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Crystal structure of ethyl 2-amino-4-(4-bromothiophen-2-yl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carboxylate, $C_{16}H_{16}BrNO_4S$

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Abstract

$C_{16}H_{16}BrNO_4S$, monoclinic, $P2_1/c$ (no. 14), $a = 9.649(4)$ Å, $b = 15.108(4)$ Å, $c = 14.994(5)$ Å, $\beta = 128.65(2)^\circ$, $V = 1707.0(11)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0444$, $wR_{ref}(F^2) = 0.0999$, $T = 293(2)$ 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.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.35 \times 0.28 \times 0.21$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.5 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
θ_{max} , completeness:	25° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8410, 2998, 0.079
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1664
$N(\text{param})_{\text{refined}}$:	209
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.08082(6)	0.29052(3)	0.18484(4)	0.0757(3)
S1	0.65234(15)	0.34792(8)	0.41637(8)	0.0592(4)
O1	0.6373(4)	0.49053(17)	0.2006(2)	0.0586(8)
O2	0.9127(4)	0.5413(2)	0.3384(2)	0.0766(10)
O3	1.0846(3)	0.27806(17)	0.43244(19)	0.0541(7)
O4	0.5269(4)	0.16242(17)	0.1219(2)	0.0593(8)
N1	1.1659(4)	0.4189(2)	0.4691(3)	0.0619(10)
H1A	1.151133	0.475222	0.460052	0.074*
H1B	1.269916	0.397393	0.520269	0.074*
C7	0.7941(5)	0.2252(2)	0.2797(3)	0.0390(9)
C11	0.7731(6)	0.0632(3)	0.2269(4)	0.0868(16)
H11A	0.689380	0.017044	0.208911	0.104*
H11B	0.800525	0.056580	0.175019	0.104*
C10	0.9363(7)	0.0493(3)	0.3436(4)	0.110(2)
H10A	0.997051	−0.002091	0.344055	0.132*
H10B	0.904352	0.035852	0.391973	0.132*
C14	0.4068(5)	0.3013(2)	0.2115(3)	0.0431(10)
H14	0.344292	0.284541	0.135641	0.052*
C13	0.5831(5)	0.3193(2)	0.2824(3)	0.0388(9)
C15	0.3280(5)	0.3104(2)	0.2647(4)	0.0491(10)
C3	0.8105(6)	0.4785(3)	0.2908(3)	0.0532(11)
C4	0.8539(5)	0.3859(2)	0.3187(3)	0.0418(9)
C9	1.0619(6)	0.1250(3)	0.3944(4)	0.0679(13)
H9A	1.143037	0.118878	0.476818	0.081*
H9B	1.131150	0.124144	0.367695	0.081*
C12	0.6844(6)	0.1512(3)	0.2044(3)	0.0484(10)
C1	0.3943(8)	0.5819(3)	0.0618(4)	0.119(2)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H1C	0.352445	0.641870	0.041497	0.179*
H1D	0.394287	0.556150	0.003276	0.179*
H1E	0.317662	0.548377	0.069366	0.179*
C8	0.7109(5)	0.3164(2)	0.2557(3)	0.0392(9)
H8	0.642214	0.327886	0.173776	0.047*
C6	0.9672(5)	0.2108(3)	0.3634(3)	0.0448(10)
C5	1.0274(5)	0.3647(3)	0.4036(3)	0.0445(10)
C16	0.4423(6)	0.3346(3)	0.3748(4)	0.0612(12)
H16	0.412053	0.343016	0.422194	0.073*
C2	0.5736(6)	0.5811(3)	0.1696(4)	0.0834(15)
H2A	0.651272	0.615492	0.162590	0.100*
H2B	0.574452	0.607699	0.228848	0.100*

Source of material

The title compound was synthesized according to a reported procedure. A mixture of 3,5-cyclohexanedione (10 mmol), 4-bromothiophene-2-carbaldehyde (10 mmol), ethyl cyanoacetate (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C–H = 0.93/0.96/0.97/0.98 Å and N–H = 0.86 Å with *U*_{iso}(H) = 1.2 times *U*_{eq}(C) and 1.2 times *U*_{eq}(N).

Discussion

It is known that many pyran derivatives have biological activity with their antibacterial, antidepressive, anticancer and many others [4–6]. There is one molecule in the asymmetric unit of the title crystal structure.

In the crystal structure of the title compound (*cf.* the figure), the angle between the mean planes of six-membered ring containing one oxygen atom [C(4), C(5), O(3), C(6), C(7) and C(8)] and the adjacent five-membered ring containing one S atom [C(13)–C(16) and S(1)] is 77°. The bond lengths of C–Br and C–N are in the range of 1.909(4) Å and 1.333(4) Å. The C–S, C–O and C–C bond lengths fall in the range of 1.721(4)–1.728(4) Å, 1.229(4) Å–1.386(4) Å and 1.3336(5) Å–1.519(4) Å, respectively. These bond distances are all in their normal scopes and they are similar with those previously reported compounds [4, 7]. It is noting that one H atom presented in the compound ethyl 2-amino-5-oxo-4-(thiophen-3-yl)-5,6,7,8-tetrahydro-4*H*-chromene-3-carboxylate was replaced by one Br atom in the title compound.

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