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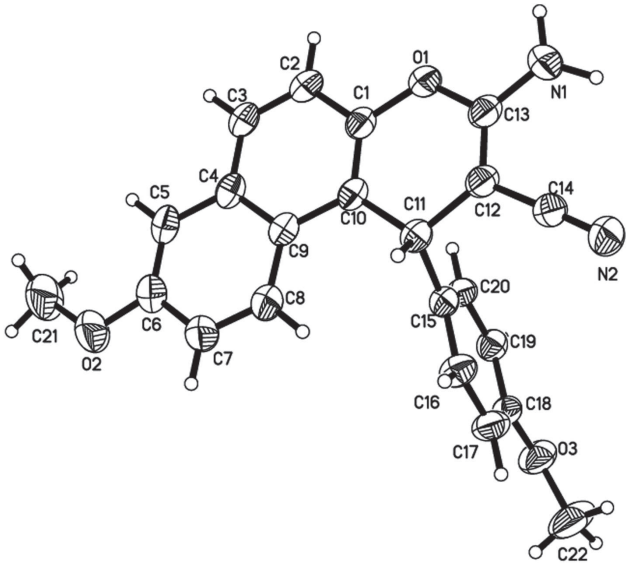
Crystal structure of 3-amino-8-methoxy-1-(4-methoxy phenyl)-1*H*-benzo[*f*]chromene-2-carbonitrile, $C_{22}H_{18}N_2O_3$



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Abstract

$C_{22}H_{18}N_2O_3$, monoclinic, *C*2/*c* (no. 15), $a = 35.4037(10)$ Å, $b = 6.0294(2)$ Å, $c = 20.1702(7)$ Å, $\beta = 122.239(4)°$, $V = 3641.8(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0482$, $wR_{ref}(F^2) = 0.1406$, $T = 296(2)$ K.

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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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.33 × 0.26 × 0.15 mm
Wavelength:	Cu *Kα* radiation (1.54178 Å)
μ:	7.1 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	133.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15808, 3215, 0.051
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2112
$N(param)_{refined}$:	254
Programs:	SHELX [19], Bruker programs [20]

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Source of material

A mixture of 6-methoxy-2-naphthol (0.01 mol), 4-anisaldehyde (0.01 mol), and malononitrile (0.01 mol), in absolute ethanol (30 mL) in the presence of piperidine (0.5 mL) was heated under microwave irradiation conditions for 2 min at 140 °C. The formed solid product was collected by filtration, washed with methanol and recrystallized from ethanol to give the title compound as yellow crystals; yield 88%; M.p.: 495–496 K (Lit. M.p.: 493–494 K [1]).

Experimental details

The methyl groups were idealized and refined using rigid groups allowed to rotate about the C—C bond (AFIX 137 option of the SHELX program [13]). The U_{iso} values of the hydrogen

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.39568(5)	0.4510(3)	0.35450(9)	0.0520(4)
O2	0.18840(6)	0.2801(4)	0.38573(13)	0.0857(7)
O3	0.45879(6)	0.6747(3)	0.77610(9)	0.0675(5)
N1	0.45598(8)	0.6474(4)	0.38627(15)	0.0655(7)
N2	0.45686(7)	1.1159(4)	0.49854(13)	0.0604(6)
C1	0.35758(7)	0.4052(4)	0.35625(12)	0.0442(5)
C2	0.33393(7)	0.2191(4)	0.31141(13)	0.0509(6)
H2A	0.3439	0.1396	0.2829	0.061*
C3	0.29653(7)	0.1542(4)	0.30926(13)	0.0515(6)
H3A	0.2808	0.0266	0.2798	0.062*
C4	0.28068(7)	0.2727(4)	0.34997(12)	0.0464(5)
C5	0.24123(7)	0.2065(4)	0.34635(14)	0.0559(6)
H5A	0.2256	0.0779	0.3175	0.067*
C6	0.22572(8)	0.3270(5)	0.38428(15)	0.0604(7)
C7	0.24831(8)	0.5201(5)	0.42562(16)	0.0666(7)
H7A	0.2368	0.6054	0.4506	0.080*
C8	0.28637(8)	0.5860(4)	0.43024(15)	0.0593(7)
H8A	0.3012	0.7163	0.4588	0.071*
C9	0.30428(7)	0.4645(4)	0.39334(12)	0.0447(5)
C10	0.34458(7)	0.5288(4)	0.39763(12)	0.0427(5)
C11	0.37116(7)	0.7288(4)	0.44528(12)	0.0438(5)
H11A	0.3508	0.8603	0.4259	0.053*
C12	0.40867(7)	0.7727(4)	0.43146(12)	0.0459(5)
C13	0.42061(7)	0.6313(4)	0.39378(13)	0.0478(6)
C14	0.43557(7)	0.9628(4)	0.46799(13)	0.0470(5)
C15	0.39082(7)	0.7074(4)	0.53316(12)	0.0411(5)
C16	0.38758(8)	0.8802(4)	0.57454(13)	0.0536(6)
H16A	0.3698	1.0055	0.5469	0.064*
C17	0.40960(8)	0.8754(4)	0.65561(14)	0.0570(6)
H17A	0.4070	0.9966	0.6831	0.068*
C18	0.43500(7)	0.6955(4)	0.69573(12)	0.0482(6)
C19	0.43809(7)	0.5169(4)	0.65544(13)	0.0478(6)
H19A	0.4552	0.3901	0.6832	0.057*
C20	0.41615(7)	0.5245(4)	0.57464(12)	0.0448(5)
H20A	0.4185	0.4026	0.5472	0.054*
C21	0.16506(11)	0.0831(6)	0.3491(2)	0.1030(13)
H21A	0.1418	0.0585	0.3607	0.154*
H21B	0.1859	−0.0422	0.3687	0.154*
H21C	0.1512	0.0962	0.2922	0.154*
C22	0.46633(12)	0.8713(6)	0.82080(17)	0.0942(11)
H22A	0.4871	0.8389	0.8765	0.141*
H22B	0.4379	0.9242	0.8124	0.141*
H22C	0.4792	0.9858	0.8041	0.141*
H2N1	0.4776(9)	0.720(4)	0.4173(16)	0.063(8)*
H1N1	0.4586(10)	0.546(6)	0.3549(19)	0.097(11)*

atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C, N).

Discussion

Heterocyclic compounds containing oxygen are of interest due to their properties as new drugs. Literature reveals that chromenes and benzochromenes are an important chemical synthon, associated with a broad range of pharmacological activities such as antimicrobial [2–5], anticancer agent [6] hypolipidemic [7], antioxidant [8, 9], analgesic [10], antileishmanial [11, 12], vascular-disrupting activity [13], estrogenic anticoagulant and antispasmolytic [14], blood platelet antiaggregating [15] effects and activities. As part of our programmed aim to develop new methodologies for the preparation of 1*H*-benzo[*f*]chromene derivatives [16–18], we have synthesized the title compound under microwave irradiation conditions.

In the title compound, the asymmetric unit of the title compound contains one independent molecule. The molecules are packed in the crystal structure *via* two strong classical intermolecular hydrogen bonds, N1—H2N1···N2^{i} and N1—H1N1···O3^{ii}. The H···A distances are 2.25(3) and 2.08(4) Å, respectively and the angles are 172(3) and 175(3)°, respectively. Symmetry codes: (i) $-x+1$, $-y+2$, $-z+1$; (ii) x, $-y+1$, $z-1/2$. All bond lengths and angles are in the expected ranges.

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