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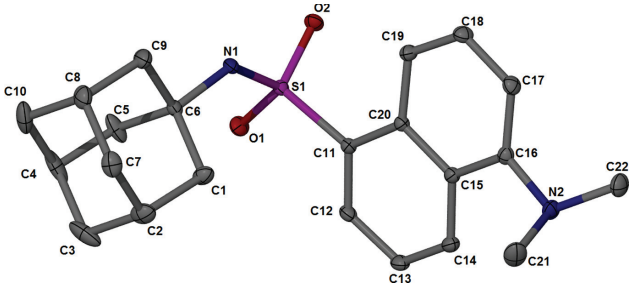
Crystal structure of *N*-(adamantan-1-yl)-5-(dimethylamino)naphthalene-1-sulfonamide, $C_{22}H_{28}N_2O_2S$



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Abstract

$C_{22}H_{28}N_2O_2S$, monoclinic, $P2_1/n$ (no. 14), $a = 9.2516(7)$ Å, $b = 10.5122(8)$ Å, $c = 19.7782(15)$ Å, $\beta = 98.9530(10)°$, $V = 1900.1(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0356$, $wR_{ref}(F^2) = 0.1004$, $T = 100(2)$ K.

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The molecular structure is shown in the figure (H atoms are omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless shard
Size:	0.40 × 0.20 × 0.06 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.19 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II DUO, φ and ω
θ_{max}, completeness:	30.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	60522, 5782, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5118
$N(param)_{refined}$:	250
Programs:	Bruker [1], SHELX [2], X-Seed [3, 4], WinGX/ORTEP [5]

Source of materials

A solution of dansyl chloride (systematic name: 5-(dimethylamino)naphthalene-1-sulfonyl chloride) (1.0 eq.) and amantadine (systematic name: 1-aminodiamantane; 1.0 eq.) in dichloromethane (DCM) (10 mL) was stirred in a sealed tude under microwave irradiation conditions (150 W, 373 K, 150 psi) for 10 min. The solvent was evaporated under reduced pressure and the residue was purified *via* repeated crystallization from ethanol at room temperature to afford the product. Yield 95%; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ/ppm = 8.53–8.51 (dd, $J = 6.3$, 2.2 Hz, 1H), 8.32–8.29 (dd, $J = 6.3$, 2.1 Hz, 1H), 8.25–8.23 (dd, $J = 6.3$, 2.1 Hz, 1H), 7.58–7.50 (m, 2H), 7.19–7.17 (dd, $J = 6.4$, 2.1 Hz, 1H), 2.90 (s, 6H), 1.95 (s, 3H), 1.74 (s, 6H), 1.59–1.48 (m, 6H); **^{13}C-NMR** (100 MHz, $CDCl_3$) δ/ppm = 151.6, 138.7, 130.0, 129.9, 129.6, 129.0, 128.0, 123.4, 119.2, 115.0, 55.5, 45.5, 43.0, 35.8, 29.5. The title compound (10.0 mg) was dissolved in ethanol, and the solvent was evaporated slowly at ambient conditions. Crystals formed over a period of 3 days.

Experimental details

Single-crystal X-ray intensity data were collected on a Bruker 3-circle Apex II DUO X-ray diffractometer equipped with an INCOATEC IμS HB microsource. Data collection and reduction were carried out using the Bruker software package APEX3 [1] using standard procedures. The structure was solved and refined using SHELX-2016 [2] employed within the X-Seed [3, 4] environment. Hydrogen atoms were placed in calculated positions using riding models. ORTEP-3 [5] was used to generate the publication material.

Comment

The title compound was investigated as part of an ongoing study into the development of adamantane conjugated fluorophores that could be used as potential neurobiological fluorescent ligands [6, 7]. The 5-dimethylamino-naphtalene sulfonyl (dansyl) moeity acts as the fluorophore [7, 8]. Continued development of fluorescent adamantane molecules is

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.14508(12)	0.61431(11)	0.60688(6)	0.0162(2)
H1A	0.111191	0.632305	0.557847	0.019*
H1B	0.189105	0.692946	0.628658	0.019*
C2	0.01439(13)	0.57337(12)	0.64151(7)	0.0205(2)
H2	−0.060563	0.642667	0.636652	0.025*
C3	0.06800(14)	0.54803(15)	0.71770(7)	0.0286(3)
H3A	0.112141	0.626258	0.739896	0.034*
H3B	−0.015560	0.523213	0.740613	0.034*
C4	0.18177(14)	0.44104(16)	0.72476(6)	0.0274(3)
H4	0.216718	0.424695	0.774418	0.033*
C5	0.31256(13)	0.48128(14)	0.69008(6)	0.0212(2)
H5A	0.358414	0.558699	0.712484	0.025*
H5B	0.386789	0.412701	0.694924	0.025*
C6	0.25962(11)	0.50790(10)	0.61396(5)	0.01121(18)
C7	−0.05386(13)	0.45170(12)	0.60823(6)	0.0189(2)
H7A	−0.138054	0.426079	0.630508	0.023*
H7B	−0.090066	0.467374	0.559163	0.023*
C8	0.05972(13)	0.34525(11)	0.61546(6)	0.0188(2)
H8	0.014643	0.266312	0.593145	0.023*
C9	0.19056(13)	0.38521(11)	0.58088(6)	0.0167(2)
H9A	0.157359	0.399610	0.531432	0.020*
H9B	0.264473	0.316370	0.585765	0.020*
C10	0.11244(14)	0.31939(14)	0.69147(7)	0.0266(3)
H10A	0.185234	0.249644	0.696665	0.032*
H10B	0.028897	0.293318	0.714087	0.032*
C11	0.42199(11)	0.79573(10)	0.55727(5)	0.01079(18)
C12	0.40813(12)	0.89081(10)	0.60349(5)	0.01268(19)
H12	0.441036	0.877155	0.650868	0.015*
C13	0.34528(12)	1.00857(10)	0.58094(6)	0.0145(2)
H13	0.338342	1.074640	0.613051	0.017*
C14	0.29411(12)	1.02813(10)	0.51283(6)	0.0138(2)
H14	0.250869	1.107455	0.498261	0.017*
C15	0.30502(11)	0.93113(10)	0.46381(5)	0.01112(18)
C16	0.25293(11)	0.95183(10)	0.39230(5)	0.01220(19)
C17	0.28194(12)	0.86282(11)	0.34488(5)	0.0138(2)
H17	0.252056	0.878057	0.297414	0.017*
C18	0.35586(12)	0.74932(11)	0.36682(6)	0.0143(2)
H18	0.377207	0.689726	0.333641	0.017*
C19	0.39766(12)	0.72296(10)	0.43492(5)	0.01245(19)
H19	0.442377	0.643691	0.448414	0.015*
C20	0.37448(11)	0.81346(10)	0.48535(5)	0.01025(18)
C21	0.02252(14)	1.05688(13)	0.38842(7)	0.0240(3)
H21A	−0.032854	0.995380	0.357334	0.036*
H21B	−0.024275	1.140561	0.381979	0.036*
H21C	0.024324	1.029099	0.435846	0.036*
C22	0.17367(14)	1.10701(12)	0.30315(6)	0.0187(2)
H22A	0.274874	1.113415	0.294488	0.028*
H22B	0.126240	1.190364	0.296221	0.028*
H22C	0.120377	1.045240	0.271502	0.028*
N1	0.38407(10)	0.53698(9)	0.57701(5)	0.01117(16)
H1	0.3742(18)	0.5081(17)	0.5354(9)	0.024(4)*
N2	0.17296(10)	1.06525(9)	0.37360(5)	0.01513(18)
O1	0.54260(9)	0.66769(8)	0.66343(4)	0.01504(16)
O2	0.61620(8)	0.61719(8)	0.55114(4)	0.01376(15)
S1	0.50335(3)	0.65063(2)	0.59084(2)	0.01044(7)

also motivated by their good biological activity, e.g. as nitric oxide synthase inhibitors [6, 9], anti-oxidants [9], *N*-methyl-*D*-aspartate receptor antagonists and voltage-gated calcium channel blockers [7, 9, 10].

The bond lengths, angles and torsion angles of the adamantane ring system and the conjugated 5-dimethylamino-naphtalene sulfonyl moiety are consistent with previously published structures [11–14]. The conformation of the investigated molecule is dependant on the sulfonylamide link, connecting the naphtalene ring and adamantane fragment. Two important torsion angles t1 [C6—N1—S1—C11, −77.19(10)°] and t2 [C12—C11—S1—N1, 115.88(9)°] describe the conformation of the molecule. Crystal packing analysis reveals that the molecules associate into cyclic dimers. These dimers are connected *via* a classical intermolecular hydrogen bond, N1—H1···O2i with an H···A distance of 2.38 Å, and an angle of 119.8°. Symmetry code: (i) $-x+1, -y+1, -z+1$. The dimers are uniformly packed in the (*ac*) plane and create alternate stacking in the proximity of the (*ac*) plane.

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