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Crystal structure of (2*OS*)-20-(dimethylamino)-3-(tigloylamino)-5 α -pregn-2,16-dien-4-one, C₂₈H₄₂N₂O₂

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Abstract

C₂₈H₄₂N₂O₂, monoclinic, *P*₂₁ (no. 4), *a* = 12.679(3) Å, *b* = 6.9067(14) Å, *c* = 15.577(3) Å, β = 113.821(4)°, *V* = 1247.8(5) Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0485, *wR*_{ref}(*F*²) = 0.1266, *T* = 173 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.

Source of material

The stems and roots of *Sarcococca hookeriana* (14.5 kg) were extracted three times using MeOH with ultrasonic. The extract was concentrated and then partitioned between EtOAc and 1% aq·H₂SO₄. The acid-soluble fraction was alkalinized with aq·Na₂CO₃ to pH 9 and followed by exhaustive extraction with CH₂Cl₂ to afford crude alkaloids. The crude alkaloids were roughly separated by CC (SiO₂;

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.28 × 0.15 × 0.12 mm
Wavelength:	Mo <i>K</i> α radiation (0.71073 Å)
μ:	0.07 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	26.4°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	8510, 4672, 0.041
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 3863
<i>N</i> (<i>param</i>) _{refined} :	297
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

CH₂Cl₂/MeOH/Et₂NH, 100:0:0–10:1:0–5:1:1) to give five fractions: *Frs. A–E* [5]. The title compound Sarcovagenine C was obtained from *Fr. A* by silica gel column chromatography using dichloromethane/methanol (50:1) as the eluent and recrystallization with ethyl acetate [6].

Experimental details

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.

Discussion

The plant *Sarcococca hookeriana* is widely distributed in Yunnan and Guizhou provinces, and other areas in China. The whole plants of *S. hookeriana* are used as TCM and traditional folk medicine to treat stomach pain, rheumatism and swollen

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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	0.96818(19)	0.8848(3)	0.57882(16)	0.0313(6)
O2	1.15370(19)	0.3551(4)	0.78893(16)	0.0331(6)
N1	1.0893(2)	0.6239(4)	0.69971(18)	0.0259(6)
H1	1.1079	0.7451	0.6951	0.031*
N2	0.1678(2)	0.2293(5)	0.18656(19)	0.0324(7)
C1	0.8296(3)	0.3349(4)	0.5317(2)	0.0225(7)
H1A	0.7868	0.2419	0.5541	0.027*
H1B	0.8455	0.2704	0.4814	0.027*
C2	0.9430(3)	0.3816(5)	0.6120(2)	0.0259(7)
H2	0.9836	0.2809	0.6538	0.031*
C3	0.9880(2)	0.5585(5)	0.6261(2)	0.0228(7)
C4	0.9309(3)	0.7186(5)	0.5607(2)	0.0225(7)
C5	0.8299(2)	0.6672(4)	0.4715(2)	0.0202(6)
H5	0.8626	0.6050	0.4296	0.024*
C6	0.7653(3)	0.8457(5)	0.4200(2)	0.0251(7)
H6A	0.8192	0.9335	0.4076	0.030*
H6B	0.7338	0.9157	0.4599	0.030*
C7	0.6676(3)	0.7903(5)	0.3280(2)	0.0267(7)
H7A	0.7000	0.7328	0.2856	0.032*
H7B	0.6241	0.9080	0.2974	0.032*
C8	0.5858(3)	0.6456(5)	0.3431(2)	0.0208(7)
H8	0.5503	0.7078	0.3830	0.025*
C9	0.6522(2)	0.4626(5)	0.3940(2)	0.0198(6)
H9	0.6894	0.4089	0.3535	0.024*
C10	0.7521(2)	0.5122(4)	0.4896(2)	0.0193(6)
C11	0.5706(3)	0.3029(5)	0.4005(2)	0.0231(7)
H11A	0.5415	0.3418	0.4481	0.028*
H11B	0.6156	0.1823	0.4228	0.028*
C12	0.4665(2)	0.2595(5)	0.3076(2)	0.0222(7)
H12A	0.4935	0.1976	0.2628	0.027*
H12B	0.4135	0.1685	0.3194	0.027*
C13	0.4024(2)	0.4472(5)	0.2652(2)	0.0207(6)
C14	0.4902(2)	0.5837(5)	0.2509(2)	0.0210(7)
H14	0.5284	0.5054	0.2176	0.025*
C15	0.4141(3)	0.7308(5)	0.1790(2)	0.0289(7)
H15A	0.3889	0.8365	0.2093	0.035*
H15B	0.4537	0.7865	0.1415	0.035*
C16	0.3147(3)	0.6028(5)	0.1204(2)	0.0291(8)
H16	0.2613	0.6349	0.0588	0.035*
C17	0.3099(2)	0.4392(5)	0.1645(2)	0.0241(7)
C18	0.3477(3)	0.5364(5)	0.3281(2)	0.0257(7)
H18A	0.3161	0.6643	0.3039	0.039*
H18B	0.4064	0.5496	0.3923	0.039*
H18C	0.2855	0.4521	0.3283	0.039*
C19	0.7056(3)	0.5866(5)	0.5603(2)	0.0241(7)
H19A	0.6623	0.4832	0.5747	0.036*
H19B	0.6546	0.6973	0.5333	0.036*
H19C	0.7701	0.6269	0.6180	0.036*
C20	0.2293(3)	0.2707(5)	0.1257(2)	0.0273(7)
H20	0.2792	0.1555	0.1304	0.033*
C21	0.1513(3)	0.2941(7)	0.0214(2)	0.0425(10)
H21A	0.1035	0.1781	−0.0010	0.064*
H21B	0.1989	0.3117	−0.0143	0.064*
H21C	0.1016	0.4074	0.0128	0.064*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C22	0.0776(3)	0.3681(7)	0.1758(3)	0.0489(11)
H22A	0.1056	0.4992	0.1731	0.073*
H22B	0.0560	0.3580	0.2294	0.073*
H22C	0.0102	0.3408	0.1177	0.073*
C23	0.1238(3)	0.0323(6)	0.1747(3)	0.0473(10)
H23A	0.0672	0.0142	0.1101	0.071*
H23B	0.0869	0.0088	0.2182	0.071*
H23C	0.1876	−0.0590	0.1879	0.071*
C24	1.1631(3)	0.5278(5)	0.7771(2)	0.0250(7)
C25	1.2581(3)	0.6504(5)	0.8454(2)	0.0254(7)
C26	1.2441(3)	0.8378(5)	0.8567(2)	0.0330(8)
H26	1.1717	0.8935	0.8188	0.040*
C27	1.3665(3)	0.5415(6)	0.9021(2)	0.0384(9)
H27A	1.3728	0.5259	0.9665	0.058*
H27B	1.4334	0.6137	0.9027	0.058*
H27C	1.3642	0.4137	0.8740	0.058*
C28	1.3329(3)	0.9680(6)	0.9242(3)	0.0439(10)
H28A	1.3269	1.0968	0.8962	0.066*
H28B	1.4098	0.9148	0.9387	0.066*
H28C	1.3202	0.9780	0.9821	0.066*

sore throat. In order to explore the active chemical ingredients of *S. hookeriana*, phytochemical investigation led to isolation of Sarcovagenine C.

Geometric parameters of the title structure are in the usual ranges. The compound contains one carbonyl, one amide, two double bonds and one dimethylamino group. The C4–O1 distance is 1.230(4) Å. The amide was confirmed by the distance of 1.220(4) Å, and 1.363(4) Å for C24–O2 and C24–N1, respectively. The C2–C3 and C16–C17 distance is 1.329(5) Å and 1.336(5) Å. The dimethylamino group was confirmed by the distance of 1.454(5) Å, and 1.450(5) Å for C23–N2 and C22–N2, respectively. The compound contains three sixmembered rings and one five-membered ring. The intermolecular connection was achieved by van der Waals interactions only.

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