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The crystal structure of 6-amino-2-methyl-8-(4-(methylthio)phenyl)-2,3,8,8a-tetrahydroisoquinoline-5,7,7(1*H*)-tricarbonitrile – ethanol (1/1), $C_{20}H_{19}N_5S$

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	$0.32 \times 0.26 \times 0.21$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.17 mm^{-1}
Diffractometer, scan mode:	APEX2 CCD detector, φ and ω
θ_{max} , completeness:	25.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10647, 3772, 0.023
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2904
$N(\text{param})_{\text{refined}}$:	266
Programs:	Bruker [1], Olex2 [2], SHELX [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.89482(6)	1.10345(6)	0.22381(5)	0.0652(3)
N1	0.57570(17)	0.50479(16)	0.27338(12)	0.0492(5)
N2	0.5577(2)	0.1983(2)	−0.02611(17)	0.0821(8)
N3	0.76031(18)	0.40614(19)	−0.08169(12)	0.0603(6)
H3A	0.734137	0.339881	−0.103500	0.072*
H3B	0.810033	0.445319	−0.105089	0.072*
N4	0.7876(3)	0.7167(2)	−0.10089(15)	0.0916(9)
N5	0.97092(18)	0.5070(2)	0.09582(14)	0.0656(6)
C1	0.5334(2)	0.5594(2)	0.34675(16)	0.0630(7)
H1A	0.590729	0.606709	0.376800	0.094*
H1B	0.471288	0.610207	0.328924	0.094*
H1C	0.510326	0.497012	0.382715	0.094*
C2	0.4906(2)	0.4326(2)	0.22672(17)	0.0609(7)
H2A	0.423058	0.479650	0.216974	0.073*
H2B	0.474891	0.362532	0.259741	0.073*
C3	0.5251(2)	0.3924(2)	0.14502(16)	0.0534(6)
H3	0.484219	0.331630	0.116215	0.064*
C4	0.61121(18)	0.43824(18)	0.11060(13)	0.0422(5)
C5	0.67724(17)	0.53988(17)	0.15213(13)	0.0390(5)
H5	0.747030	0.507255	0.179306	0.047*
C6	0.61451(19)	0.59728(18)	0.21851(14)	0.0454(5)
H6A	0.662518	0.653421	0.251425	0.054*
H6B	0.551644	0.641968	0.191915	0.054*
C7	0.64592(18)	0.39480(19)	0.03105(14)	0.0431(5)
C8	0.59795(19)	0.2859(2)	−0.00151(16)	0.0537(6)
C9	0.72355(18)	0.44844(19)	−0.01111(13)	0.0433(5)
C10	0.77639(17)	0.56668(18)	0.02273(12)	0.0406(5)
C11	0.7878(2)	0.6509(2)	−0.04701(15)	0.0543(6)
C12	0.8876(2)	0.5362(2)	0.06428(14)	0.0455(5)
C13	0.70411(17)	0.63056(18)	0.08542(12)	0.0382(5)
H13	0.633461	0.651058	0.053214	0.046*
C14	0.75411(17)	0.74777(18)	0.11844(12)	0.0374(5)

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Abstract

$C_{20}H_{19}N_5S \cdot C_2H_6O$, monoclinic, $P2_1/c$ (no. 14), $a = 12.1257(14)$ Å, $b = 11.0862(14)$ Å, $c = 16.0280(19)$ Å, $\beta = 95.948(2)^\circ$, $V = 2143.0(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0486$, $wR_{\text{ref}}(F^2) = 0.1487$, $T = 296(2)$ 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.

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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}
C15	0.71569(17)	0.85529(19)	0.08292(13)	0.0426(5)
H15	0.660657	0.853561	0.037984	0.051*
C16	0.75650(18)	0.96485(19)	0.11212(14)	0.0456(5)
H16	0.729492	1.035819	0.086666	0.055*
C17	0.83741(17)	0.96949(18)	0.17908(13)	0.0419(5)
C18	0.87637(18)	0.8623(2)	0.21550(13)	0.0449(5)
H18	0.930894	0.864115	0.260780	0.054*
C19	0.83568(18)	0.75372(18)	0.18571(13)	0.0422(5)
H19	0.863133	0.682793	0.210951	0.051*
C20	0.8239(3)	1.2197(2)	0.1632(2)	0.0882(10)
H20A	0.850519	1.296898	0.183637	0.132*
H20B	0.837271	1.211054	0.105517	0.132*
H20C	0.745817	1.213823	0.167825	0.132*
O1	0.7477(2)	0.3411(2)	0.34547(17)	0.0901(7)
C22	0.8390(5)	0.4212(6)	0.3735(4)	0.161(2)
H22A	0.874582	0.446942	0.325001	0.193*
H22B	0.809185	0.492522	0.398122	0.193*
C23	0.9188(5)	0.3705(7)	0.4316(4)	0.187(3)
H23A	0.975949	0.428742	0.446936	0.280*
H23B	0.885014	0.346760	0.480661	0.280*
H23C	0.950508	0.301113	0.407431	0.280*
H1	0.689(3)	0.385(4)	0.321(2)	0.115(13)*

Source of material

The title compound was synthesized according to the earlier reported procedure. A mixture of *N*-methyl-4-piperidone (10 mmol), 4-methylsulfanyl-benzaldehyde (10 mmol) and malononitrile (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. The precipitates were filtered off, washed with ice-cooled water and ethanol, and dried under vacuum.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C–H = 0.93/0.96/0.97 Å and N–H = 0.86 Å with $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ and 1.2 times $U_{\text{eq}}(\text{N})$.

Comment

Tetrahydroisoquinoline are heterocycles, which forms the important component of pharmacophores for a number of

compounds having medicinal significance [4, 5]. Recognizing the considerable importance of the compounds, research focused on the synthesis of new tetrahydroisoquinoline derivatives [6, 7].

From the diagram of the asymmetric unit including the atomic numbering scheme of the title compound (figure), we can see that there is an ethanol solvent molecule incorporated in the asymmetric unit. In the crystal structure of the title compound, the six-membered carbon ring and the adjacent six-membered ring containing one nitrogen atom both adopt a flattened chair conformation. The bond lengths and angles are all in their normal scopes [6] and they are similar with those previously reported compound 6-amino-8-(3,4-dimethylphenyl)-2-methyl-2,3,8,8a-tetrahydro-1*H*-isoquinoline-5,7,7-tricarbonitrile [7].

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