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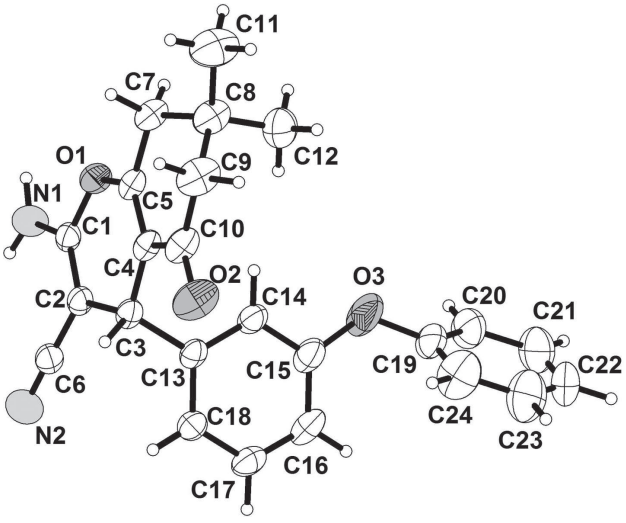
Jing Li* and Feng Sui

Crystal structure of 2-amino-7,7-dimethyl-5-oxo-4-(3-phenoxy-phenyl)-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile



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Abstract

$C_{24}H_{22}N_2O_3$, monoclinic, $C2/c$ (no. 15), $a = 30.130(8)$ Å, $b = 8.825(2)$ Å, $c = 16.021(4)$ Å, $\beta = 103.293(4)°$, $V = 4145.8(18)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0445$, $wR_{ref}(F^2) = 0.1258$, $T = 293$ K.

CCDC no.: 1451558

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.

Source of material

The title compound was synthesized according to a reported procedure [10]. A mixture of 1,1-dimethyl-3,5-cyclohexanedione (10 mmol), 3-phenoxybenzaldehyde (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

Table 1: Data collection and handling.

Crystal:	Colorless, prism, size 0.3×0.5×0.6 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.82 cm^{-1}
Diffractometer, scan mode:	Bruker APEX, ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16480, 3629
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3282
$N(param)_{refined}$:	262
Programs:	SHELX [11]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	8*f*	0.0262	0.5836	−0.1646	0.048
H(7A)	8*f*	0.0746	0.2170	0.0579	0.061
H(7B)	8*f*	0.1202	0.3014	0.0985	0.061
H(9A)	8*f*	0.1113	0.1311	−0.1390	0.084
H(9B)	8*f*	0.0677	0.1159	−0.1021	0.084
H(11A)	8*f*	0.1500	0.0262	0.0830	0.133
H(11B)	8*f*	0.1456	−0.0384	−0.0097	0.133
H(11C)	8*f*	0.1021	−0.0214	0.0275	0.133
H(12A)	8*f*	0.1887	0.2553	0.0469	0.117
H(12B)	8*f*	0.1661	0.3552	−0.0324	0.117
H(12C)	8*f*	0.1853	0.1943	−0.0462	0.117
H(14A)	8*f*	0.1444	0.5999	−0.0766	0.060
H(16A)	8*f*	0.1735	0.9117	−0.2381	0.071
H(17A)	8*f*	0.0965	0.9469	−0.2932	0.065
H(18A)	8*f*	0.0435	0.8054	−0.2443	0.058
H(20A)	8*f*	0.2824	0.8648	−0.0541	0.091
H(21A)	8*f*	0.3443	0.9018	−0.1146	0.122
H(22A)	8*f*	0.3428	0.8171	−0.2495	0.136
H(23A)	8*f*	0.2804	0.6877	−0.3244	0.138
H(24A)	8*f*	0.2191	0.6450	−0.2645	0.116
H(1A)	8*f*	0.0276	0.8194	0.0929	0.065
H(1B)	8*f*	0.0498	0.6980	0.1527	0.065

***Corresponding author: Jing Li,** School of Chemistry and Chemical Engineering, Xi'an University, Xi'an, China, e-mail: lijing3157@aliyun.com
Feng Sui: Institute of Chinese Materia Medica, China Academy of Chinese Medical Sciences, Beijing, China

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.04952(4)	0.6551(2)	0.03549(8)	0.0330(6)	0.0543(8)	0.0329(7)	0.0001(6)	0.0095(5)	−0.0009(6)
C(2)	8*f*	0.03860(4)	0.6936(2)	−0.04881(8)	0.0339(6)	0.0572(8)	0.0304(7)	−0.0006(6)	0.0084(5)	−0.0006(6)
C(3)	8*f*	0.05318(4)	0.6012(2)	−0.11803(8)	0.0355(6)	0.0579(8)	0.0270(6)	−0.0071(6)	0.0064(5)	−0.0044(6)
C(4)	8*f*	0.07048(4)	0.4505(2)	−0.08095(8)	0.0402(7)	0.0488(8)	0.0339(7)	−0.0124(6)	0.0118(5)	−0.0059(6)
C(5)	8*f*	0.07960(4)	0.4203(2)	0.00310(8)	0.0407(7)	0.0457(7)	0.0354(7)	−0.0106(6)	0.0145(5)	−0.0054(5)
C(6)	8*f*	0.01555(5)	0.8315(2)	−0.07267(8)	0.0413(7)	0.068(1)	0.0280(6)	0.0049(7)	0.0084(5)	−0.0017(6)
C(7)	8*f*	0.09899(6)	0.2774(2)	0.0448(1)	0.0650(9)	0.0468(8)	0.0461(8)	−0.0064(7)	0.0226(7)	0.0010(6)
C(8)	8*f*	0.12384(6)	0.1833(2)	−0.0107(1)	0.084(1)	0.0447(8)	0.056(1)	−0.0007(8)	0.0280(9)	−0.0050(7)
C(9)	8*f*	0.09408(8)	0.1791(2)	−0.1019(1)	0.108(2)	0.0500(9)	0.057(1)	−0.0096(9)	0.027(1)	−0.0164(8)
C(10)	8*f*	0.07777(5)	0.3299(2)	−0.13923(9)	0.0633(9)	0.0566(9)	0.0397(8)	−0.0188(7)	0.0173(7)	−0.0134(6)
C(11)	8*f*	0.13104(9)	0.0224(2)	0.0260(1)	0.143(2)	0.049(1)	0.081(1)	0.012(1)	0.039(1)	−0.0019(9)
C(12)	8*f*	0.17032(7)	0.2536(2)	−0.0106(1)	0.076(1)	0.083(1)	0.084(1)	0.013(1)	0.036(1)	0.004(1)
C(13)	8*f*	0.08836(4)	0.6877(2)	−0.15476(8)	0.0421(7)	0.0503(8)	0.0270(6)	−0.0041(6)	0.0111(5)	−0.0050(5)
C(14)	8*f*	0.13453(5)	0.6683(2)	−0.12111(9)	0.0448(8)	0.068(1)	0.0362(7)	−0.0095(7)	0.0085(6)	0.0081(7)
C(15)	8*f*	0.16602(5)	0.7498(2)	−0.1531(1)	0.0467(8)	0.090(1)	0.0413(8)	−0.0205(8)	0.0087(6)	0.0045(8)
C(16)	8*f*	0.15218(6)	0.8555(2)	−0.2174(1)	0.070(1)	0.069(1)	0.0442(8)	−0.0256(8)	0.0246(8)	−0.0030(7)
C(17)	8*f*	0.10628(6)	0.8756(2)	−0.2502(1)	0.075(1)	0.0514(8)	0.0419(8)	0.0020(7)	0.0253(7)	0.0045(7)
C(18)	8*f*	0.07442(5)	0.7919(2)	−0.22034(9)	0.0513(8)	0.0599(9)	0.0354(7)	0.0060(7)	0.0154(6)	0.0022(6)
C(19)	8*f*	0.24566(5)	0.7516(2)	−0.1541(1)	0.0445(8)	0.085(1)	0.065(1)	−0.0151(8)	0.0138(7)	0.0154(9)
C(20)	8*f*	0.28206(6)	0.8282(3)	−0.1087(1)	0.0484(9)	0.106(2)	0.069(1)	−0.016(1)	0.0056(8)	0.004(1)
C(21)	8*f*	0.31871(7)	0.8507(3)	−0.1453(2)	0.044(1)	0.128(2)	0.130(2)	−0.024(1)	0.011(1)	0.025(2)
C(22)	8*f*	0.31808(9)	0.7999(4)	−0.2251(2)	0.066(1)	0.154(3)	0.139(3)	0.032(2)	0.060(2)	0.051(2)
C(23)	8*f*	0.2810(1)	0.7232(4)	−0.2695(2)	0.114(2)	0.135(2)	0.113(2)	0.023(2)	0.062(2)	−0.015(2)
C(24)	8*f*	0.24443(9)	0.6980(3)	−0.2342(2)	0.099(2)	0.103(2)	0.090(2)	−0.029(1)	0.025(1)	−0.022(1)
O(1)	8*f*	0.07144(3)	0.5221(1)	0.06310(5)	0.0501(5)	0.0528(6)	0.0286(5)	0.0033(4)	0.0118(4)	−0.0015(4)
O(2)	8*f*	0.06920(5)	0.3526(2)	−0.21663(7)	0.102(1)	0.0775(8)	0.0362(6)	−0.0128(7)	0.0196(6)	−0.0169(5)
O(3)	8*f*	0.21102(4)	0.7228(2)	−0.11291(9)	0.0426(7)	0.213(2)	0.0700(9)	−0.0333(9)	0.0051(6)	0.051(1)
N(1)	8*f*	0.04134(5)	0.7334(2)	0.10149(7)	0.0672(8)	0.0684(8)	0.0294(6)	0.0215(7)	0.0144(5)	0.0010(5)
N(2)	8*f*	−0.00354(5)	0.9427(2)	−0.09400(8)	0.0653(8)	0.078(1)	0.0413(7)	0.0202(8)	0.0101(6)	0.0013(7)

filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Discussion

Dihydropyran derivatives represent one of the most active classes of compounds possessing a wide spectrum of biological activities, including anticoagulant, insecticidal, anthelminthic, hypnotic, antifungal, phytoalexin, and HIV protease inhibition [1–5]. The interesting biological activity of these dihydropyrans made these compounds attractive targets in organic synthesis [6–9].

In the crystal structure of the title compound, the chromene moiety is basically planar, with the CMe_2 group twisted out of this plane (see the figure). Both phenyl rings are twisted related to the chromene moiety.

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