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Crystal structure of 6-(quinolin-8-yl)benzo[*a*]phenanthridin-5(6*H*)-one, C₂₆H₁₆N₂O

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Abstract

C₂₆H₁₆N₂O, monoclinic, *P*₂/c (no. 14), *a* = 12.3329(2) Å, *b* = 12.9276(2) Å, *c* = 12.11250(10) Å, β = 114.047(2)°, *V* = 1763.55(5) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0416, *wR*_{ref}(*F*²) = 0.1137, *T* = 293(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

All reagents used in this paper were dried before use. The title compound was synthesized according to the reference [5] with slight modification: 49.6 mg *N*-(quinolin-8-yl)benzamide (0.2 mmol), 72 mg anhydrous copper acetate (0.4 mmol), 37 mg tetrabutylammonium iodide (0.1 mmol),

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.21 × 0.19 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ:	0.68 mm ^{−1}
Diffraction, scan mode:	SuperNova, ω
θ _{max} , completeness:	70.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	6573, 3340, 0.012
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2961
<i>N</i> (<i>param</i>) _{refined} :	262
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

365 mg CsF (0.24 mmol) and 139.2 mg 2-(trimethylsilyl)naphthalen-1-yl trifluoromethanesulfonate (0.4 mmol) were added in 2 mL DMF/MeCN (V/V 1:1) in a 10 mL glass vial, purged with O₂, sealed with PTFE cap, and heated at 353 K for 12 h, then cooled to room temperature. Crystals were obtained from the reaction solution after 24 h, yield 83% (based on *N*-(quinolin-8-yl)benzamide).

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms were positioned with idealized geometry and refined isotropically (*U*_{iso}(H) = 1.2*U*_{eq}(C)) using a riding model with C–H = 0.930 Å.

Comment

Phenanthridinones and their derivatives have attracted a wide attention because they are known as important core units of alkaloids and biologically active pharmaceutical agents. It's an attractive new strategy to synthesize heterocyclic molecules through selective conversion of unactivated C–H bonds by transition-metal catalysts such as Pd(II) [6–9] and Cu(II) [5, 10–13], or electrochemical method [14] and photocycloaddition [15].

Reacting *N*-(quinolin-8-yl)benzamide and 2-(trimethylsilyl)naphthalen-1-yl trifluoromethanesulfonate using anhydrous copper acetate as catalyst, yield the title compound QBPO in high yield. All atoms from the 8-hydroxyquinolinyl group are nearly co-planar, while

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.30680 (9)	0.44331 (9)	0.80847 (10)	0.0407 (3)
N2	0.21945 (9)	0.50352 (9)	0.56671 (10)	0.0428 (3)
O1	0.17358 (9)	0.32127 (9)	0.80055 (12)	0.0675 (3)
C1	0.16883 (10)	0.54492 (10)	0.63673 (11)	0.0371 (3)
C2	0.51309 (11)	0.39959 (10)	0.86680 (11)	0.0386 (3)
C3	0.21133 (11)	0.51618 (10)	0.75907 (12)	0.0400 (3)
C4	0.42169 (11)	0.47113 (10)	0.82500 (11)	0.0384 (3)
C5	0.07444 (11)	0.61624 (10)	0.59228 (12)	0.0407 (3)
C6	0.49267 (11)	0.30483 (10)	0.91896 (11)	0.0398 (3)
C7	0.37647 (12)	0.27891 (10)	0.89906 (12)	0.0427 (3)
C8	0.62358 (11)	0.42688 (11)	0.86175 (11)	0.0408 (3)
C9	0.44303 (12)	0.57344 (11)	0.80188 (12)	0.0447 (3)
H9	0.381639	0.621526	0.778225	0.054*
C10	0.03247 (12)	0.64374 (11)	0.46997 (13)	0.0486 (3)
H10	−0.030498	0.689780	0.436581	0.058*
C11	0.64410 (12)	0.53014 (12)	0.83989 (12)	0.0455 (3)
C12	0.58305 (12)	0.24185 (11)	0.99903 (12)	0.0464 (3)
H12	0.661929	0.258294	1.017351	0.056*
C13	0.27695 (12)	0.34609 (11)	0.83094 (12)	0.0460 (3)
C14	0.02616 (12)	0.65623 (11)	0.66957 (14)	0.0487 (3)
H14	−0.036164	0.703321	0.640367	0.058*
C15	0.17698 (12)	0.53367 (12)	0.45399 (13)	0.0477 (3)
H15	0.211784	0.506554	0.405108	0.057*
C16	0.55197 (12)	0.60261 (11)	0.81371 (13)	0.0489 (3)
H16	0.566556	0.671943	0.804391	0.059*
C17	0.08413 (13)	0.60289 (12)	0.40148 (13)	0.0515 (4)
H17	0.058125	0.620704	0.320344	0.062*
C18	0.16299 (13)	0.55550 (12)	0.83133 (13)	0.0485 (3)
H18	0.191813	0.535168	0.911886	0.058*
C19	0.71204 (13)	0.35445 (13)	0.87008 (12)	0.0495 (3)
H19	0.698453	0.284458	0.876898	0.059*
C20	0.06982 (14)	0.62663 (12)	0.78659 (15)	0.0534 (4)
H20	0.037664	0.653784	0.837759	0.064*
C21	0.35198 (13)	0.19202 (12)	0.95187 (14)	0.0531 (4)
H21	0.273690	0.175449	0.936192	0.064*
C22	0.55718 (14)	0.15691 (12)	1.05045 (14)	0.0536 (4)
H22	0.618705	0.115931	1.102616	0.064*
C23	0.44158 (14)	0.13074 (12)	1.02651 (15)	0.0564 (4)
H23	0.424899	0.071888	1.060929	0.068*
C24	0.75475 (14)	0.55901 (14)	0.84187 (12)	0.0577 (4)
H24	0.769456	0.628196	0.831982	0.069*
C25	0.81667 (14)	0.38492 (15)	0.86833 (15)	0.0594 (4)
H25	0.873578	0.335486	0.874282	0.071*
C26	0.84000 (15)	0.48791 (15)	0.85789 (16)	0.0632 (4)
H26	0.913845	0.508210	0.861855	0.076*

the quinolinyl moiety is turned out from this plane by a rotation about the N1-C3 bond (see the Figure). All the bond lengths of QBPO are similar to their analogues [5].

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