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Crystal structure of 6-amino-8-(2-methoxy-phenyl)-2-methyl-2,3,8,8a-tetrahydro-1*H*-iso-quinoline-5,7,7-tricarbonitrile monohydrate, $C_{20}H_{21}N_5O_2$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.21 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.8 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
2 $\theta_{\max}$, completeness:	55.8°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11291, 4406, 0.058
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2384
$N(\text{param})_{\text{refined}}$:	252
Programs:	Bruker programs [1], OLEX2 [2]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.44937(11)	0.7916(2)	0.52297(14)	0.0677(6)
N1	0.21574(12)	1.1754(2)	0.71135(15)	0.0470(5)
N2	0.02070(14)	1.1087(2)	0.32157(18)	0.0631(6)
N3	0.10546(13)	0.7542(2)	0.35244(15)	0.0558(6)
H3B	0.069953	0.795997	0.310111	0.067*
H3A	0.119588	0.665815	0.345827	0.067*
N4	0.30992(14)	0.5610(3)	0.41960(18)	0.0673(7)
N5	0.09206(15)	0.6047(2)	0.61049(19)	0.0636(7)
C1	0.33464(14)	0.7584(2)	0.63136(17)	0.0393(5)
C2	0.30775(16)	0.7022(3)	0.71698(18)	0.0483(6)
H2	0.249533	0.723152	0.736230	0.058*
C3	0.36616(18)	0.6154(3)	0.7743(2)	0.0582(7)
H3	0.347369	0.580521	0.831444	0.070*
C4	0.45242(18)	0.5818(3)	0.7450(2)	0.0650(8)
H4	0.490618	0.520969	0.781508	0.078*
C5	0.48195(16)	0.6379(3)	0.6623(2)	0.0603(7)
H5	0.540528	0.616349	0.644310	0.072*
C6	0.42448(14)	0.7273(3)	0.60479(19)	0.0480(6)
C7	0.5339(2)	0.7496(5)	0.4850(3)	0.1236(18)
H7B	0.542450	0.802795	0.428249	0.185*
H7A	0.583993	0.768568	0.530737	0.185*
H7C	0.532019	0.649465	0.470668	0.185*
C8	0.26883(13)	0.8446(2)	0.56429(16)	0.0371(5)
H8	0.306876	0.894833	0.520374	0.044*
C9	0.21083(13)	0.9596(2)	0.61114(16)	0.0378(5)
H9	0.166744	0.911557	0.649740	0.045*
C10	0.15770(14)	1.0459(2)	0.53468(17)	0.0421(6)
C11	0.12199(14)	0.9688(3)	0.44856(17)	0.0423(6)
C12	0.06474(15)	1.0455(3)	0.37805(19)	0.0472(6)

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Abstract

$C_{20}H_{21}N_5O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 14.594(4)$ Å, $b = 9.366(5)$ Å, $c = 14.147(4)$ Å, $\beta = 93.749(18)^\circ$, $V = 1929.6(12)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.061$, $wR_{\text{ref}}(F^2) = 0.213$, $T = 296(2)$ K.

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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.

Source of materials

A mixture of *N*-methylpiperidin-4-one (10 mmol), 2-methoxybenzaldehyde (10 mmol), and malononitrile (20 mmol) in ethanol (100 mL) was refluxed for 2–3 h. After

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C13	0.13910(13)	0.8276(2)	0.42941(17)	0.0405(5)
C14	0.20240(13)	0.7427(2)	0.50190(16)	0.0374(5)
C15	0.26066(15)	0.6386(2)	0.45294(17)	0.0439(6)
C16	0.14140(15)	0.6611(2)	0.56299(18)	0.0427(6)
C17	0.14534(18)	1.1868(3)	0.5469(2)	0.0562(7)
H17	0.112636	1.236972	0.498977	0.067*
C18	0.1807(2)	1.2675(3)	0.6321(2)	0.0601(7)
H18B	0.229685	1.330118	0.614543	0.072*
H18A	0.131722	1.326890	0.653586	0.072*
C19	0.27131(14)	1.0585(2)	0.67573(18)	0.0437(6)
H19A	0.298916	1.004212	0.728623	0.052*
H19B	0.320313	1.097621	0.640499	0.052*
C20	0.26917(18)	1.2600(3)	0.7836(2)	0.0628(8)
H20B	0.231516	1.335432	0.805710	0.094*
H20C	0.288851	1.199413	0.835788	0.094*
H20A	0.321900	1.300163	0.756328	0.094*
O1W	0.10596(13)	1.0467(2)	0.85330(16)	0.0632(6)
H1WA	0.131(3)	1.087(4)	0.806(2)	0.117(15)*
H1WB	0.0511(13)	1.071(3)	0.848(2)	0.080(10)*

cooling to room temperature, the precipitate was filtered off, washed with ice water and ethanol, and dried in a vacuum.

Experimental details

H atoms bonded to C were positioned geometrically and refined using a riding model, with C—H = 0.97 Å and with $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$.

Comment

N-Methylpiperidin-4-one derivatives have gained great importance due to their diverse biological properties including antitumor, antifungal, antimicrobial, anti-inflammatory and insecticidal activities [3]. A large number of structurally novel *N*-methylpiperidin-4-one derivatives have been reported to show substantial cytotoxic activity in vitro and

in vivo [4]. Recognizing the considerable importance of the compounds, research focused on the synthesis of new *N*-methylpiperidin-4-one derivatives.

The asymmetric unit contains one organic molecule and one additional water molecule. In the crystal structure of the title compound, one methoxy group is connected to the phenyl moiety (C1—C6). Three cyano groups and one amino group are linked to the ring which is constructed by C8—C14 (cf. the figure). One methyl group is connected to N1 atom from the six-membered ring of the quinoline system (C9, C10, C17, C18, N1 and C19). All bond lengths and angles fall in their normal ranges compared with those in previously reported compounds [5].

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