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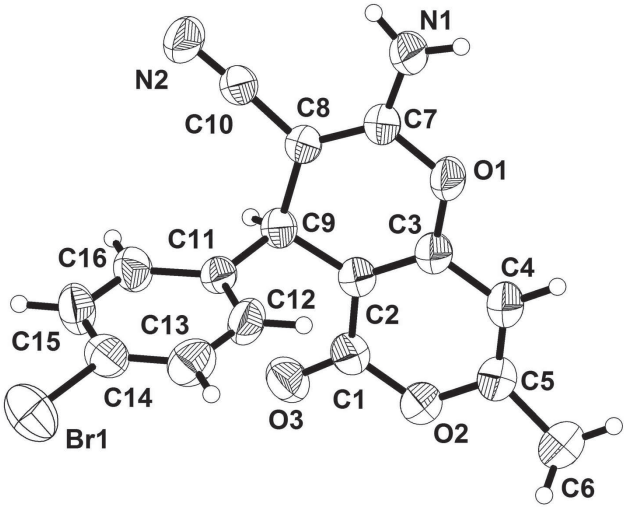
Crystal structure of 2-amino-4-(4-bromo-phenyl)-7-methyl-5-oxo-4*H*,5*H*-pyrano[4,3-*b*]pyran-3-carbonitrile, $C_{16}H_{11}BrN_2O_3$



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Abstract

$C_{16}H_{11}BrN_2O_3$, monoclinic, $C2/c$, $a = 21.504(3)$ Å, $b = 8.2788(10)$ Å, $c = 18.170(3)$ Å, $\beta = 110.511(5)°$, $V = 3029.8(7)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0429$, $wR_{ref}(F^2) = 0.1251$, $T = 296$ K.

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

Source of material

The title compound was synthesized according to a reported procedure [6]. A mixture of 4-hydroxy-6-methylpyran-2-one (10 mmol), 4-bromobenzaldehyde (10 mmol), malononitrile (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 vacuum.

Table 1: Data collection and handling.

Crystal:	Colourless blocks
Size:	0.18 × 0.16 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	27.3 cm^{-1}
Diffractometer, scan mode:	Bruker XSCANS, *ω-scans*
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9161, 2663, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2213
$N(param)_{refined}$:	199
Programs:	SHELX [7], Bruker programs [8]

Experimental details

The H atoms bonded to C and N were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and 0.86 Å and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Discussion

The synthesis of pyrans and their derivatives has attracted considerable attention from organic and medicinal chemists for many years as a large number of natural and synthetic products contain this heterocyclic nucleus [1, 2]. Pyrans possess diverse pharmacological and biological activities such as antitumor, analgesic and ulcerogenic, anti-inflammatory, anticoagulant, phototriggering, and fungicidal properties, and can act as anticoagulants in the production of pesticides [3, 4]. In particular, the anticancer activity of pyran compounds has received considerable attention because of their cytotoxic activity against numerous types of cancers, including malignant melanoma, leukemia, renal cell carcinoma, prostate and breast cancer cell progression [5].

In the crystal structure of the title compound, the 4H-pyran ring is nearly planar and the adjacent pyran-2-on-3,4-diyl ring also adopts an almost planar conformation. The

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.15561(14)	0.7447(4)	−0.23949(17)	0.0474(7)
C2	−0.16301(14)	0.8920(3)	−0.20193(17)	0.0432(6)
C3	−0.15003(15)	1.0332(3)	−0.23085(18)	0.0472(7)
C4	−0.12421(19)	1.0379(4)	−0.2927(2)	0.0598(9)
H4A	−0.1156	1.1363	−0.3119	0.072*
C5	−0.11255(18)	0.9000(4)	−0.3229(2)	0.0593(8)
C6	−0.0816(2)	0.8764(6)	−0.3836(3)	0.0862(13)
H6A	−0.0710	0.9796	−0.4002	0.129*
H6B	−0.1121	0.8204	−0.4278	0.129*
H6C	−0.0417	0.8139	−0.3619	0.129*
C7	−0.18528(14)	1.1865(3)	−0.14360(17)	0.0462(7)
C8	−0.19815(14)	1.0511(3)	−0.10985(17)	0.0424(6)
C9	−0.18439(13)	0.8813(3)	−0.13198(17)	0.0419(6)
H9A	−0.2256	0.8188	−0.1466	0.050*
C10	−0.22607(16)	1.0648(3)	−0.0507(2)	0.0507(7)
C11	−0.13245(13)	0.7989(3)	−0.06265(16)	0.0406(6)
C12	−0.06519(14)	0.8254(4)	−0.04449(18)	0.0518(7)
H12A	−0.0512	0.8939	−0.0761	0.062*
C13	−0.01846(15)	0.7528(4)	0.01928(19)	0.0589(8)
H13A	0.0266	0.7709	0.0301	0.071*
C14	−0.03920(15)	0.6534(4)	0.06666(18)	0.0534(7)
C15	−0.10578(17)	0.6263(4)	0.0509(2)	0.0589(8)
H15A	−0.1196	0.5600	0.0836	0.071*
C16	−0.15181(15)	0.6982(4)	−0.01385(19)	0.0518(7)
H16A	−0.1968	0.6786	−0.0249	0.062*
O1	−0.15946(12)	1.1804(2)	−0.20255(13)	0.0556(5)
O2	−0.12832(12)	0.7553(3)	−0.29775(13)	0.0585(6)
O3	−0.17084(11)	0.6110(3)	−0.22451(13)	0.0578(6)
N1	−0.19546(15)	1.3389(3)	−0.12754(18)	0.0633(8)
H1A	−0.2121	1.3599	−0.0919	0.076*
H1B	−0.1854	1.4165	−0.1528	0.076*
N2	−0.24789(19)	1.0700(3)	−0.0018(2)	0.0748(10)
Br1	0.02508(2)	0.55281(5)	0.15404(2)	0.0848(2)

plane of the heterocyclic moiety is almost perpendicular to the mean plane of the bromophenyl substituent (*cf*. the figure).

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