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Crystal structure of 5-((bis(pyridin-2-ylmethyl)amino)methyl)quinolin-8-ol, $C_{22}H_{20}N_4O$

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Abstract

$C_{22}H_{20}N_4O$, monoclinic, $P2_1/n$ (no. 14), $a = 11.1861(14)$ Å, $b = 8.1418(10)$ Å, $c = 20.461(3)$ Å, $\beta = 97.433(1)^\circ$, $V = 1847.8(4)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0357$, $wR_{ref}(F^2) = 0.1002$, $T = 296(2)$ 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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.22 \times 0.20 \times 0.18$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.5° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13705, 3436, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2957
$N(\text{param})_{\text{refined}}$:	246
Programs:	Bruker [1], SHELX [2, 4], Diamond [3]

Source of material

To a mixture of 8-hydroxyquinoline-5-carbaldehyde (1.04 g, 6 mmol) and bis(pyridin-2-ylmethyl)amine (1.22 g, 6 mmol) in CH_2Cl_2 (50 mL) was added sodium triacetoxyborohydride (1.4 g, 6.6 mmol) at 0°C . The mixture was stirred for 24 h at room temperature. Dichloromethane was removed by evaporation and the residue was purified by chromatography on silica gel (eluent: ethyl acetate) to give the yellow solid [5, 6]. m.p. 102°C – 106°C ; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ [ppm] 8.78–8.73 (m, 1H), 8.65–8.57 (m, 1H), 8.56–8.51 (m, 2H), 7.67–7.60 (m, 2H), 7.55–7.49 (m, 1H), 7.48–7.42 (m, 1H), 7.41–7.35 (m, 2H), 7.21–7.13 (m, 2H), 7.06 (d, $J = 7.8$ Hz, 1H), 4.25–4.14 (m, 2H), 4.06–3.95 (m, 4H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ [ppm] 158.07, 152.08, 148.51, 147.48, 138.48, 136.81, 133.99, 129.86, 127.77, 124.02, 123.65, 122.31, 121.53, 108.94, 59.681, 56.29.

Experimental details

All H atoms were included in calculated positions and refined with $\text{O}–\text{H} = 0.82$ Å with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{O})$, with $\text{C}–\text{H} = 0.93$ Å 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 [4].

Comment

The tailorability of hydroquinoline (HQ) arises from the activation of positions 5 and 7 and leads to numerous HQ-based

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}
C1	−0.06848(10)	0.98768(15)	0.09543(6)	0.0360(3)
C2	−0.13990(10)	0.84683(16)	0.07603(6)	0.0368(3)
C3	−0.08755(11)	0.71292(17)	0.05185(7)	0.0428(3)
H3	−0.1340	0.6208	0.0392	0.051*
C4	0.03643(11)	0.71287(16)	0.04585(6)	0.0418(3)
H4	0.0702	0.6194	0.0295	0.050*
C5	0.10890(10)	0.84489(16)	0.06307(6)	0.0367(3)
C6	0.05632(10)	0.98692(15)	0.08864(6)	0.0355(3)
C7	0.12055(12)	1.13266(17)	0.10691(7)	0.0446(3)
H7	0.2022	1.1397	0.1028	0.054*
C8	0.06307(13)	1.26273(18)	0.13053(8)	0.0556(4)
H8	0.1048	1.3591	0.1427	0.067*
C9	−0.05961(14)	1.24927(19)	0.13625(9)	0.0594(4)
H9	−0.0974	1.3387	0.1531	0.071*
C10	0.24011(10)	0.84430(17)	0.05288(6)	0.0405(3)
H10A	0.2579	0.7432	0.0310	0.049*
H10B	0.2551	0.9351	0.0243	0.049*
C11	0.43829(11)	0.92599(17)	0.10635(6)	0.0405(3)
H11A	0.4278	1.0070	0.0713	0.049*
H11B	0.4885	0.8386	0.0927	0.049*
C12	0.50203(11)	1.00515(16)	0.16792(6)	0.0391(3)
C13	0.44034(13)	1.0796(2)	0.21454(7)	0.0526(4)
H13	0.3566	1.0760	0.2102	0.063*
C14	0.50437(15)	1.1587(2)	0.26718(8)	0.0624(4)
H14	0.4645	1.2101	0.2987	0.075*
C15	0.62819(15)	1.1609(2)	0.27255(8)	0.0621(4)
H15	0.6738	1.2129	0.3078	0.074*
C16	0.68226(13)	1.0846(2)	0.22464(7)	0.0575(4)
H16	0.7660	1.0863	0.2283	0.069*
C17	0.32884(12)	0.70665(18)	0.15481(7)	0.0456(3)
H17A	0.2478	0.6670	0.1574	0.055*
H17B	0.3651	0.7325	0.1993	0.055*
C18	0.40046(12)	0.57078(17)	0.12827(7)	0.0455(3)
C19	0.52143(14)	0.5511(2)	0.15003(10)	0.0662(5)
H19	0.5588	0.6161	0.1841	0.079*
C20	0.58645(16)	0.4348(2)	0.12099(12)	0.0790(6)
H20	0.6682	0.4203	0.1351	0.095*
C21	0.52980(17)	0.3411(2)	0.07140(10)	0.0687(5)
H21	0.5720	0.2624	0.0506	0.082*
C22	0.40921(17)	0.3655(2)	0.05279(9)	0.0661(4)
H22	0.3706	0.3005	0.0191	0.079*
N1	−0.12520(10)	1.11830(15)	0.11940(6)	0.0490(3)
N2	0.32105(9)	0.85852(13)	0.11536(5)	0.0387(3)
N3	0.62209(9)	1.00775(15)	0.17292(5)	0.0458(3)
N4	0.34360(11)	0.47737(15)	0.08026(7)	0.0570(3)
O1	−0.25972(7)	0.84404(12)	0.08057(5)	0.0479(3)
H1	−0.2762	0.9176	0.1053	0.072*

derivatives [7–11]. In this article we report the synthesis and crystal structure of 5-((bis(pyridin-2-ylmethyl)amino)methyl)quinolin-8-ol, which is a fluorescence probe.

There is one crystallographic independent molecule in the asymmetric unit (see the figure). In the molecule of the

title compound bond lengths and angles are very similar to those given in the literature [11]. The quinoline ring and two pyridine rings are connected to the same nitrogen atom (N2) through the methylene group. The dihedral angles of quinoline ring and two pyridine rings to each other are 32.3°, 37.3° and 20.7°. The molecular conformation is characterized by the C5–C10–N2–C11, C18–C12–N2–C10 and C13–C11–N2–C10, torsion angles of 155.8°, 73.4° and 156.5° respectively.

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References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
4. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr. C* **71** (2015) 3–8.
5. Lim, N. C.; Schuster, J. V.; Porto, M. C.; Tanudra, M. A.; Yao, L. L.; Freake, H. C.; Brückner, C.: Coumarin-based chemosensors for zinc(II): toward the determination of the design algorithm for cheif-type and ratiometric probes. *Inorg. Chem.* **44** (2005) 2018–2030.
6. Xie, X.; Jiang, X. J.; Liu, J.; Ren, X. Y.; Wang, H. M.; Liu, X. M.: A novel ditopic ligand derived from 8-hydroxyquinoline: synthesis, characterisation, and its coordination chemistry with selected metal ions. *Inorg. Chim. Acta* **383** (2012) 132–136.
7. Mei, Y.; Bentley, P. A.; Wang, W.: A selective and sensitive chemosensor for Cu²⁺ based on 8-hydroxyquinoline. *Tetrahedron Lett.* **47** (2006) 2447–2449.
8. Prachayasittikul, V.; Prachayasittikul, S.; Ruchirawat, S.; Prachayasittikul, V.: 8-hydroxyquinolines: a review of their metal chelating properties and medicinal applications. *Drug. Des. Dev. Ther.* **7** (2013) 1157–1178.
9. Ghedini, M.; Deda, M. L.; Aiello, I.; Grisolia, A.: Fine-tuning the luminescent properties of metal-chelating 8-hydroxyquinolines through amido substituents in 5-position. *Inorg. Chim. Acta* **357** (2004) 33–40.
10. Shah, B. A.; Shah, A. V.; Bhandari, B. N.; Bhatt, R. R.: Synthesis, characterization and chelation ion-exchange studies

of a resin copolymer derived from 8-hydroxyquinoline-formaldehyde-catechol. *J. Iran. Chem. Soc.* **5** (2008) 252–261.

11. Song, X. Q.; Cheng, G. Q.; Liu, Y. A.: Enhanced Tb(III) luminescence by d^{10} transition metal coordination. *Inorg. Chim. Acta* **450** (2016) 386–394.