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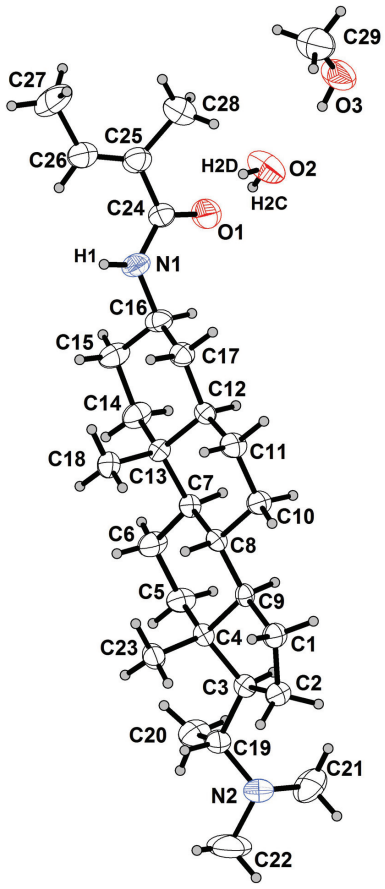
Crystal structure of (*E*)-*N*-((3*R*,5*S*,10*S*, 13*S*,14*S*,17*S*)-17-((*S*)-1-(dimethylamino)ethyl)-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)-2-methylbut-2-enamide – water – methanol (1/1/1), $C_{29}H_{54}N_2O_3$



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Abstract

$C_{29}H_{54}N_2O_3$, monoclinic, $P2_1$ (no. 4), $a = 9.8091(11)$ Å, $b = 7.6661(8)$ Å, $c = 19.095(2)$ Å, $\beta = 93.171(4)°$, $V = 1433.7(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0657$, $wR_{ref}(F^2) = 0.1362$, $T = 250$ K.

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The molecular structure is shown in the figure. 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 block
Size:	0.19 × 0.08 × 0.05 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.07 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.9°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14394, 6775, 0.058
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3520
$N(param)_{refined}$:	320
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

The stems and roots of *Sarcococcahookeriana* (14.5 kg) collected from Hezhang country, Guizhou province, were extracted by methanol with ultrasonic three times. The extract was concentrated and then partitioned between EtOAc and 1% aq. H_2SO_4. The acid-soluble fraction was alkalinized with aq. Na_2CO_3 to pH 9 and followed by exhaustive extraction with CH_2Cl_2 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 and then recrystallized with dichloromethane to get the title compound as colorless crystals.

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	0.7378(3)	0.5316(5)	0.85083(14)	0.0681(9)
N1	0.5101(3)	0.5420(5)	0.84832(14)	0.0471(9)
H1	0.437562	0.543957	0.872548	0.056*
N2	0.0938(4)	0.5137(5)	0.21028(17)	0.0526(9)
C1	0.2116(4)	0.2185(5)	0.40154(19)	0.0382(10)
H1A	0.131620	0.165700	0.421626	0.046*
H1B	0.283751	0.130434	0.399698	0.046*
C2	0.1744(4)	0.2938(5)	0.3274(2)	0.0403(11)
H2A	0.235172	0.246006	0.293170	0.048*
H2B	0.080061	0.263994	0.312543	0.048*
C19	0.0978(4)	0.5952(5)	0.28057(19)	0.0405(11)
H19	0.004336	0.587170	0.297370	0.049*
C3	0.1914(4)	0.4925(5)	0.33275(18)	0.0305(9)
H3	0.286723	0.520011	0.322282	0.037*
C4	0.1766(3)	0.5295(6)	0.41217(17)	0.0278(8)
C5	0.2392(4)	0.6967(5)	0.44215(19)	0.0408(11)
H5A	0.184545	0.796424	0.424905	0.049*
H5B	0.331472	0.710182	0.425728	0.049*
C6	0.2461(5)	0.6967(5)	0.5222(2)	0.0482(13)
H6A	0.152863	0.702258	0.538039	0.058*
H6B	0.293884	0.802371	0.538821	0.058*
C7	0.3179(3)	0.5380(5)	0.55608(16)	0.0285(8)
H7	0.414649	0.545785	0.544183	0.034*
C8	0.2629(4)	0.3661(5)	0.52405(19)	0.0271(9)
H8	0.167861	0.350396	0.537834	0.032*
C9	0.2611(4)	0.3761(5)	0.44422(18)	0.0268(8)
H9	0.356651	0.396786	0.432204	0.032*
C10	0.3475(4)	0.2129(5)	0.55287(18)	0.0381(10)
H10A	0.304575	0.103884	0.536417	0.046*
H10B	0.438613	0.218472	0.534343	0.046*
C11	0.3619(4)	0.2107(5)	0.63262(19)	0.0407(11)
H11A	0.273393	0.181629	0.650978	0.049*
H11B	0.426767	0.119104	0.647723	0.049*
C12	0.4105(4)	0.3827(5)	0.66347(18)	0.0303(9)
H12	0.501525	0.404408	0.645320	0.036*
C13	0.3184(3)	0.5365(5)	0.63746(16)	0.0294(8)
C14	0.3831(5)	0.7052(6)	0.6678(2)	0.0456(11)
H14A	0.470448	0.724494	0.646471	0.055*
H14B	0.323207	0.803623	0.654666	0.055*
C15	0.4077(5)	0.7033(6)	0.7476(2)	0.0500(12)
H15A	0.454270	0.811084	0.762765	0.060*
H15B	0.319747	0.699130	0.769513	0.060*
C16	0.4933(4)	0.5482(6)	0.77190(18)	0.0411(10)
H16	0.584950	0.561315	0.753231	0.049*
C17	0.4311(4)	0.3776(6)	0.74303(19)	0.0385(10)
H17A	0.491491	0.280133	0.756730	0.046*
H17B	0.342987	0.357747	0.763530	0.046*
C18	0.1736(4)	0.5190(6)	0.66259(19)	0.0466(11)
H18A	0.131507	0.414208	0.642848	0.070*
H18B	0.120116	0.619948	0.647484	0.070*
H18C	0.177446	0.511760	0.713381	0.070*
C20	0.1347(5)	0.7895(6)	0.2792(2)	0.0554(13)
H20A	0.229513	0.802484	0.268184	0.083*
H20B	0.121101	0.840699	0.324720	0.083*
H20C	0.076738	0.848210	0.243743	0.083*
C23	0.0261(3)	0.5178(6)	0.42891(19)	0.0406(10)
H23A	−0.016645	0.421416	0.403169	0.061*
H23B	−0.019813	0.625728	0.415349	0.061*
H23C	0.019308	0.498998	0.478832	0.061*
C22	−0.0156(6)	0.5910(8)	0.1634(3)	0.097(2)
H22A	−0.102612	0.579310	0.184732	0.145*
H22B	−0.019566	0.530640	0.118642	0.145*
H22C	0.003690	0.713505	0.156036	0.145*
C21	0.2242(5)	0.5226(8)	0.1760(2)	0.0726(15)
H21A	0.218052	0.453577	0.133323	0.109*
H21B	0.296733	0.477228	0.207410	0.109*
H21C	0.243802	0.642971	0.164475	0.109*
C24	0.6322(4)	0.5336(6)	0.88238(19)	0.0415(10)
C25	0.6400(4)	0.5170(6)	0.9607(2)	0.0492(11)
C26	0.5445(5)	0.5769(7)	0.9983(2)	0.0576(13)
H26	0.468001	0.627580	0.974387	0.069*
C27	0.5460(6)	0.5719(8)	1.0781(2)	0.0866(18)
H27A	0.538767	0.451908	1.093607	0.130*
H27B	0.469506	0.638541	1.093902	0.130*
H27C	0.630699	0.621876	1.097493	0.130*
C28	0.7707(5)	0.4354(8)	0.9917(3)	0.0827(19)
H28A	0.788454	0.327649	0.967142	0.124*
H28B	0.761027	0.410795	1.040991	0.124*
H28C	0.846106	0.515449	0.986735	0.124*
O2	0.9965(3)	0.6660(5)	0.8360(2)	0.0778(12)
H2C	0.977259	0.758986	0.812309	0.117*
H2D	0.919488	0.631922	0.850582	0.117*
O3	1.2276(3)	0.5247(7)	0.89842(17)	0.0837(11)
H3A	1.161022	0.556122	0.872776	0.126*
C29	1.1818(6)	0.4456(8)	0.9585(3)	0.0805(18)
H29A	1.109155	0.364139	0.945447	0.121*
H29B	1.147718	0.534076	0.989386	0.121*
H29C	1.256792	0.383512	0.982606	0.121*

Experimental details

The crystal structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 using Olex2 [3]. 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.

Comment

The *S. hookeriana* possesses a variety of bioactivities. Experimental study shows that the main chemical compositions of *S. hookeriana* are pregnane-type steroidal alkaloids [4]. These compounds have cholinesterase inhibition [5], antileishmanial [6], antibacterial [7] and estrogen biosynthesis-promoting [8] activities. Besides, as a folk prescription in miao area of Guizhou province, *S. hookeriana* is used to treat Alzheimer's diseases and other diseases.

The space group in which the title compound crystallizes is monoclinic $P2_1$. 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, and one dimethylamino group. The dimethylamino group was confirmed by the distance of 1.470(5) Å, 1.483(5) Å (*C21—N2* and *C22—N2*), respectively. Then the amide group was confirmed by the distance of 1.226(4) Å, 1.460(4) Å, 1.332(4) Å (*C24—O1*, *C16—N1* and *C24—N1*).

In this crystal exist additionally one water and one MeOH molecule. Actually, because of the water and MeOH it makes this crystal existent. There is intermolecular hydrogen bond between the O1 atom and the oxygen of water (O2). There is a hydrogen bond between the O1 and H2D. And also O2 forms the hydrogen bond with the hydrogen of MeOH (H3A). Finally, the O3 is involved in a hydrogen bond.

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