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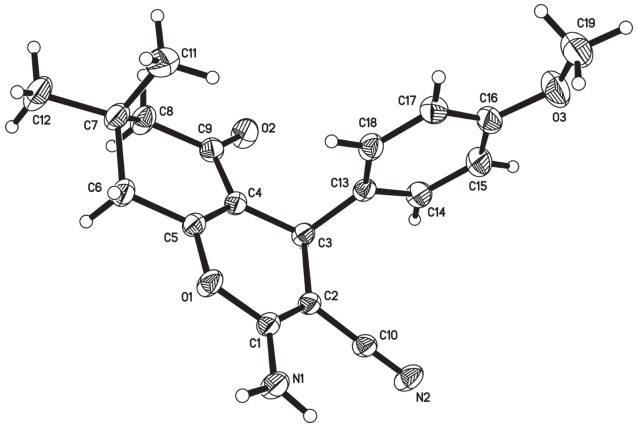
Crystal structural of 2-amino-4-(4-methoxyphenyl)-3-cyano-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo[*b*]pyran, $C_{19}H_{20}N_2O_3$



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Abstract

$C_{19}H_{20}N_2O_3$, monoclinic, $C2/c$ (no. 15), $a = 30.474(9)$ Å, $b = 9.331(2)$ Å, $c = 26.269(7)$ Å, $\beta = 112.591(3)°$, $V = 6897(3)$ Å^3, $Z = 16$, $R_{gt}(F) = 0.0462$, $wR_{ref}(F^2) = 0.1379$, $T = 293$ 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 [3]. A mixture of 3,5-cyclohexanedione (10 mmol), 4-methoxybenzaldehyde (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.

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Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker XSCANS, ω-scans
$2\theta_{max}$, completeness:	25°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	20997, 5987, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5246
$N(param)_{refined}$:	434
Programs:	Bruker programs [1], SHELX [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.14809(5)	1.02297(16)	0.17399(6)	0.0381(3)
C2	−0.18002(5)	1.06137(16)	0.12390(6)	0.0354(3)
C3	−0.22547(5)	0.97826(15)	0.09332(5)	0.0356(3)
H3A	−0.2289	0.9673	0.0549	0.043*
C4	−0.22039(5)	0.83177(16)	0.11853(6)	0.0359(3)
C5	−0.18641(5)	0.79948(16)	0.16732(6)	0.0378(3)
C6	−0.18050(6)	0.65953(17)	0.19612(7)	0.0449(4)
H6A	−0.1701	0.6763	0.2355	0.054*
H6B	−0.1559	0.6050	0.1899	0.054*
C7	−0.22636(6)	0.57136(17)	0.17659(7)	0.0465(4)
C8	−0.24676(7)	0.57154(18)	0.11336(7)	0.0522(4)
H8A	−0.2257	0.5171	0.1008	0.063*
H8B	−0.2773	0.5231	0.1001	0.063*
C9	−0.25327(5)	0.71834(17)	0.08817(6)	0.0411(4)
C10	−0.17424(5)	1.19432(16)	0.10165(6)	0.0380(3)
C11	−0.26249(7)	0.6359(2)	0.19723(9)	0.0659(5)
H11A	−0.2699	0.7320	0.1835	0.099*
H11B	−0.2494	0.6374	0.2368	0.099*
H11C	−0.2909	0.5791	0.1844	0.099*
C12	−0.21415(8)	0.4187(2)	0.19850(8)	0.0674(6)
H12A	−0.1917	0.3782	0.1850	0.101*
H12B	−0.2425	0.3616	0.1862	0.101*
H12C	−0.2005	0.4205	0.2381	0.101*
C13	−0.26786(5)	1.06294(16)	0.09403(5)	0.0362(3)
C14	−0.29055(5)	1.15886(18)	0.05198(6)	0.0460(4)
H14A	−0.2824	1.1616	0.0214	0.055*
C15	−0.32516(6)	1.2507(2)	0.05464(7)	0.0554(5)
H15A	−0.3397	1.3152	0.0261	0.067*
C16	−0.33828(5)	1.24758(19)	0.09935(7)	0.0518(4)
C17	−0.31740(6)	1.14923(18)	0.14079(7)	0.0493(4)
H17A	−0.3269	1.1433	0.1704	0.059*
C18	−0.28222(5)	1.05935(17)	0.13802(6)	0.0436(4)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H18A	−0.2678	0.9947	0.1665	0.052*
C19	−0.38051(8)	1.3629(3)	0.14812(11)	0.0812(7)
H19A	−0.4056	1.4313	0.1420	0.122*
H19B	−0.3521	1.3960	0.1773	0.122*
H19C	−0.3898	1.2723	0.1582	0.122*
C20	0.10800(5)	0.67061(15)	0.41374(6)	0.0360(3)
C21	0.07336(5)	0.63017(15)	0.36553(5)	0.0352(3)
C22	0.02588(5)	0.70811(16)	0.34203(5)	0.0358(3)
H22A	0.0159	0.7125	0.3019	0.043*
C23	0.03361(5)	0.85852(16)	0.36377(6)	0.0362(3)
C24	0.07000(5)	0.89322(15)	0.41014(6)	0.0366(3)
C25	0.07924(6)	1.03556(16)	0.43747(6)	0.0432(4)
H25A	0.1055	1.0808	0.4314	0.052*
H25B	0.0888	1.0222	0.4769	0.052*
C26	0.03608(5)	1.13558(16)	0.41659(6)	0.0410(4)
C27	0.01303(6)	1.12305(17)	0.35373(7)	0.0467(4)
H27A	−0.0157	1.1803	0.3404	0.056*
H27B	0.0346	1.1624	0.3381	0.056*
C28	0.00079(5)	0.97200(17)	0.33351(6)	0.0412(4)
C29	0.05269(7)	1.28811(18)	0.43382(8)	0.0551(4)
H29A	0.0260	1.3520	0.4204	0.083*
H29B	0.0756	1.3150	0.4187	0.083*
H29C	0.0670	1.2937	0.4733	0.083*
C30	0.00003(7)	1.0937(2)	0.44122(9)	0.0639(5)
H30A	−0.0112	0.9982	0.4298	0.096*
H30B	−0.0263	1.1592	0.4286	0.096*
H30C	0.0148	1.0973	0.4807	0.096*
C31	0.07914(5)	0.50469(16)	0.33874(5)	0.0368(3)
C32	−0.01213(5)	0.62672(15)	0.35475(6)	0.0364(3)
C33	−0.01510(6)	0.63171(17)	0.40595(6)	0.0432(4)
H33A	0.0049	0.6934	0.4326	0.052*
C34	−0.04697(6)	0.54737(19)	0.41877(7)	0.0502(4)
H34A	−0.0483	0.5531	0.4535	0.060*
C35	−0.07662(6)	0.45504(19)	0.37961(7)	0.0501(4)
C36	−0.07495(6)	0.4511(2)	0.32769(7)	0.0552(4)
H36A	−0.0954	0.3911	0.3007	0.066*
C37	−0.04302(6)	0.53591(18)	0.31586(6)	0.0476(4)
H37A	−0.0423	0.5319	0.2808	0.057*
C38	−0.11020(8)	0.3585(3)	0.44067(10)	0.0849(7)
H38A	−0.1353	0.2953	0.4400	0.127*
H38B	−0.1162	0.4530	0.4509	0.127*
H38C	−0.0804	0.3243	0.4670	0.127*
O1	−0.15176(4)	0.89493(11)	0.19767(4)	0.0470(3)
O2	−0.28406(4)	0.74220(13)	0.04271(5)	0.0579(3)
O3	−0.37177(5)	1.34656(16)	0.09906(7)	0.0776(4)
O4	0.10456(4)	0.79598(11)	0.43922(4)	0.0435(3)
O5	−0.03424(4)	0.94284(14)	0.29234(5)	0.0608(4)
O6	−0.10828(5)	0.36276(16)	0.38775(6)	0.0706(4)
N1	−0.11059(5)	1.09670(15)	0.20739(6)	0.0540(4)
H1A	−0.1044	1.1799	0.1977	0.065*
H1B	−0.0926	1.0611	0.2386	0.065*
N2	−0.17060(5)	1.30126(16)	0.08240(6)	0.0530(4)
N3	0.14843(5)	0.60331(15)	0.44313(5)	0.0495(4)
H3B	0.1551	0.5230	0.4318	0.059*
H3C	0.1678	0.6402	0.4734	0.059*
N4	0.08292(5)	0.40415(15)	0.31550(5)	0.0513(4)

Experimental details

Carbon-bound hydrogen atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). U_{iso}(H) = 1.5U_{eq}(C).

Discussion

Dihydropyrans represent one of the most active classes of compounds possessing a wide spectrum of biological activities [4–6], including anticoagulant, insecticidal, anthelminthic, hypnotic, antifungal, phytoalexin, and HIV protease inhibition. Thus, dihydropyrans are attractive targets in organic synthesis [7].

In the crystal structure of the title compound, the 7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[*b*]pyran unit is typically folded [8]. Generally all bond lengths and angles are as expected [9].

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