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The crystal structure of 4-(3-chloro-4-fluorophenyl amino)-7-methoxyquinazolin-6-ol, $C_{15}H_{11}ClFN_3O_2$

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Abstract

$C_{15}H_{11}ClFN_3O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 12.9430(8)$ Å, $b = 9.3196(5)$ Å, $c = 12.5959(8)$ Å, $\beta = 115.861(8)^\circ$, $V = 1367.21(16)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0512$, $wR_{ref}(F^2) = 0.1359$, $T = 296(2)$ K.

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

Source of material

The title compound is commercially available. The crystal suitable for X-ray diffraction was obtained by slow evaporation from DMF:MeOH (1:2) at room temperature.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.23 \times 0.20 \times 0.18$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.30 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
$\theta_{\max}$, completeness:	25.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5456, 2497, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1838
$N(\text{param})_{\text{refined}}$:	201
Programs:	Olex2 [1], SHELX [2, 3], CrysAlis ^{PRO} [4]

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [2, 3]. The hydrogen atoms were placed at calculated positions with isotropic displacement parameters. The U_{iso} values were fixed at 1.2 times of the parent atoms for all C(H) groups and all N(H) groups, and 1.5 times for all C(H,H,H) groups, all O(H) groups.

Comment

The title compound has been paid much attention because it is an analog of anti-cancer pharmaceutical Gefitinib. The crystal structures of some other derivatives of the title compound have been reported [5, 6].

There is one molecule in the asymmetric unit of the title structure (cf. left part of the figure).

The angle between the 6-membered aromatic plane and 10-membered aromatic heterocyclic plane is $47.38(12)^\circ$. This

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cl1	−0.14134(8)	0.71513(12)	0.27001(8)	0.0732(3)
F1	−0.10006(19)	0.8728(2)	0.4855(2)	0.0848(7)
O1	0.53829(16)	0.0694(2)	0.72281(15)	0.0408(5)
H1	0.496095	0.075128	0.755842	0.061*
O2	0.65394(15)	0.0681(2)	0.59872(15)	0.0393(5)
N1	0.24197(18)	0.4831(2)	0.54733(18)	0.0363(5)
H1A	0.266363	0.434263	0.611614	0.044*
N2	0.25849(18)	0.5312(2)	0.37509(18)	0.0341(5)
N3	0.40419(18)	0.4236(2)	0.33419(17)	0.0326(5)
C1	0.4136(2)	0.2574(3)	0.6049(2)	0.0320(6)
H1B	0.374911	0.260870	0.652099	0.038*
C2	0.5028(2)	0.1633(3)	0.6317(2)	0.0300(6)
C3	0.5634(2)	0.1599(3)	0.5606(2)	0.0297(6)
C4	0.5297(2)	0.2450(3)	0.4629(2)	0.0317(6)
H4	0.568296	0.240384	0.415601	0.038*
C5	0.4365(2)	0.3396(3)	0.4339(2)	0.0278(6)
C6	0.3798(2)	0.3492(3)	0.5061(2)	0.0276(6)
C7	0.2917(2)	0.4551(3)	0.4738(2)	0.0296(6)
C8	0.3174(2)	0.5091(3)	0.3114(2)	0.0355(6)
H8	0.292991	0.561553	0.241828	0.043*
C9	0.7270(2)	0.0731(3)	0.5407(3)	0.0473(8)
H9A	0.684979	0.042643	0.460252	0.071*
H9B	0.791479	0.010546	0.580295	0.071*
H9C	0.753846	0.169515	0.542337	0.071*
C10	0.1544(2)	0.5843(3)	0.5284(2)	0.0319(6)
C11	0.0613(2)	0.5999(3)	0.4194(2)	0.0358(6)
H11	0.056040	0.544725	0.355754	0.043*
C12	−0.0235(2)	0.6971(3)	0.4056(3)	0.0438(7)
C13	−0.0164(3)	0.7770(3)	0.5003(3)	0.0510(8)
C14	0.0740(3)	0.7611(3)	0.6092(3)	0.0545(8)
H14	0.077737	0.815198	0.672837	0.065*
C15	0.1599(2)	0.6636(3)	0.6236(2)	0.0434(7)
H15	0.221345	0.651379	0.697383	0.052*

angle is similar to the structure of the related derivative (CCDC 873811) [5], but differs from the derivative (CCDC 259805) [6], in which the two rings are almost coplanar.

In the crystal, intermolecular O1–H1···N3' [*i*' = *x*, 1/2−*y*, 1/2 + *z*] hydrogen bonds link the molecules into a chain along the *c* axis. Intermolecular C15–H15···O2'' [*i*'' = 1−*x*, 1/2 + *y*, 3/2−*z*] non-classical hydrogen bonds link the molecules into a chain along the *b* axis. These two hydrogen bonding interactions construct a network parallel to the *bc* plane (*cf.* right part of the figure). Intramolecular hydrogen bonds C–H···N also exist in the crystal.

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