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Structure of the mixed crystal (S)-(6-(bromo/chloro)-2-methoxy-2,6-dihydroquinolin-3-yl)(phenyl)methanol, $C_{17}H_{14}Br_{0.5}Cl_{0.5}NO_2$

The crystal structure is shown in the Figure above. Tables 1 and 2 contain details of the measurement method and a list of atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals, reagents and solvents are of analytical grade and are commercially available. Preparation of the title compound: 6-bromo-3-(chloro(phenyl)methyl)-2-methoxyquinoline (5 g, 14.63 mmol) was dissolved

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Abstract

$C_{17}H_{14}Br_{0.5}Cl_{0.5}NO_2$, monoclinic, $P2_1/n$ (no. 14), $a = 11.7324(5)$ Å, $b = 7.4663(3)$ Å, $c = 17.2211(8)$ Å, $\beta = 104.890(1)^\circ$, $V = 1457.87(11)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0334$, $wR_{ref}(F^2) = 0.0693$, $T = 296(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.22 \times 0.20 \times 0.18$ mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.54 mm ⁻¹
Diffractometer, scan mode:	φ and ω
θ_{max} , completeness:	25.4° , >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	9234, 2657, 0.025
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2002
$N(param)_{refined}$:	200
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Olex2 [4]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Br1 ^a	1.41206 (3)	1.04035 (6)	0.18054 (3)	0.07752 (18)
Cl1 ^a	1.41206 (3)	1.04035 (6)	0.18054 (3)	0.07752 (18)
O1	0.84304 (15)	0.4759 (2)	0.01229 (11)	0.0548 (4)
H1	0.843 (3)	0.379 (4)	0.0451 (18)	0.107 (11)*
N2	0.88432 (14)	1.1003 (2)	0.08544 (10)	0.0356 (4)
O2	0.70300 (11)	0.96332 (17)	0.04293 (9)	0.0414 (4)
C1	1.24940 (18)	1.0518 (3)	0.15357 (13)	0.0493 (6)
C2	1.19650 (19)	1.2169 (3)	0.15795 (13)	0.0513 (6)
H2	1.2426	1.3173	0.1761	0.062*
C3	1.07620 (19)	1.2308 (3)	0.13540 (13)	0.0467 (6)
H3	1.0408	1.3414	0.1379	0.056*
C4	1.00544 (17)	1.0799 (2)	0.10843 (11)	0.0340 (4)
C5	1.06005 (17)	0.9130 (3)	0.10586 (12)	0.0354 (5)
C6	1.18443 (18)	0.9014 (3)	0.12836 (13)	0.0461 (5)
H6	1.2216	0.7923	0.1260	0.055*
C7	0.98660 (17)	0.7620 (3)	0.07995 (12)	0.0377 (5)
H7	1.0204	0.6507	0.0763	0.045*
C8	0.86783 (16)	0.7786 (2)	0.06057 (11)	0.0317 (4)
C9	0.82178 (16)	0.9555 (2)	0.06403 (11)	0.0319 (4)
C10	0.64854 (19)	1.1335 (3)	0.04726 (16)	0.0568 (7)
H10A	0.6824	1.1872	0.0987	0.085*
H10B	0.5654	1.1172	0.0404	0.085*
H10C	0.6614	1.2103	0.0056	0.085*
C11	0.78257 (17)	0.6236 (2)	0.03587 (12)	0.0350 (5)
H11	0.7160 (15)	0.651 (2)	-0.0170 (10)	0.025 (5)*
C12	0.72397 (16)	0.5738 (2)	0.10156 (11)	0.0321 (4)
C13	0.60314 (18)	0.5884 (3)	0.08942 (14)	0.0456 (5)
H13	0.5577	0.6358	0.0415	0.055*
C14	0.5494 (2)	0.5329 (3)	0.14807 (17)	0.0605 (7)
H14	0.4681	0.5431	0.1392	0.073*
C15	0.6149 (2)	0.4633 (3)	0.21893 (16)	0.0603 (7)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
H15	0.5782	0.4247	0.2578	0.072*
C16	0.7352 (2)	0.4507 (3)	0.23245 (14)	0.0546 (6)
H16	0.7801	0.4045	0.2808	0.065*
C17	0.78936 (19)	0.5066 (3)	0.17443 (12)	0.0418 (5)
H17	0.8710	0.4990	0.1844	0.050*

^aOccupancy: 0.5.

in acetonitrile (100 mL) at room temperature, followed by addition of H₂O (0.30 g, 16.10 mmol), (2*S*,3*R*)-2-amino-3-hydroxy-*N*-((*S*)-1-(naphthalen-1-yl) ethyl)-*N*-(4-nitrobenzyl)butanamide (0.56 g, 1.463 mmol), potassium carbonate (5.72 g, 43.89 mmol) and tetrabutylammonium iodide (0.51 g, 1.463 mmol) in order. Then the mixture reacted under stirring at 82 °C for 2 h. Subsequently, the reaction mixture was poured into water and extracted with dichloromethane. The organic phase was dehydrated with anhydrous sodium sulfate and concentrated at reduced pressure to afford a deep yellow liquid. The crude mixture was purified by silica gel column chromatography, eluted with petroleum ether and ethyl acetate (8:1) to give a white solid (3.71 g, yield 78.2%). Crystals were grown in *n*-hexane and ethyl acetate (3:1) at room temperature.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

The title compound is an important intermediate for the synthesis of quinoline compounds and can be used in the

fields of medicine and organic synthesis [5, 6]. It can participate in the synthesis of alkaloids like Bedaquiline for the preparation of anti-tuberculosis drugs [7]. The structure of the title compound was elucidated by X-ray diffraction and the derived parameters are as expected.

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