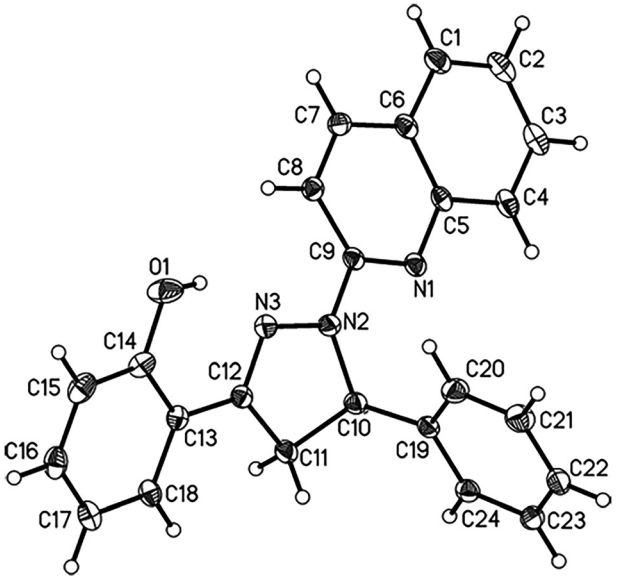
Dai Zeng, Kai Tan, Ziyan Fang and Aiping Xing*

Crystal structure of 2-(5-phenyl-1-(quinolin-2-yl)-4,5-dihydro-1*H*-pyrazol-3-yl)phenol, $C_{24}H_{19}N_3O$



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Abstract

$C_{24}H_{19}N_3O$, monoclinic, $P2_1/c$ (no. 14), a = 10.4783(5) Å, b = 11.7171(5) Å, c = 14.9194(6) Å, β = 93.991(2)°, Z = 4, V = 1827.29(14) Å^3, $R_{gt}(F)$ = 0.0521, $wR_{ref}(F^2)$ = 0.1508, T = 150(2) K.

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The asymmetric unit of 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.

***Corresponding author: Aiping Xing**, Henan University of Chinese Medicine, Zhengzhou 450046, P.R. China, E-mail: hnxingaiping@126.com. https://orcid.org/0000-0002-3173-7430
Dai Zeng, Kai Tan and Ziyan Fang, Henan University of Chinese Medicine, Zhengzhou 450046, P.R. China

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.21 × 0.21 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX II, ω
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16,737, 4165, 0.058
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3391
$N(param)_{refined}$:	254
Programs:	Bruker [1], SHELX [2–4]

1 Source of materials

All chemicals were of reagent grade and used without further purification. The title compound was prepared following a literature procedure [5].

The intermediate is synthesized through aldol condensation reaction. 2-Hydroxy acetophenone (5.25 mmol) and benzaldehyde (5 mmol) were added to a 100 mL round-bottom flask along with 20 mL ethanol (EtOH). The mixture was stirred at room temperature for 10 min. An aqueous solution (2 mL) of NaOH (5 mmol) was then added drop wise. Subsequently, the mixture was agitated and allowed to stand at room temperature for additional 10 h. After completion, the reaction mixture was poured into ice cold water and neutralized with diluted HCl. Finally, the precipitated solid was filtered, washed with excess of cold water, dried and then recrystallized from ethanol to obtain the 2′-hydroxychalcone as yellow crystals. Yield: ca. 86 %.

The second step of synthetic procedure is as follows: A mixture of 2′-hydroxychalcone (10 mmol) and 2-hydrazinoquinoline (10 mmol) was dissolved in ethanol (30 ml) containing sodium hydroxide (30 mmol) and then refluxed for 4 h on an oil-bath. Completion of the reaction was confirmed by TLC. Five milliliter H_2O were added and the reaction mixture was further stirred for 5 min. The above mixture was neutralized with diluted HCl. The orange yellow precipitates were isolated by filtration and recrystallized from ethanol. Yield: ca. 81 %. Suitable crystals of the title compound were grown from the mixed solution of ethanol and DMF by slow evaporation at room temperature.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.03398 (15)	−0.07204 (13)	0.67616 (9)	0.0363 (3)
H1	0.062716	−0.148579	0.683588	0.044*
C2	−0.06060 (15)	−0.03185 (15)	0.72672 (10)	0.0413 (4)
H2	−0.096828	−0.080328	0.769236	0.050*
C3	−0.10401 (15)	0.08103 (15)	0.71570 (10)	0.0404 (4)
H3	−0.170155	0.108159	0.750592	0.049*
C4	−0.05199 (14)	0.15283 (14)	0.65503 (9)	0.0340 (3)
H4	−0.082184	0.229043	0.648630	0.041*
C5	0.04618 (13)	0.11395 (12)	0.60210 (8)	0.0274 (3)
C6	0.08931 (13)	−0.00042 (12)	0.61288 (8)	0.0287 (3)
C7	0.18571 (13)	−0.03735 (11)	0.55713 (9)	0.0294 (3)
H7	0.216026	−0.113715	0.561185	0.035*
C8	0.23475 (13)	0.03607 (11)	0.49802 (9)	0.0272 (3)
H8	0.299106	0.012112	0.460326	0.033*
C9	0.18704 (12)	0.15010 (11)	0.49406 (8)	0.0248 (3)
C10	0.17755 (13)	0.33883 (11)	0.41141 (8)	0.0273 (3)
H10	0.082264	0.331514	0.405260	0.033*
C11	0.22931 (13)	0.35856 (12)	0.31828 (9)	0.0303 (3)
H11A	0.162265	0.344883	0.269386	0.036*
H11B	0.263078	0.436977	0.312805	0.036*
C12	0.33452 (12)	0.27128 (11)	0.31697 (8)	0.0248 (3)
C13	0.42898 (12)	0.26678 (11)	0.25005 (9)	0.0261 (3)
C14	0.53215 (13)	0.18893 (12)	0.25635 (10)	0.0329 (3)
C15	0.62124 (14)	0.18941 (13)	0.19190 (12)	0.0409 (4)
H15	0.691157	0.137646	0.196868	0.049*
C16	0.60912 (14)	0.26466 (13)	0.12044 (11)	0.0386 (4)
H16	0.670182	0.263616	0.076270	0.046*
C17	0.50872 (14)	0.34143 (13)	0.11281 (10)	0.0350 (3)
H17	0.500725	0.392986	0.063596	0.042*
C18	0.41994 (13)	0.34274 (12)	0.17732 (9)	0.0299 (3)
H18	0.351585	0.396124	0.172185	0.036*
C19	0.21608 (12)	0.43379 (11)	0.47688 (8)	0.0259 (3)
C20	0.32214 (15)	0.42667 (13)	0.53781 (10)	0.0370 (3)
H20	0.370847	0.358272	0.542364	0.044*
C21	0.35727 (16)	0.51964 (14)	0.59228 (11)	0.0426 (4)
H21	0.430532	0.514630	0.633336	0.051*
C22	0.28623 (15)	0.61915 (13)	0.58699 (10)	0.0367 (3)
H22	0.310240	0.682138	0.624555	0.044*
C23	0.17973 (14)	0.62663 (12)	0.52661 (10)	0.0330 (3)
H23	0.130452	0.694733	0.522873	0.040*
C24	0.14526 (13)	0.53458 (11)	0.47169 (9)	0.0287 (3)
H24	0.072643	0.540280	0.430111	0.034*
N1	0.09585 (10)	0.18889 (9)	0.54289 (7)	0.0268 (3)
N2	0.23597 (11)	0.22764 (9)	0.43640 (7)	0.0279 (3)
N3	0.33205 (10)	0.19781 (9)	0.38185 (7)	0.0263 (3)
O1	0.54848 (11)	0.11343 (10)	0.32507 (9)	0.0489 (3)
H1A	0.486348	0.117934	0.357564	0.073*

2 Experimental details

The H atoms were added using riding models. Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms.

3 Comment

The construction of 1,3,5-trisubstituted pyrazolines has attracted great attention in recent years due to their wide applications in pharmaceuticals, fluorescent probes and electroluminescent materials [6–10]. In view of the structural diversity and important significance of 1,3,5-trisubstituted pyrazolines, research involving single crystal structures is of great significance to reveal the structure-activity relationship. The title compound 2-(5-phenyl-1-(quinolin-2-yl)-4,5-dihydro-1*H*-pyrazol-3-yl)phenol bearing both pyrazoline and quinoline moieties belongs to an important kind of 1, 3, 5-trisubstituted pyrazoline compounds.

The title compound crystallizes in the monoclinic space group $P2_1/c$ with one molecule in the asymmetric unit (see the Figure). Bond lengths and angles are all in the normal range. A pyrazoline group as the five-membered core ring is formed by addition cyclization reaction of chalcone and hydrazine. The bond length of C12–N3 (1.2970 Å) indicates the existence of C=N in the pyrazoline ring, which is typical for similar compounds [11–13]. The pyrazoline ring is close to being planar with the largest deviation from the mean plane being 0.0883° for atom C10. In addition to the pyrazoline ring, it is substituted by a quinoline group, and a phenyl group and a phenol-2-yl group. The pyrazoline ring makes dihedral angles of 9.9° and 19.2° with the phenol and quinoline rings, respectively. Due to the steric hindrance effect, the benzene ring is almost perpendicular to the attached pyrazoline ring, showing a dihedral angle of approximately 103.9°. Phenolic hydroxyl group forms an intramolecular hydrogen bond to the pyrazoline N3 atom [O1···N3: 2.6656(16) Å]. $\pi\cdots\pi$ interactions are observed for parallel quinoline rings with the centroid-centroid distance between the two pyridine moieties of 3.66 Å.

Research ethics: This study did not involve any animal experiments.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Competing interests: The authors declare no conflicts of interest regarding this article.

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Data availability: All relevent data are within the paper.

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