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Crystal structure of 2-amino-4-(3,5-dibromo-4-hydroxyphenyl)-7-methyl-5-oxo-2*H*,5*H*-pyrano [4,3-*b*]pyran-3-carbonitrile, C₁₆H₁₀Br₂N₂O₄

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.96 mm ⁻¹
Diffractometer, scan mode:	APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	24.9°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8012, 2889, 0.069
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1832
$N(\text{param})_{\text{refined}}$:	217
Programs:	Olex2 [4], SHELX [5], Bruker [6]

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Abstract

C₁₆H₁₀Br₂N₂O₄, monoclinic, $P2_1/n$ (no. 14), $a = 12.885(9)$ Å, $b = 8.781(6)$ Å, $c = 15.692(11)$ Å, $\beta = 112.258(13)^\circ$, $V = 1643.2(19)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0526$, $wR_{\text{ref}}(F^2) = 0.1284$, $T = 296(2)$ 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 materials

H atoms bonded to C were positioned geometrically and refined using a riding model, with C–H = 0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.25726(5)	0.56893(8)	0.23751(5)	0.0502(3)
Br2	0.62738(6)	0.88761(9)	0.48930(5)	0.0524(3)
O1	0.4205(3)	0.7010(5)	0.4265(3)	0.0521(12)
H1	0.363550	0.648687	0.409863	0.078*
O2	0.6831(3)	1.1530(4)	0.1428(3)	0.0383(11)
O3	0.8021(3)	0.7246(4)	0.1485(3)	0.0391(10)
O4	0.6401(4)	0.6212(5)	0.1299(4)	0.0540(13)
C1	0.3807(4)	0.7020(6)	0.2620(4)	0.0326(14)
C2	0.4419(5)	0.7442(7)	0.3521(4)	0.0328(14)
C3	0.5364(5)	0.8353(6)	0.3673(4)	0.0320(14)
C4	0.5644(5)	0.8837(6)	0.2953(4)	0.0312(14)
H4	0.627028	0.945179	0.307032	0.037*
C5	0.5012(5)	0.8425(6)	0.2069(4)	0.0272(13)
C6	0.4093(4)	0.7491(6)	0.1908(4)	0.0309(13)
H6	0.366612	0.718097	0.131104	0.037*
C7	0.5290(5)	0.9030(6)	0.1262(4)	0.0283(13)
H7	0.483606	0.846058	0.070898	0.034*
C8	0.4966(5)	1.0690(6)	0.1097(4)	0.0332(14)
C9	0.3824(6)	1.1106(7)	0.0858(4)	0.0367(15)
C10	0.5702(5)	1.1799(7)	0.1194(4)	0.0337(15)
C11	0.7172(5)	1.0051(6)	0.1460(4)	0.0292(13)
C12	0.6474(5)	0.8865(6)	0.1384(4)	0.0296(13)
C13	0.6918(5)	0.7390(7)	0.1384(4)	0.0369(15)
C14	0.8679(5)	0.8487(7)	0.1549(4)	0.0373(15)
C15	0.9812(5)	0.8043(8)	0.1602(5)	0.0526(19)
H15A	0.985945	0.695292	0.158190	0.079*
H15B	0.994634	0.847505	0.109105	0.079*
H15C	1.036536	0.841206	0.216766	0.079*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C16	0.8294(5)	0.9864(7)	0.1556(4)	0.0365(15)
H16	0.875554	1.070665	0.162356	0.044*
N1	0.2918(5)	1.1447(7)	0.0677(4)	0.0608(18)
N2	0.5499(5)	1.3300(6)	0.1080(4)	0.0532(16)
H2A	0.482635	1.363701	0.092646	0.064*
H2B	0.604269	1.392442	0.116080	0.064*

The title compound was synthesized according to a reported procedure. A mixture of 4-hydroxy-6-methylpyran-2-one (10 mmol), 3,5-dibromobenzaldehyde (10 mmol), malonitrile (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.

Comment

Pyran derivatives may possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity [1]. A large number of structurally novel pyran derivatives have been reported to show substantial cytotoxic activity in vitro and in vivo. Considering their importance, research focused on the synthesis of the dihydropyran derivatives [2]. Herein, we reported the structure of such a pyran derivative.

In the crystal structure of the title compound (Figure), a methyl group, a cyano group and one amino group are linked to the 4*H*,5*H*-pyrano[4,3-*b*]pyran-5-one moiety. One hydroxy group and two Br substituents are connected to C14, C13 and C15 atoms respectively. All bond lengths fall in their normal ranges compared with those in previously studies [3].

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