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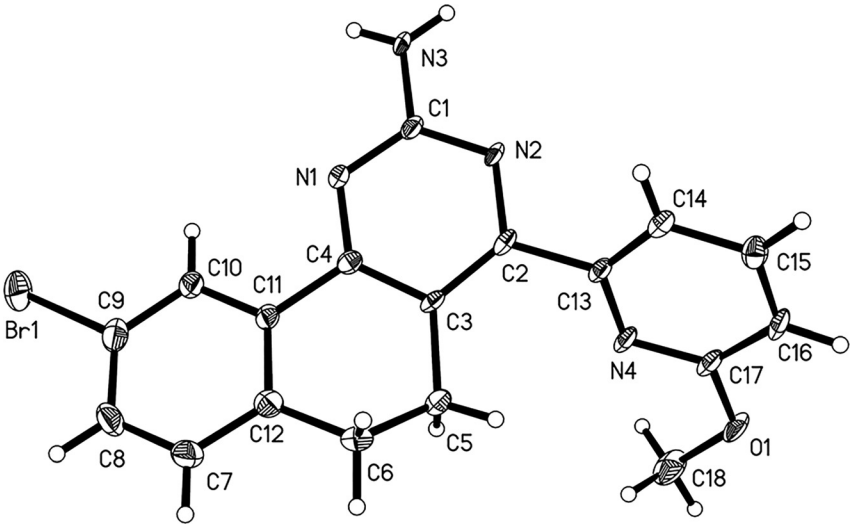
Crystal structure of 9-bromo-4-(6-methoxypyridin-2-yl)-5,6-dihydrobenzo[*h*] quinazolin- 2-amine, $C_{18}H_{15}BrN_4O$



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Abstract

$C_{18}H_{15}BrN_4O$, monoclinic, $P2_1/c$ (no. 14), a = 10.5097(7) Å, b = 6.6990(5) Å, c = 23.5623(15) Å, β = 92.038(6)°, V = 1657.8(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0393, $wR_{ref}(F^2)$ = 0.0863, T = 150.00(10) K.

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The molecular structure is shown in the figure. Displacement ellipsoids are drawn at the 40% probability level. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.16 × 0.15 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.49 mm^{-1}
Diffractometer, scan mode:	SuperNova
θ_{max}, completeness:	25.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7896, 3081, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2406
$N(param)_{refined}$:	220
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3]

Source of material

The intermediate (*E*)-7-bromo-2-((6-methoxypyridin-2-yl) methylene)-3,4-dihydronaphthalen-1(*H*)-one (BDHN) was prepared according to the literature method [4]. BDHN (1.00 g, 2.9 mmol), guanidine hydrochloride (1.39 g, 14.50 mmol), potassium hydroxide (0.81 g, 14.50 mmol), and potassium carbonate (0.80 g, 5.80 mmol) were dissolved in a mixed solution of anhydrous ethanol (15 mL) and 1,2-dichloroethane (15 mL). The mixture was heated to reflux for about 1.5 h. The process of the reaction was monitored by thin-layer chromatography (TLC). When the reaction was completed, the mixture was cooled to room temperature. After filtration, the filtrate was removed by

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.33315 (3)	0.83230 (5)	0.25784 (2)	0.04477 (14)
C1	1.0454 (2)	0.2605 (4)	0.44531 (10)	0.0183 (6)
C2	0.8366 (2)	0.3564 (3)	0.44517 (10)	0.0169 (6)
C3	0.8597 (2)	0.5074 (4)	0.40596 (10)	0.0167 (6)
C4	0.9861 (2)	0.5274 (3)	0.39116 (10)	0.0174 (6)
C5	0.7615 (3)	0.6493 (3)	0.38055 (11)	0.0215 (6)
H5A	0.688012	0.653329	0.404280	0.026*
H5B	0.733657	0.602393	0.343191	0.026*
C6	0.8179 (3)	0.8588 (4)	0.37575 (12)	0.0256 (6)
H6A	0.757810	0.943720	0.354915	0.031*
H6B	0.831027	0.914592	0.413498	0.031*
C7	0.9816 (3)	1.0156 (4)	0.31287 (12)	0.0298 (7)
H7	0.928966	1.126253	0.307889	0.036*
C8	1.0970 (3)	1.0121 (4)	0.28694 (12)	0.0329 (7)
H8	1.121989	1.118502	0.264583	0.039*
C9	1.1749 (3)	0.8473 (4)	0.29477 (11)	0.0283 (7)
C10	1.1400 (3)	0.6885 (4)	0.32797 (10)	0.0228 (6)
H10	1.193906	0.579433	0.333027	0.027*
C11	1.0239 (2)	0.6933 (4)	0.35366 (10)	0.0185 (6)
C12	0.9416 (3)	0.8579 (4)	0.34633 (11)	0.0227 (6)
C13	0.7096 (2)	0.3163 (3)	0.46814 (11)	0.0172 (6)
C14	0.6942 (3)	0.2976 (4)	0.52584 (11)	0.0210 (6)
H14	0.763109	0.313489	0.551330	0.025*
C15	0.5740 (3)	0.2548 (4)	0.54503 (11)	0.0232 (6)
H15	0.561393	0.240731	0.583695	0.028*
C16	0.4739 (3)	0.2333 (4)	0.50670 (11)	0.0222 (6)
H16	0.392304	0.204662	0.518408	0.027*
C17	0.4993 (2)	0.2562 (4)	0.44955 (11)	0.0220 (6)
C18	0.4196 (3)	0.2705 (5)	0.35378 (12)	0.0399 (8)
H18A	0.448715	0.405307	0.349457	0.060*
H18B	0.341712	0.252032	0.331866	0.060*
H18C	0.482925	0.179878	0.340707	0.060*
N1	1.07908 (19)	0.4041 (3)	0.40919 (8)	0.0185 (5)
N2	0.92941 (19)	0.2351 (3)	0.46611 (9)	0.0177 (5)
N3	1.1375 (2)	0.1310 (3)	0.46273 (10)	0.0259 (6)
H3A	1.205834	0.138369	0.446948	0.039 (10)*
H3B	1.118257	0.025852	0.481458	0.018 (7)*
N4	0.6112 (2)	0.2987 (3)	0.42948 (9)	0.0196 (5)
O1	0.39776 (17)	0.2323 (3)	0.41240 (8)	0.0280 (5)

rotary evaporator and the residue was purified on a silica-gel column with petroleum ether/ethyl acetate (8:1 v/v). Suitable single crystals of the title compound were collected by slow evaporation of a methanol solution under the ambient condition.

Experimental details

The H atoms were placed in idealized positions and treated as riding on their parent atoms, with *d*(C—H) = 0.96 Å(methyl), U_{iso}(H) = 1.5U_{eq}(C), and *d*(C—H) = 0.97 Å(methylene), U_{iso}(H) = 1.2U_{eq}(C), and *d*(C—H) = 0.93 Å(aromatic), U_{iso}(H) = 1.2U_{eq}(C). H atoms at the N atom were located in difference maps and treated as riding with free U_{iso}.

Comment

Curcumin is one kind of *α*,*β*-unsaturated ketone compounds. It possesses suitable anti-inflammatory and antioxidant effects in humans [5, 6]. However, its poor solubility and instability hinder its application in medicine. In order to solve the disadvantages of curcumin derivatives, it is very important that their structures will be modified to improve their stability and solubility. Base on this point, 9-bromo-4-(6-methoxypyridin-2-yl)-5,6-dihydrobenzo[*h*]quinazolin- 2-amine was obtained by Claisen–Schmidt condensation and Michael addition reaction.

The single-crystal structure analysis shows that the title compound crystallizes in the monoclinic space group $P2_1/c$ with one molecule in the asymmetric unit (*cf*. the figure). All bond lengths and bond angles are within the normal range and agree with the previously reported values [7, 8]. Because the C5 and C6 atoms are sp^3 hybridized, pyrimidine and neighbouring benzene rings are not coplanar with a dihedral angle of 17.49(12)° between their planes. As shown in the figure, the pyridine ring has a significant rotation with respect to the pyrimidine ring. The dihedral angle between pyridine and pyrimidine ring is 50.83(8)°.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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