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The crystal structure of 2-amino-4-(4-nitrophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile, $C_{16}H_{13}N_3O_4$

Table 1: Data collection and handling.

Crystal:	Colourless blocks
Wavelength:	Size $0.16 \times 0.14 \times 0.13$ mm
μ :	Mo K_{α} radiation (0.71073 Å)
Diffractometer, scan mode:	1.0 cm ⁻¹
2 $\theta_{\max}$, completeness:	Bruker FRAMBO, φ and ω
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	50.2°, >99%
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	5300, 2630, 0.024
$N(\text{param})_{\text{refined}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1612
Programs:	208
	SHELX [7], Bruker programs [8]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.28139(18)	0.6659(2)	0.43156(12)	0.0425(6)
C2	0.26607(17)	0.54934(19)	0.44472(12)	0.0410(6)
C3	0.16180(17)	0.49328(18)	0.42857(12)	0.0401(6)
H3A	0.1554	0.4605	0.4716	0.048*
C4	0.08253(18)	0.58851(18)	0.40315(12)	0.0415(6)
C5	0.10368(17)	0.7018(2)	0.39009(12)	0.0448(6)
C6	0.02913(19)	0.7992(2)	0.36293(15)	0.0600(8)
H6A	0.0490	0.8455	0.3290	0.072*
H6B	0.0287	0.8530	0.4002	0.072*
C7	-0.0764(2)	0.7482(3)	0.33002(19)	0.0868(11)
H7A	-0.1260	0.8129	0.3218	0.104*
H7B	-0.0808	0.7126	0.2858	0.104*
C8	-0.1013(2)	0.6543(2)	0.3753(2)	0.0887(11)
H8A	-0.1078	0.6931	0.4162	0.106*
H8B	-0.1665	0.6181	0.3507	0.106*
C9	-0.02312(18)	0.5574(2)	0.39671(14)	0.0530(7)
C10	0.35026(19)	0.4738(2)	0.47195(13)	0.0486(7)
C11	0.14793(16)	0.38958(18)	0.37728(12)	0.0377(6)
C12	0.13462(19)	0.27397(19)	0.39707(13)	0.0501(7)
H12A	0.1304	0.2604	0.4410	0.060*
C13	0.12739(19)	0.1782(2)	0.35316(14)	0.0567(7)
H13A	0.1186	0.1003	0.3672	0.068*
C14	0.13320(18)	0.1989(2)	0.28918(14)	0.0487(7)
C15	0.1440(2)	0.3131(2)	0.26668(14)	0.0587(7)
H15A	0.1465	0.3260	0.2223	0.070*
C16	0.15119(19)	0.4080(2)	0.31136(13)	0.0554(7)
H16A	0.1583	0.4859	0.2968	0.066*
O1	0.20223(12)	0.74254(13)	0.40217(9)	0.0505(5)

DOI 10.1515/ncrs-2016-0063

Received February 29, 2016; accepted June 9, 2016; available online June 30, 2016

Abstract

$C_{16}H_{13}N_3O_4$, monoclinic, $C2/c$ (No. 15), $a = 13.8536(12)$ Å, $b = 11.0922(7)$ Å, $c = 20.432(2)$ Å, $\beta = 108.491(10)^\circ$, $V = 2977.6(4)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0533$, $wR_{\text{ref}}(F^2) = 0.1452$, $T = 293(2)$ K.

CCDC no.: 1484300

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
O2	−0.04389(13)	0.45638(15)	0.41209(11)	0.0727(6)
O3	0.1369(3)	0.1106(2)	0.18894(15)	0.1443(13)
O4	0.1262(3)	−0.0012(2)	0.26441(17)	0.1930(19)
N1	0.37054(15)	0.72362(17)	0.44523(11)	0.0577(6)
H1A	0.4265	0.6858	0.4646	0.069*
H1B	0.3718	0.7985	0.4346	0.069*
N2	0.41695(18)	0.4076(2)	0.49423(13)	0.0721(8)
N3	0.1304(2)	0.0956(2)	0.24350(16)	0.0752(8)

Source of material

The title compound was synthesized according to a reported procedure [6]. 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 temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

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

Discussion

Pyrans are well-known natural products displaying a broad range of biological activities [1]. More recently, other activities have been described for these compounds such as antidepressant, vasorelaxant and platelet-aggregation inhibition, enzymatic inhibition of MAO, antioxidant activity, and proapoptotic activity [2, 3]. The discovery, structural

modification and structure-activity relationships studies of pyrans as anti-cancer agents, have been the object of several updates and reviews [4, 5].

In the crystal structure of the title compound, the 4*H*-pyran ring is nearly planar whereas the adjacent ring containing the keto group is folded. The 4*H*-pyran ring plane is almost perpendicular to the nitro-phenyl moiety.

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