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Crystal structure of 3 β -methoxy-20 α -dimethylamino-pregn-5-ene, C₂₄H₄₁NO

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Abstract

C₂₄H₄₁NO, orthorhombic, $P2_12_12_1$ (no. 19), $a = 6.3752(16)$ Å, $b = 12.607(3)$ Å, $c = 26.943(7)$ Å, $V = 2165.4(10)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0573$, $wR_{\text{ref}}(F^2) = 0.1232$, $T = 173$ K.

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The crystal structure of the title compound (systematic name: 1-((3S,9S,10R,13S,14S)-3-methoxy-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-17-yl)-N,N-dimethylethan-1-amine) 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 treatment. The extract was concentrated and 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 separated by CC (SiO₂; CH₂Cl₂/MeOH/Et₂NH, 100:0:0–10:1:0–5:1:1) to give five fractions: Frs. A–E. The title compound *Pachyaximine A* was obtained from Fr. A by silica gel column chromatography

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Table 1: Crystal collection and handling.

Crystal:	Block, colorless
Size:	0.28 × 0.15 × 0.12 mm
Wavelength:	Mo K α radiation ($\lambda = 0.71073$ Å)
μ :	0.065 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, Φ and ω -scans
θ_{max} , completeness:	27.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12457, 4735, 0.0721
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3072
$N(\text{param})_{\text{refined}}$:	242
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3, 4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0452(4)	0.77064(18)	0.28539(9)	0.0418(7)
N1	0.7851(4)	−0.0559(2)	0.47607(10)	0.0337(7)
C1	0.1107(5)	0.4805(2)	0.31164(12)	0.0298(8)
H1A	0.0594	0.4181	0.2929	0.036
H1B	0.0447	0.4787	0.3449	0.036
C2	0.0382(5)	0.5811(2)	0.28480(12)	0.0319(8)
H2A	0.0906	0.5805	0.2502	0.038
H2B	−0.1170	0.5831	0.2838	0.038
C3	0.1198(5)	0.6787(3)	0.31136(12)	0.0323(8)
H3	0.0628	0.6796	0.3459	0.039
C4	0.3575(5)	0.6743(2)	0.31374(12)	0.0330(8)
H4A	0.4152	0.6769	0.2796	0.040
H4B	0.4102	0.7371	0.3320	0.040
C5	0.4329(5)	0.5747(2)	0.33921(11)	0.0275(8)
C6	0.5569(6)	0.5800(3)	0.37860(12)	0.0318(8)
H6	0.5959	0.6486	0.3899	0.038
C7	0.6405(6)	0.4869(2)	0.40664(12)	0.0324(8)
H7A	0.5660	0.4813	0.4388	0.039
H7B	0.7911	0.4986	0.4138	0.039
C8	0.6154(5)	0.3824(2)	0.37817(11)	0.0246(7)
H8	0.7237	0.3789	0.3514	0.030
C9	0.3953(5)	0.3769(2)	0.35448(11)	0.0232(7)
H9	0.2928	0.3850	0.3823	0.028
C10	0.3517(5)	0.4702(2)	0.31808(11)	0.0257(7)
C11	0.3554(5)	0.2666(2)	0.33200(12)	0.0280(8)
H11A	0.2081	0.2631	0.3205	0.034
H11B	0.4467	0.2578	0.3026	0.034
C12	0.3956(5)	0.1740(2)	0.36785(12)	0.0277(8)
H12A	0.2924	0.1768	0.3953	0.033
H12B	0.3757	0.1060	0.3500	0.033

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C13	0.6177(5)	0.1780(2)	0.38937(11)	0.0217(7)
C14	0.6440(5)	0.2883(2)	0.41290(11)	0.0237(7)
H14	0.5287	0.2943	0.4379	0.028
C15	0.8459(6)	0.2803(3)	0.44299(12)	0.0346(8)
H15A	0.8460	0.3318	0.4707	0.042
H15B	0.9702	0.2933	0.4218	0.042
C16	0.8438(6)	0.1654(3)	0.46253(13)	0.0358(9)
H16A	0.8206	0.1646	0.4989	0.043
H16B	0.9792	0.1301	0.4554	0.043
C17	0.6628(5)	0.1075(2)	0.43561(11)	0.0246(7)
H17	0.5368	0.1120	0.4576	0.029
C18	0.7811(5)	0.1557(3)	0.34859(12)	0.0309(8)
H18A	0.7730	0.2112	0.3232	0.046
H18B	0.7525	0.0866	0.3334	0.046
H18C	0.9218	0.1553	0.3632	0.046
C19	0.4556(5)	0.4500(3)	0.26713(11)	0.0324(8)
H19A	0.6026	0.4298	0.2720	0.049
H19B	0.4487	0.5147	0.2471	0.049
H19C	0.3813	0.3926	0.2500	0.049
C20	0.7118(5)	−0.0103(3)	0.42859(12)	0.0298(8)
H20	0.8328	−0.0144	0.4050	0.036
C21	0.5321(6)	−0.0718(3)	0.40469(15)	0.0493(11)
H21A	0.5636	−0.1479	0.4055	0.074
H21B	0.5152	−0.0489	0.3702	0.074
H21C	0.4021	−0.0582	0.4230	0.074
C22	0.6242(6)	−0.0649(3)	0.51411(14)	0.0523(11)
H22A	0.5575	0.0043	0.5191	0.079
H22B	0.6883	−0.0885	0.5453	0.079
H22C	0.5184	−0.1166	0.5036	0.079
C23	0.8974(6)	−0.1556(3)	0.47009(16)	0.0506(11)
H23A	0.8001	−0.2105	0.4586	0.076
H23B	0.9581	−0.1768	0.5020	0.076
H23C	1.0098	−0.1465	0.4456	0.076
C24	0.0485(8)	0.8629(3)	0.31472(15)	0.0541(12)
H24A	−0.0087	0.9225	0.2956	0.081
H24B	0.1932	0.8788	0.3244	0.081
H24C	−0.0369	0.8518	0.3445	0.081

using petroleum ether/ethylacetate/diethylamine (50:1:1) as the eluent and recrystallization with dichloromethane.

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 methyl groups, at 1.5 times $U_{eq}(C)$ for the methyl groups.

Discussion

Previous studies have shown that *S. hookeriana* contains pregnane-type steroidal alkaloids [5]. These compounds have

cholinesterase inhibition [6], antileishmanial [7], antibacterial [8] and estrogen biosynthesis-promoting [9] activities.

The title compound crystallizes with one molecule in the asymmetric unit of the space group $P2_12_12_1$ (cf. the figure). Geometric parameters of the title structure are in the usual ranges. The compound contains one methoxy, one double bond and one dimethylamino group. The double bond were confirmed by the distance of 1.325(4) Å (C5–C6). The dimethylamino group was confirmed by the distance of 1.455(4) Å, 1.454(4) Å (C22–N1 and C23–N1), respectively. The compound contains three six membered rings and one five membered ring. The intermolecular connection was achieved by van der Waals interactions only.

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