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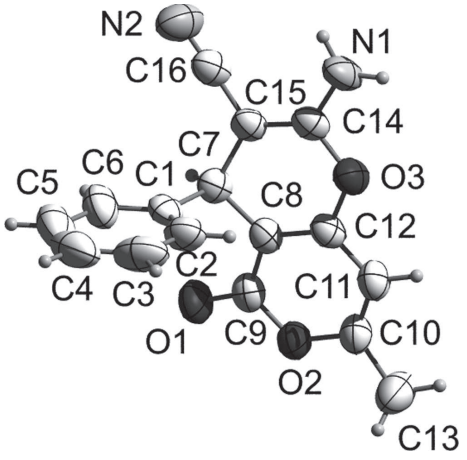
Crystal structure of 2-amino-7-methyl-5-oxo-4-phenyl-4*H*,5*H*-pyrano[4,3-*b*]pyran-3-carbonitrile, $C_{16}H_{12}N_2O_3$



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Abstract

$C_{16}H_{12}N_2O_3$, monoclinic, $P2_1/n$ (no. 14), $a = 10.707(5)$ Å, $b = 8.749(5)$ Å, $c = 15.002(6)$ Å, $\beta = 103.65(2)°$, $V = 1365.7(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0557$, $wR_{ref}(F^2) = 0.1309$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow, block, size 0.21×0.25×0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.96 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6301, 2365
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1148
$N(param)_{refined}$:	192
Programs:	Bruker programs [11], SHELX [12]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*e*	0.4194	0.4446	0.3570	0.086
H(1B)	4*e*	0.3817	0.4100	0.2570	0.086
H(2)	4*e*	0.6639	0.7978	0.2484	0.082
H(3)	4*e*	0.8794	0.8504	0.2870	0.113
H(4)	4*e*	0.9612	1.0188	0.4050	0.129
H(5)	4*e*	0.8231	1.1424	0.4752	0.125
H(6)	4*e*	0.6050	1.0920	0.4349	0.093
H(7)	4*e*	0.4250	0.9704	0.3463	0.063
H(11)	4*e*	0.3095	0.7249	0.0248	0.072
H(13A)	4*e*	0.2555	0.9007	−0.1080	0.133
H(13B)	4*e*	0.3519	1.0384	−0.0916	0.133
H(13C)	4*e*	0.2112	1.0608	−0.0801	0.133

Source of material

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

Experimental details

The hydrogen atoms were set on calculated positions with the help of the SHELX program (AFIX 13, 43, 93 or 137 option) [12].

Discussion

Pyran derivatives form an important class of compounds. These compounds possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant,

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Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.4040(3)	0.4749(3)	0.3009(2)	0.103(3)	0.041(2)	0.080(2)	−0.009(2)	0.041(2)	−0.002(2)
N(2)	4*e*	0.5035(4)	0.6770(4)	0.5121(2)	0.137(4)	0.072(2)	0.075(2)	0.006(2)	0.048(2)	0.004(2)
C(1)	4*e*	0.6119(4)	0.9392(4)	0.3376(2)	0.076(3)	0.036(2)	0.059(2)	−0.007(2)	0.021(2)	0.003(2)
C(2)	4*e*	0.6956(4)	0.8683(4)	0.2946(2)	0.062(3)	0.080(3)	0.067(3)	−0.008(2)	0.025(2)	0.009(2)
C(3)	4*e*	0.8248(5)	0.8985(6)	0.3180(3)	0.068(3)	0.127(4)	0.095(4)	−0.001(3)	0.033(3)	0.040(3)
C(4)	4*e*	0.8733(6)	0.9998(7)	0.3872(4)	0.090(4)	0.112(5)	0.109(4)	−0.034(4)	−0.001(4)	0.048(4)
C(5)	4*e*	0.7912(6)	1.0716(5)	0.4292(4)	0.131(5)	0.060(3)	0.099(4)	−0.028(3)	−0.018(4)	0.014(3)
C(6)	4*e*	0.6599(5)	1.0418(4)	0.4050(3)	0.086(4)	0.054(2)	0.086(3)	−0.008(2)	0.005(3)	−0.008(2)
C(7)	4*e*	0.4699(3)	0.9003(3)	0.3135(2)	0.061(2)	0.045(2)	0.058(2)	0.003(2)	0.027(2)	−0.006(2)
C(8)	4*e*	0.4129(3)	0.9147(4)	0.2131(2)	0.049(2)	0.044(2)	0.058(2)	0.002(2)	0.020(2)	−0.006(2)
C(9)	4*e*	0.3944(3)	1.0644(4)	0.1730(2)	0.055(2)	0.054(2)	0.060(2)	−0.001(2)	0.011(2)	−0.007(2)
C(10)	4*e*	0.3246(4)	0.9484(4)	0.0262(2)	0.063(3)	0.055(2)	0.059(2)	−0.003(2)	0.006(2)	−0.010(2)
C(11)	4*e*	0.3327(3)	0.8103(4)	0.0620(2)	0.067(3)	0.049(2)	0.063(2)	−0.002(2)	0.012(2)	−0.012(2)
C(12)	4*e*	0.3769(3)	0.7947(4)	0.1568(2)	0.059(2)	0.041(2)	0.063(2)	0.002(2)	0.024(2)	−0.003(2)
C(13)	4*e*	0.2821(5)	0.9908(4)	−0.0721(2)	0.126(4)	0.072(3)	0.062(3)	0.005(3)	0.010(3)	−0.001(2)
C(14)	4*e*	0.4146(3)	0.6232(4)	0.2820(2)	0.051(2)	0.050(2)	0.066(2)	−0.000(2)	0.027(2)	−0.001(2)
C(15)	4*e*	0.4478(3)	0.7384(4)	0.3410(2)	0.055(2)	0.049(2)	0.057(2)	−0.002(2)	0.025(2)	−0.003(2)
C(16)	4*e*	0.4781(4)	0.7032(4)	0.4353(3)	0.072(3)	0.050(2)	0.074(3)	−0.000(2)	0.034(2)	−0.002(2)
O(1)	4*e*	0.4140(3)	1.1832(3)	0.2156(2)	0.100(2)	0.045(2)	0.074(2)	0.000(1)	0.009(2)	−0.013(1)
O(2)	4*e*	0.3541(2)	1.0743(2)	0.0800(2)	0.079(2)	0.051(2)	0.060(2)	−0.004(1)	0.007(1)	−0.004(1)
O(3)	4*e*	0.3823(2)	0.6466(2)	0.1889(2)	0.081(2)	0.043(1)	0.065(2)	0.000(1)	0.024(1)	−0.005(1)

spasmolytic, and antibacterial activity [1–4]. A large number of structurally novel pyran derivatives have been reported to show substantial cytotoxic activity in vitro and in vivo [5–8]. Recognizing the considerable importance of the compounds, research focused on the synthesis of the dihydropyran derivatives [10].

In the crystal structure of the title compound, the pyrano[4,3-*b*]pyranyl moiety is basically planar, and the two planes are also essentially parallel to each other. However, the phenyl ring makes a torsion angle to the pyran ring in the compound.

NH···N and NH···O hydrogen bonds connect the molecules of the title structure to a ladder structure which runs along the crystallographic *b* direction.

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