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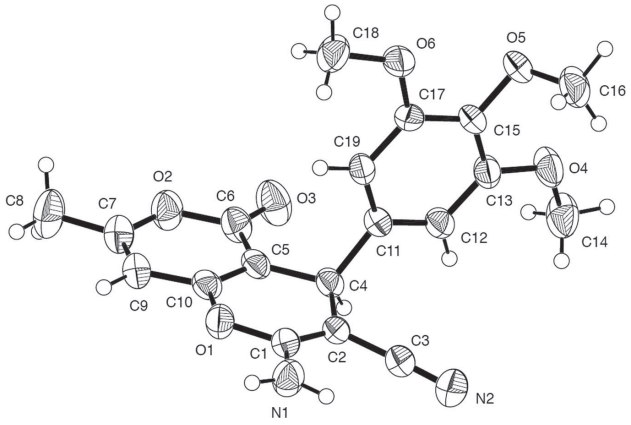
Crystal structure of 2-amino-4-(3,4,5-trimethoxyphenyl)-7-methyl-5-oxo-4*H*,5*H*-pyrano[4,3-*b*]-pyran-3-carbonitrile, $C_{19}H_{18}N_2O_6$



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Abstract

$C_{19}H_{18}N_2O_6$, monoclinic, $P2_1/c$ (no. 14), $a = 13.052(8)$ Å, $b = 10.091(6)$ Å, $c = 14.454(9)$ Å, $\beta = 114.446(11)°$, $V = 1733.0(19)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0490$, $wR_{ref}(F^2) = 0.1518$, $T = 296(2)$ 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 material

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

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.26 × 0.21 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8487, 3041, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2083
$N(param)_{refined}$:	245
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.07794(13)	0.62356(15)	0.66102(12)	0.0482(5)
O2	0.07846(13)	0.74641(16)	0.50112(11)	0.0511(5)
O3	0.20953(14)	0.95803(17)	0.51976(12)	0.0549(5)
O4	0.29142(12)	0.91682(15)	1.07149(11)	0.0426(4)
O5	0.54182(13)	0.70363(17)	1.04858(13)	0.0536(5)
O6	0.51258(15)	0.7891(2)	0.89905(14)	0.0647(6)
N1	0.1548(2)	1.2354(2)	0.80994(18)	0.0689(7)
N2	0.15421(17)	1.0640(2)	1.02712(15)	0.0528(6)
H2A	0.111785	1.126552	0.990864	0.063*
H2B	0.147108	1.036796	1.080554	0.063*
C1	0.0811(2)	0.5490(3)	0.74526(19)	0.0581(7)
H1A	0.032470	0.473498	0.721429	0.087*
H1B	0.156752	0.519646	0.784835	0.087*
H1C	0.056375	0.603344	0.786571	0.087*
C2	0.14261(18)	0.7354(2)	0.68065(17)	0.0371(6)
C3	0.14185(18)	0.7982(2)	0.59557(16)	0.0389(6)
C4	−0.0175(2)	0.8244(3)	0.4445(2)	0.0626(8)
H4A	−0.058673	0.784011	0.379407	0.094*
H4B	−0.064701	0.830260	0.480578	0.094*
H4C	0.006005	0.911625	0.435180	0.094*
C5	0.20973(18)	0.9080(2)	0.60686(16)	0.0403(6)
C6	0.2868(2)	1.0616(3)	0.5288(2)	0.0636(8)
H6A	0.278490	1.088305	0.462299	0.095*
H6B	0.271873	1.135721	0.562968	0.095*
H6C	0.362180	1.030683	0.567140	0.095*
C7	0.27312(17)	0.9567(2)	0.70329(16)	0.0395(6)
H7	0.318132	1.031006	0.711375	0.047*
C8	0.27008(17)	0.8959(2)	0.78732(16)	0.0358(5)
C9	0.20601(18)	0.7840(2)	0.77693(17)	0.0389(6)
H9	0.205381	0.741761	0.833860	0.047*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C10	0.48324(19)	0.7879(2)	0.96785(19)	0.0454(6)
C11	0.39532(17)	0.8643(2)	0.97471(16)	0.0370(6)
C12	0.25409(18)	1.0380(2)	0.92081(17)	0.0374(6)
C13	0.1978(2)	1.1457(2)	0.85876(18)	0.0460(6)
C14	0.23108(18)	1.0091(2)	1.00046(17)	0.0393(6)
C15	0.37471(17)	0.8509(2)	1.05793(16)	0.0367(6)
C16	0.43902(18)	0.7670(2)	1.13985(18)	0.0432(6)
H16	0.424258	0.762000	1.197426	0.052*
C17	0.5198(2)	0.6965(2)	1.13289(19)	0.0481(6)
C18	0.5964(2)	0.6030(3)	1.2090(2)	0.0699(9)
H18A	0.647405	0.565111	1.183892	0.105*
H18B	0.638312	0.649445	1.271275	0.105*
H18C	0.553089	0.533740	1.221261	0.105*
C19	0.33355(17)	0.9592(2)	0.89078(16)	0.0370(6)
H19	0.388731	1.020886	0.885187	0.044*

Experimental details

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

Discussion

Pyran derivatives may possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity [5]. A large number of structurally novel pyran derivatives have been reported to show substantial cytotoxic activity *in vitro* and *in vivo* [6]. Research focused on the synthesis of the dihydropyran derivatives [7]. Herein, we reported the structure of a new pyran derivative.

In the crystal structure of the title compound (*cf.* the figure), one methoxy group is connected to 4*H*,5*H*-pyrano[4,3-*b*]pyran-5-one unit at the 7-position. One cyano group and one amino group are linked to the 2 and 4-position of this system, respectively. Three methoxy groups are connected to C13, C15 and C17 atoms respectively. Above mentioned bond lengths fall in their normal ranges compared with those in previously studies [8, 9].

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References

1. Bruker: APEX3, SAINT-Plus, XPREP. Bruker AXS Inc., Madison, Wisconsin, USA (2016).
2. Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. Acta Crystallogr. **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Cryst. **42** (2009) 339–341.
5. Li, J.; Sui, Y.; Xin, J.; Du, X. L.; Li, J. T.; Huo, H. R.; Ma, H.; Wang, W. H.; Zhou, H. Y.; Zhan, H. D.; Wang, Z. J.; Li, C.; Sui, F; Li, X.: Synthesis of biscoumarin and dihydropyran derivatives with promising antitumor and antibacterial activities. Bioorg. Med. Chem. Lett. **25** (2015) 5520–5523.
6. Khan, K. M.; Iqbal, S.; Lodhi, M. A; Maharvi, G. M.; Ullah, Z.; Choudhary, M. I.; Rahman, A. U.; Perveen, S.: Biscoumarin: New class of urease inhibitors; economical synthesis and activity. Bioorg. Med. Chem. **12** (2004) 1963–1968.
7. Khan, A. T.; Lal, M.; Ali, S.; Musawwer, K.: One-pot three-component reaction for the synthesis of pyran annulated heterocyclic compounds using DMAP as a catalyst. Tetrahedron Lett. **52** (2011) 5327–5332.
8. Li, J.; Lv, C.-W.; Li, X.-J.; Qu, D.; Hou, Z.; Jia, M.; Luo, X. X.; Li, X.; Li, M. K.: Synthesis of biscoumarin and dihydropyran derivatives and evaluation of their antibacterial activity. Molecules. **20** (2015) 17469–17482.
9. Song, J.: Crystal structure of 2-amino-7-methyl-5-oxo-4-phenyl-4*H*,5*H*-pyrano[4,3-*b*]pyran-3-carbonitrile, $C_{16}H_{12}N_2O_3$. Z. Kristallogr. NCS **231** (2016) 747–748.