Synthesis and Crystal Structure of 5-chloroquinolin-8-yl Acrylate

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Abstract. In this paper, a new Quinoline derivative (**probe 1**) was synthesized. **Probe 1** was characterized by 1H NMR and their structures determined by single crystal X-ray diffraction. Single crystal X-ray diffraction (SCXRD) analysis revealed that **probe 1** crystallized in the monoclinic space group P21/c. There is one ketone carbonyl, one double bond and pyridine ring in this structure. CCDC no.: 2394064.

Keywords: Crystal structure, molecular design, quinoline.

1. Source of materials

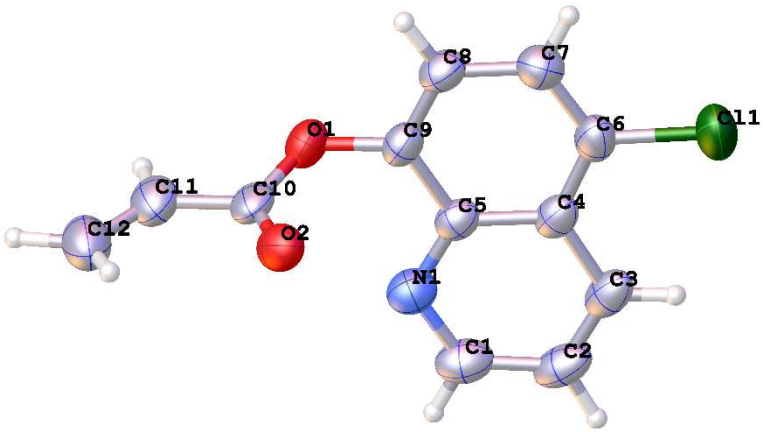

Figure 1. The crystal structure of **probe 1.**

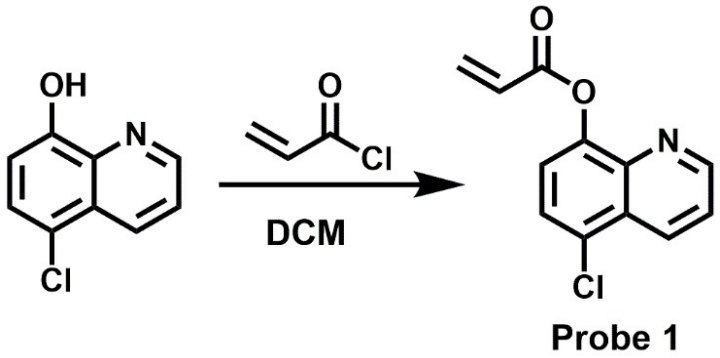

Figure 2. The crystal structure of **probe 1**.

The 5-chloro-8-hydroxyquinoline and acryloyl chloride were commercial purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. In a round-bottomed flask, 5-chloro-8-hydroxyquinoline (1.0 mmol) was dissolved by methylene chloride (20 ml), and then acryloyl chloride was slowly added to the flask under ice bath. The resulting solution is stirred 3 hours at 0° C, then 2 hours at ambient temperature. After the solvent was removed by vacuum distillation, the crude product was separated by silica gel chromatographic column (petroleum ether: ethyl acetate,

10 : 1 by volume) to obtain the yellow compound with a yield of 45%. Subsequently, the title compound was further recrystallized with methylene chloride at room temperature by evaporation.

2. Experimental

Table 1. Data collection and handling.

Crystal:	Colourless needle
Size:	0.25 × 0.21 × 0.2 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.338 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int:}$	19725, 2440, 0.0505
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2144
$N(param)_{refined}$:	153
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond[4]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Cl1	0.84401(10)	0.7059(3)	0.53546(11)	0.0787(3)
O2	0.8505(2)	0.38376(9)	0.8905(2)	0.0575(4)
O1	0.58533(19)	0.42876(8)	0.79650(19)	0.0512(4)
N1	0.7199(2)	0.42723(10)	0.4851(2)	0.0476(4)
C5	0.7187(2)	0.4920.8(11)	0.5722(2)	0.0401(4)
C9	0.6562(2)	0.49299(11)	0.7387(2)	0.0416(4)
C4	0.7781(2)	0.55924(11)	0.5068(2)	0.0427(4)
C8	0.6526(3)	0.55503(12)	0.8349(3)	0.0470(5)
C10	0.6956(3)	0.37543(12)	0.8607(2)	0.0488(5)
C3	0.8446(3)	0.55635(13)	0.3438(3)	0.0516(5)
C7	0.7104(3)	0.62155(12)	0.7701(3)	0.0516(5)
C2	0.8475(3)	0.49107(15)	0.2589(3)	0.0561(6)
C1	0.7830(3)	0.42807(14)	0.3335(3)	0.0550(5)
C6	0.7708(3)	0.62281(11)	0.6103(3)	0.0494(5)
C11	0.5937(4)	0.30787(13)	0.8914(3)	0.0623(6)
C12	0.6667(5)	0.25193(16)	0.9709(4)	0.0767(8)
H8	0.610966	0.553657	0.946065	0.056
H3	0.886672	0.599643	0.294302	0.062
H7	0.707668	0.665438	0.836977	0.062
H2	0.892851	0.488049	0.149758	0.067
H1	0.78492	0.38307	0.270732	0.066
H11	0.472511	0.306489	0.85117	0.075
H12A	0.7940(50)	0.2630(20)	0.10110(40)	0.093(10)
H12B	0.5920(50)	0.2040(20)	0.10010(50)	0.109(11)

The crystallographic data for the compound was captured on a Bruker D8 Venture diffractometer equipped with Mo-Kα radiation at a wavelength of 0.71073 Å. The collection and processing of data were facilitated by the SMART and SAINT software. Unit cell parameters were derived by integrating reflections from comprehensive frame data. The crystal structures were decoded using ShelXT through Intrinsic Phasing and refined using ShelXL, which utilized Least Squares

minimization within the Olex2 environment. For organic compounds, non-hydrogen atoms underwent anisotropic refinement, while hydrogen atoms were geometrically positioned with fixed isotropic thermal parameters. The molecular structure is shown in the Figure 1. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

3. Conclusion

Quinoline and its derivatives have fluorescence properties because of their conjugated systems. In terms of fluorescence detection, some quinolines can be used for biological imaging, marking biomolecules such as proteins, nucleic acids, etc., to help researchers observe the distribution and dynamic changes of these biomolecules in cells. [5-7] As pharmacophore, the structure of quinoline is a key part of many drugs. Some quinolines have anti-malarial activity and antibacterial activity, which can inhibit the growth and reproduction of malaria or bacteria. [8-10] In order to better study of fluorescence detection for cysteine, we introduced acrylate group as cysteine recognition group on 8-hydroxyquinoline to design the title compound. In this paper, we reported the synthesis and crystal structure of the title compound.

The compound contains one chlorine atom, one nitrogen atom, and an ester group attached to a benzene ring. The bond lengths and bond angles derived from the molecular structure are within expected ranges. The C–Cl bond was determined by the distance of approximately 1.738(2) Å (C6–Cl1), and the C–N bond by the distance of 1.319(3) Å (C1–N1), 1.356(3) Å (C5–N1). The ester group includes a C=O bond with a distance of 1.202(3) Å (C10–O2) and two C–O single bonds with distances of 1.388(2) Å (C9–O1), 1.343(3) Å (C10–O1). In general, all bond lengths and angles fall within the typical values for such functional groups in aromatic systems.

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