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Crystal structure of *N*-((3*R*,10*S*,13*S*,17*S*)-17-((*S*)-1-(dimethylamino)ethyl)-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)-*N*-methylbenzamide, C₃₁H₄₈N₂O

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Abstract

C₃₁H₄₈N₂O, orthorhombic, *P*₂₁₂₁₂₁ (no. 19), *a* = 6.530(2) Å, *b* = 11.728(4) Å, *c* = 35.236(12) Å, *V* = 2698.6(15) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0492, *wR*_{ref}(*F*²) = 0.1034, *T* = 173 K.

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The asymmetric unit of the title 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.

Source of material

The stems and roots of *Sarcococca hookeriana* (14.5 kg) collected from Hezhang country, Guizhou province, were extracted from methanol solution with ultrasonic radiation three times. The extract was concentrated and then

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.19 × 0.15 × 0.12 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.07 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	26.3°, 98%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	10811, 5075, 0.075
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3693
<i>N</i> (<i>param</i>) _{refined} :	314
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

partitioned between EtOAc and 1% aq. H₂SO₄. The acid-soluble fraction was alkalized with aq. Na₂CO₃ to pH 9 and followed by exhaustive extraction with CH₂Cl₂ to afford crude alkaloids. The crude alkaloids were roughly divided into five fractions: *Frs. A–E*. Subsequently, fraction A was separated by silica gel column chromatography using petroleum ether/ethylacetate/diethylamine (50:1:1) as the eluent. The raw product was re-crystallized from dichloromethane to get the title compound as block colorless crystals.

Experimental details

The crystal structure was solved by direct methods and refined by full-matrix least-squares methods on *F*² using Olex2 [2]. All hydrogen atoms were positioned geometrically, with the *d*(C–H) = 0.95–1.00 Å. The *U*_{iso}(H) were set to 1.2 times *U*_{eq}(C) for the methylene groups as well as methenyl groups, at 1.5 times *U*_{eq}(C) for the methyl groups.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.7321(3)	0.3748(3)	0.41142(6)	0.0603(8)
N1	0.3179(4)	0.3464(2)	0.06283(7)	0.0331(6)
N2	0.3911(3)	0.4076(2)	0.41407(6)	0.0293(6)
C1	0.1818(5)	0.3185(2)	0.31635(7)	0.0277(7)
H1A	0.0578	0.2766	0.3092	0.033 [*]
H1B	0.2999	0.2735	0.3080	0.033 [*]
C2	0.1887(5)	0.3292(2)	0.35985(8)	0.0323(7)
H2A	0.0640	0.3670	0.3688	0.039 [*]
H2B	0.1940	0.2530	0.3712	0.039 [*]
C3	0.3746(5)	0.3972(2)	0.37219(7)	0.0315(7)
H3	0.4973	0.3555	0.3633	0.038 [*]
C4	0.3766(5)	0.5137(3)	0.35313(8)	0.0336(7)
H4A	0.5008	0.5551	0.3605	0.040 [*]
H4B	0.2580	0.5581	0.3616	0.040 [*]
C5	0.3704(4)	0.5012(2)	0.30987(8)	0.0290(7)
H5	0.4916	0.4547	0.3031	0.035 [*]
C6	0.3986(6)	0.6137(3)	0.28948(8)	0.0427(9)
H6A	0.5181	0.6532	0.3000	0.051 [*]
H6B	0.2781	0.6618	0.2938	0.051 [*]
C7	0.4283(5)	0.5966(3)	0.24718(8)	0.0390(8)
H7A	0.5604	0.5591	0.2428	0.047 [*]
H7B	0.4325	0.6713	0.2347	0.047 [*]
C8	0.2589(4)	0.5250(2)	0.22924(8)	0.0252(6)
H8	0.1319	0.5711	0.2284	0.030 [*]
C9	0.2156(4)	0.4143(2)	0.25192(7)	0.0220(6)
H9	0.3417	0.3679	0.2497	0.026 [*]
C10	0.1834(4)	0.4336(2)	0.29524(7)	0.0218(6)
C11	0.0479(5)	0.3452(2)	0.23215(7)	0.0294(7)
H11A	−0.0799	0.3889	0.2325	0.035 [*]
H11B	0.0249	0.2744	0.2463	0.035 [*]
C12	0.1021(5)	0.3157(2)	0.19081(7)	0.0287(7)
H12A	0.2202	0.2640	0.1906	0.034 [*]
H12B	−0.0135	0.2754	0.1792	0.034 [*]
C13	0.1527(4)	0.4212(2)	0.16696(7)	0.0225(6)
C14	0.3158(4)	0.4903(2)	0.18871(8)	0.0261(7)
H14	0.4341	0.4382	0.1914	0.031 [*]
C15	0.3843(5)	0.5808(2)	0.16023(8)	0.0373(8)
H15A	0.5228	0.6080	0.1660	0.045 [*]
H15B	0.2901	0.6459	0.1600	0.045 [*]
C16	0.3788(5)	0.5158(3)	0.12179(8)	0.0373(8)
H16A	0.3002	0.5590	0.1030	0.045 [*]
H16B	0.5179	0.5046	0.1121	0.045 [*]
C17	0.2753(4)	0.3994(2)	0.12963(7)	0.0247(6)
H17	0.3862	0.3451	0.1360	0.030 [*]
C18	−0.0415(5)	0.4900(2)	0.15915(8)	0.0361(8)
H18A	−0.0081	0.5554	0.1435	0.054 [*]
H18B	−0.0996	0.5158	0.1830	0.054 [*]
H18C	−0.1401	0.4424	0.1460	0.054 [*]
C19	−0.0206(4)	0.4964(2)	0.30276(8)	0.0342(7)
H19A	−0.0272	0.5644	0.2872	0.051 [*]
H19B	−0.0284	0.5176	0.3293	0.051 [*]
H19C	−0.1342	0.4465	0.2965	0.051 [*]
C20	0.1669(5)	0.3521(2)	0.09436(8)	0.0303(7)
H20	0.0617	0.4086	0.0869	0.036 [*]
C21	0.0556(6)	0.2390(3)	0.10125(9)	0.0479(9)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H21A	0.0118	0.2072	0.0772	0.072 [*]
H21B	−0.0630	0.2521	0.1172	0.072 [*]
H21C	0.1476	0.1859	0.1137	0.072 [*]
C22	0.4702(5)	0.2566(3)	0.06679(9)	0.0516(10)
H22A	0.5369	0.2633	0.0913	0.077 [*]
H22B	0.5715	0.2638	0.0468	0.077 [*]
H22C	0.4038	0.1828	0.0649	0.077 [*]
C23	0.2192(6)	0.3397(3)	0.02586(9)	0.0505(10)
H23A	0.1518	0.2664	0.0233	0.076 [*]
H23B	0.3214	0.3480	0.0061	0.076 [*]
H23C	0.1187	0.4002	0.0236	0.076 [*]
C24	0.2143(4)	0.4536(3)	0.43436(9)	0.0370(8)
H24A	0.1523	0.5139	0.4194	0.055 [*]
H24B	0.2582	0.4840	0.4586	0.055 [*]
H24C	0.1149	0.3934	0.4385	0.055 [*]
C25	0.5765(5)	0.3940(3)	0.43033(8)	0.0337(7)
C26	0.5956(4)	0.3995(2)	0.47302(8)	0.0278(7)
C27	0.4740(5)	0.3348(3)	0.49721(8)	0.0321(7)
H27	0.3677	0.2898	0.4872	0.039 [*]
C28	0.5087(5)	0.3361(3)	0.53606(8)	0.0353(8)
H28	0.4247	0.2932	0.5523	0.042 [*]
C29	0.6668(5)	0.4006(3)	0.55080(8)	0.0350(8)
H29	0.6911	0.4006	0.5771	0.042 [*]
C30	0.7895(5)	0.4652(3)	0.52716(8)	0.0345(8)
H30	0.8961	0.5096	0.5373	0.041 [*]
C31	0.7544(4)	0.4643(3)	0.48833(8)	0.0309(7)
H31	0.8385	0.5077	0.4722	0.037 [*]

Comment

The *S.hookeriana* possess a variety of bioactivity. A recently reported experimental study shows that the main chemical compositions of *S.hookeriana* are pregnane-type steroidal alkaloids [5]. These compounds have shown cholinesterase inhibition [6], and they poved antileishmanial [7], antibacterial [8] and estrogen biosynthesis-promoting [9] activities. As a folk prescription in miao area of guizhou province, *S.hookeriana* is used to treat Alzheimer's diseases and other diseases.

The title compound crystallizes with one molecule in the asymmetric unit of the space group *P*2₁2₁2₁ (the figure). Geometric parameters of the title structure are in the usual ranges [10]. The compound contains one amide group, one five-membered ring, three six-membered rings, one phenyl moiety and one dimethylamino group. The dimethylamino group was confirmed by the distance of 1.456(4) Å, 1.456(4) Å (C22—N1 and C23—N1), respectively. The amide group was confirmed by the distance of 1.235(4) Å, 1.461(4) Å, 1.349(4) Å (C25—O1, C24—N2 and C25—N2). The five-membered ring and three six-membered rings show intermolecular interactions by van der Waals forces only.

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