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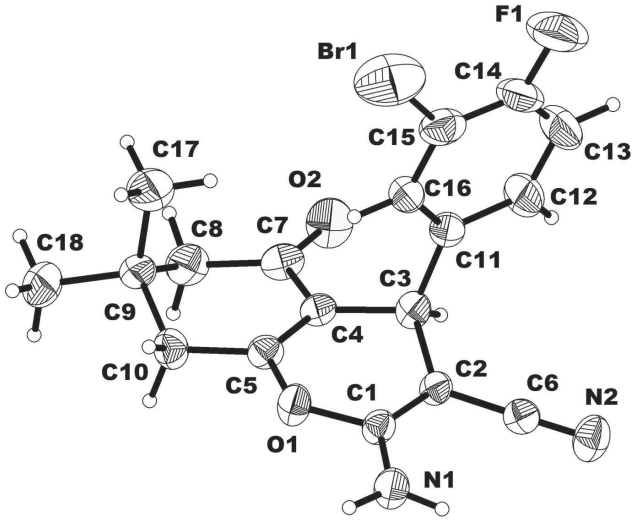
Jing Song*

Crystal structure of 2-amino-4-(3-bromo-4-fluoro-phenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile, $C_{18}H_{16}BrFN_2O_2$



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Abstract

$C_{18}H_{16}BrFN_2O_2$, monoclinic, $C2/c$ (no. 15), $a = 23.5259(15)$ Å, $b = 9.3637(6)$ Å, $c = 15.7104(13)$ Å, $\beta = 93.786(6)°$, $V = 3453.3(4)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0486$, $wR_{ref}(F^2) = 0.1366$, $T = 296$ K.

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The crystal structure is shown in the figure. Table 1 contains details on crystal structure and measurement conditions. Table 2 lists the atoms with their coordinates and displacement parameters.

Source of materials

The title compound was synthesized according to a reported procedure [3]. A mixture of 3,5-cyclohexanedione (10 mmol), 3-bromo-4-fluoro-benzaldehyde (10 mmol), mal-

***Corresponding author: Jing Song,** School of Chemical Engineering, Xi'an University, Xi'an, Shaanxi, China, e-mail: song_jing666@163.com

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.18 × 0.16 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	24.0 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII, φ and ω
$2\theta_{max}$, completeness:	46.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6434, 3029, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1903
$N(param)_{refined}$:	218
Programs:	Bruker [1], SHELX [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	0.21175(2)	0.70247(7)	0.11977(4)	0.1004(4)
F1	0.25649(13)	0.5221(3)	0.26595(19)	0.1064(12)
C1	0.43634(14)	0.8184(3)	−0.0118(2)	0.0333(8)
C2	0.45189(14)	0.7788(3)	0.0695(2)	0.0313(8)
C3	0.43144(14)	0.8577(3)	0.1455(2)	0.0347(8)
H3A	0.4630	0.8620	0.1893	0.042*
C4	0.41708(14)	1.0075(3)	0.1181(2)	0.0324(8)
C5	0.40462(15)	1.0416(3)	0.0368(2)	0.0345(8)
C6	0.48302(15)	0.6516(4)	0.0837(2)	0.0338(8)
C7	0.41650(15)	1.1199(4)	0.1823(2)	0.0403(9)
C8	0.40311(19)	1.2688(4)	0.1522(3)	0.0515(11)
H8A	0.4382	1.3154	0.1387	0.062*
H8B	0.3874	1.3219	0.1982	0.062*
C9	0.36140(16)	1.2748(3)	0.0745(2)	0.0413(9)
C10	0.38451(17)	1.1823(3)	0.0047(2)	0.0429(9)
H10A	0.3548	1.1682	−0.0404	0.051*
H10B	0.4158	1.2318	−0.0197	0.051*
C11	0.38248(16)	0.7782(4)	0.1826(2)	0.0375(9)
C12	0.3930(2)	0.6884(4)	0.2512(2)	0.0592(12)
H12A	0.4293	0.6850	0.2783	0.071*
C13	0.3505(3)	0.6032(5)	0.2805(3)	0.0744(15)
H13A	0.3579	0.5424	0.3266	0.089*
C14	0.2981(2)	0.6100(4)	0.2409(3)	0.0655(14)
C15	0.28532(18)	0.6995(4)	0.1726(3)	0.0547(11)
C16	0.32817(16)	0.7836(3)	0.1439(2)	0.0411(9)
H16A	0.3204	0.8446	0.0980	0.049*
O1	0.40883(11)	0.9452(2)	−0.02864(14)	0.0418(6)
O2	0.42833(12)	1.0922(3)	0.25764(17)	0.0592(8)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.44415(14)	0.7502(3)	−0.08436(18)	0.0469(8)
H1A	0.4613	0.6690	−0.0834	0.056*
H1B	0.4321	0.7873	−0.1323	0.056*
N2	0.50901(14)	0.5502(3)	0.0977(2)	0.0509(9)
C17	0.30378(18)	1.2199(5)	0.0988(3)	0.0694(13)
H17A	0.3080	1.1248	0.1211	0.104*
H17B	0.2776	1.2190	0.0492	0.104*
H17C	0.2894	1.2812	0.1413	0.104*
C18	0.3547(2)	1.4273(4)	0.0407(3)	0.0632(13)
H18A	0.3394	1.4867	0.0835	0.095*
H18B	0.3292	1.4277	−0.0096	0.095*
H18C	0.3911	1.4635	0.0271	0.095*

ononitrile (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) were refluxed in ethanol (100 mL) for 2–3 h and then cooled to room temperature. After filtering the precipitate, it was washed with ice-cooled water and ethanol sequentially and then dried under a vacuum.

Experimental details

Hydrogen atoms bonded to C and N were positioned geometrically and refined using a riding model, with C···H = 0.97 Å and N···H = 0.86 Å. U_{iso}(H) was set to 1.2 times U_{eq}(C) and U_{eq}(N).

Comment

The importance of the pyranyl moiety has been well proven as illustrated by a large number of patents for its use as chemotherapeutic agent [4, 5]. A number of biological activities have been associated with it, such as antihypertensive, antiasthemic, analgesic, cardiovascular, platelate aggregation inhibitor and calcium antagonist [6]. With this in mind, some new pyran derivatives have been synthesized in search of better therapeutic agents, by a convenient single pot method [7].

In the crystal structure of the title compound, the pyran ring is folded slightly (dihedral angle C4—C3—C2—C1 = 25°). The plane of the 3-bromo-4-fluoro-phenyl moiety is almost perpendicular to it. The adjacent dimethyl-cyclo-hexen-1-onyl ring adopts a "chair" conformation with an dihedral angle C4—C7—C8—C9 of 33°. Bond lengths and angles are almost in the expected ranges.

References

1. Bruker AXS Inc., APEX2, SAINT, SHELXTL, Madison, Wisconsin, USA, (2001).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Gordon, R. J.; Lowy, F. D.: Pathogenesis of methicillin-resistant Staphylococcus aureus infection. Clin. Infect. Dis. **46** (2008) S350–S359.
4. Khan, A. T.; Lal, M.; Ali, S.; Khan, M. M.: One-pot three-component reaction for the synthesis of pyran annulated heterocyclic compounds using DMAP as a catalyst. Tetrahedron Lett. **52** (2011) 5327–5332.
5. Eshghi, H.; Zohuri, G. H.; Sandaroos, R.; Damavandi, S.: Synthesis of novel benzo chromene compounds catalyzed by ionic liquid. Heterocycl. Commun. **18** (2012) 67–70.
6. Bonsignore, L.; Loy, G.; Secci, D.; Calignano, A.: Synthesis and pharmacological activity of 2-oxo-(2*H*) 1-benzopyran-3-carboxamide derivatives. Eur. J. Med. Chem. **28** (1993) 517–520.
7. Gacche, R. N.; Jadhav, S. G.: Antioxidant activities and cytotoxicity of selected coumarin derivatives: preliminary results of a structure-activity relationship study using computational tools. J. Exp. Clin. Med. **4** (2012) 165–169.