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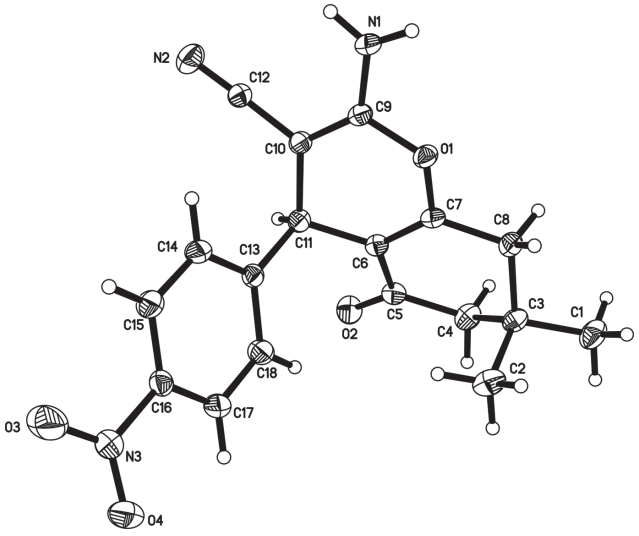
Crystal structure of 2-amino-4-(4-nitro-phenyl)-7, 7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile, $C_{18}H_{17}N_3O_4$



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Abstract

$C_{18}H_{17}N_3O_4$, monoclinic, $P2_1/n$ (no. 14), $a = 9.5820(6)$ Å, $b = 16.5417(11)$ Å, $c = 10.6951(7)$ Å, $\beta = 111.060(8)°$, $V = 1581.97(18)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0437$, $wR_{ref}(F^2) = 0.1307$, $T = 293$ 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 [4]. A mixture of 3,5-cyclohexanedione (10 mmol), 4-nitrobenzaldehyde (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

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Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	0.18 × 0.17 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.10 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, Completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6244, 2787, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2163
$N(param)_{refined}$:	233
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	−0.3273(2)	0.07147(14)	0.1299(2)	0.0499(6)
H1A	−0.3711	0.0698	0.1977	0.075*
H1B	−0.3342	0.0190	0.0897	0.075*
H1C	−0.3797	0.1102	0.0625	0.075*
C2	−0.0814(2)	0.03662(14)	0.3041(2)	0.0508(6)
H2A	−0.1268	0.0368	0.3709	0.076*
H2B	0.0216	0.0523	0.3443	0.076*
H2C	−0.0875	−0.0167	0.2670	0.076*
C3	−0.1632(2)	0.09601(12)	0.19325(18)	0.0348(5)
C4	−0.1515(2)	0.18125(12)	0.2511(2)	0.0397(5)
H4A	−0.2178	0.2165	0.1831	0.048*
H4B	−0.1864	0.1801	0.3257	0.048*
C5	0.0028(2)	0.21668(11)	0.29855(18)	0.0322(4)
C6	0.10666(19)	0.18889(11)	0.23535(17)	0.0289(4)
C7	0.05238(19)	0.14015(11)	0.12927(17)	0.0285(4)
C8	−0.0943(2)	0.09839(12)	0.08433(18)	0.0365(5)
H8A	−0.0816	0.0435	0.0583	0.044*
H8B	−0.1624	0.1259	0.0061	0.044*
C9	0.25074(19)	0.17119(11)	0.05648(17)	0.0294(4)
C10	0.31476(19)	0.22024(11)	0.16249(17)	0.0303(4)
C11	0.26542(19)	0.21979(11)	0.28212(17)	0.0301(4)
H11A	0.2649	0.2759	0.3116	0.036*
C12	0.4290(2)	0.27405(12)	0.16063(18)	0.0343(5)
C13	0.37009(19)	0.17106(11)	0.40153(17)	0.0297(4)
C14	0.5130(2)	0.15067(13)	0.41102(19)	0.0381(5)
H14A	0.5459	0.1658	0.3425	0.046*
C15	0.6091(2)	0.10840(13)	0.51943(19)	0.0404(5)
H15A	0.7055	0.0957	0.5245	0.049*
C16	0.55888(19)	0.08571(11)	0.61917(17)	0.0332(5)
C17	0.4173(2)	0.10426(13)	0.61380(18)	0.0403(5)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H17A	0.3849	0.0881	0.6821	0.048*
C18	0.3238(2)	0.14713(13)	0.50571(18)	0.0402(5)
H18A	0.2280	0.1604	0.5020	0.048*
O1	0.13056(13)	0.12379(8)	0.04652(11)	0.0331(3)
O2	0.03981(15)	0.27017(9)	0.38411(14)	0.0439(4)
O3	0.78893(19)	0.03345(14)	0.74607(17)	0.0874(7)
O4	0.60883(18)	0.01126(12)	0.81242(16)	0.0709(6)
N1	0.2906(2)	0.16040(11)	−0.05033(16)	0.0394(4)
H1D	0.373(2)	0.1866(13)	−0.052(2)	0.047*
H1E	0.225(2)	0.1448(14)	−0.120(2)	0.047*
N2	0.5227(2)	0.31769(12)	0.16173(19)	0.0520(5)
N3	0.65883(19)	0.03974(11)	0.73353(16)	0.0441(5)

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

Experimental details

Hydrogen atoms bonded to C and N were positioned geometrically and refined using a riding model, with C···H = 0.97 Å and N···H = 0.86 Å. U_{iso}(H) was set to 1.2 times U_{eq}(C) and U_{eq}(N).

Discussion

Dihydropyrans represent classes of compounds possessing a wide spectrum of biological activities [3–5]. The biological activity of dihydropyrans made these compounds attractive targets in organic synthesis [6–8].

The core moiety of the title molecule, consisting of two rings and sharing one edge (*cf*. the figure), is well known. In all the known compounds both rings are not planar. Especially the related bond lengths are all in their normal ranges. The structural features of the title compound are similar with those previously reported compounds [9–11].

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