figure) are in normal ranges. The title crystal structure contains a benzofuran moiety p1 (C22–C29 and O3), biphenylmoiety rings p2 (C2–C7), p3 (C8–C13), and a cyclohexyl ring p4 (C15–C20) exhibiting chair conformation. The dihedral angles between them are as follows: p1 and p2: 53.8°, p1 and p3: 64.5°, p2 and p3: 19.4°. In the title structure, there is an intermolecular N2–H2···O2ⁱⁱ (1 – x, 1 – y, 1 – z) hydrogen bond, through which the title compound dimerizes with its adjacent molecule.

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