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Crystal structure of (1*S*)-*N*-(chloromethyl)-1-((4*S*,6*aR*,8*aS*, 8*bR*, 9*aR*)-4-methoxy-6*a*,8*a*-dimethyl-1,3,4, 5,6,6*a*,6*b*,7,8,8*a*,9*a*,10,10*a*, 10*b*-tetradecahydro-8*bH*-naphtho[2',1':4,5] indeno[1,2-*b*]oxiren-8*b*-yl)-*N*-methylethan-1-amine, C₂₄H₄₆ClNO₅

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Abstract

C₂₄H₄₆ClNO₅, orthorhombic, *P*₂₁₂₁₂₁ (no. 19), *a* = 7.5992(3) Å, *b* = 9.6634(5) Å, *c* = 34.7499(7) Å, *V* = 2551.8(10) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0554, *wR*_{ref}(*F*²) = 0.1479, *T* = 170 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.

Source of material

The stems and roots of *Sarcococca hookeriana* (14.5 kg) were abstracted three times using MeOH with ultrasonic. The extract was concentrated and then 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 to five parts: *Frs. A–E*. The title compound was obtained by recrystallization of dichloromethane and silica gel column chromatography with petroleum ether/ethyl acetate/diethylamine (50:1:1).

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.12 × 0.08 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.18 mm ^{−1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ _{max} , completeness:	26.4°, 98%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	16563, 5154, 0.049
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3926
<i>N</i> (<i>param</i>) _{refined} :	295
Programs:	Bruker [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>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.9835(5)	0.4613(4)	0.55299(12)	0.0295(9)
H1A	1.091529	0.430545	0.539551	0.035*
H1B	0.992044	0.562794	0.556494	0.035*
C2	0.9780(6)	0.3933(5)	0.59250(13)	0.0335(10)
H2A	0.979351	0.291407	0.589574	0.040*
H2B	1.083120	0.420734	0.607496	0.040*
C3	0.8123(6)	0.4373(5)	0.61382(12)	0.0337(10)
H3	0.814737	0.539799	0.617775	0.040*
C4	0.6492(5)	0.3990(5)	0.59047(12)	0.0313(10)
H4A	0.639590	0.296913	0.589016	0.038*
H4B	0.543126	0.434239	0.603799	0.038*
C5	0.6551(5)	0.4573(4)	0.55039(13)	0.0272(9)
C6	0.5187(5)	0.5277(4)	0.53617(12)	0.0297(9)
H6	0.420327	0.542167	0.552569	0.036*
C7	0.5093(5)	0.5859(4)	0.49610(12)	0.0286(9)
H7A	0.520348	0.687880	0.497445	0.034*
H7B	0.392584	0.564239	0.484989	0.034*
C8	0.6520(5)	0.5295(4)	0.46943(12)	0.0253(9)
H8	0.619957	0.433480	0.461275	0.030*
C9	0.8282(5)	0.5257(4)	0.49134(12)	0.0245(9)
H9	0.846753	0.621355	0.501544	0.029*
C10	0.8222(5)	0.4295(4)	0.52689(12)	0.0257(9)
C11	0.9865(5)	0.4955(5)	0.46491(12)	0.0314(10)
H11A	1.096165	0.509565	0.479778	0.038*
H11B	0.982258	0.397056	0.457089	0.038*
C12	0.9941(5)	0.5852(5)	0.42867(12)	0.0301(10)
H12A	1.092234	0.554131	0.412053	0.036*
H12B	1.015903	0.682818	0.435930	0.036*
C13	0.8203(5)	0.5745(4)	0.40656(12)	0.0260(9)
C14	0.6719(5)	0.6202(4)	0.43403(12)	0.0251(9)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H14	0.708351	0.713053	0.443922	0.030*
C15	0.5155(5)	0.6478(5)	0.40723(12)	0.0311(10)
H15A	0.453464	0.561064	0.400506	0.037*
H15B	0.430916	0.713382	0.418954	0.037*
C16	0.6046(6)	0.7101(5)	0.37271(14)	0.0337(10)
H16	0.538770	0.718908	0.347912	0.040*
C17	0.7927(5)	0.6722(4)	0.37225(13)	0.0299(9)
C18	0.7921(6)	0.4272(4)	0.39093(13)	0.0323(10)
H18A	0.775174	0.363131	0.412471	0.049*
H18B	0.895395	0.398945	0.375995	0.049*
H18C	0.687715	0.425766	0.374370	0.049*
C19	0.8245(5)	0.2757(4)	0.51543(13)	0.0291(9)
H19A	0.797185	0.218740	0.537990	0.044*
H19B	0.941411	0.251183	0.505668	0.044*
H19C	0.736419	0.259212	0.495356	0.044*
C20	0.9175(6)	0.6627(5)	0.33856(13)	0.0332(10)
H20	0.964004	0.565946	0.338241	0.040*
C21	1.0759(6)	0.7581(6)	0.34336(15)	0.0442(13)
H21A	1.151473	0.751251	0.320591	0.066*
H21B	1.142635	0.730641	0.366244	0.066*
H21C	1.035303	0.853806	0.346323	0.066*
C22	0.9252(7)	0.6175(7)	0.26821(15)	0.0592(17)
H22A	1.044702	0.655225	0.266900	0.089*
H22B	0.864535	0.634435	0.243767	0.089*
H22C	0.930863	0.517648	0.273020	0.089*
C23	0.7937(8)	0.8342(6)	0.29036(17)	0.0580(15)
H23A	0.746446	0.881845	0.313012	0.087*
H23B	0.708495	0.839813	0.269247	0.087*
H23C	0.904166	0.878199	0.282513	0.087*
C24	0.9151(8)	0.4202(8)	0.67892(16)	0.0645(18)
H24A	1.035962	0.406843	0.669793	0.097*
H24B	0.898031	0.368940	0.702959	0.097*
H24C	0.894079	0.518930	0.683455	0.097*
Cl1	0.46639(16)	0.55202(14)	0.28313(4)	0.0486(4)
N1	0.8270(5)	0.6860(4)	0.29999(11)	0.0394(9)
H1	0.709538	0.639726	0.301555	0.047*
O1	0.7388(4)	0.8122(3)	0.38271(9)	0.0350(7)
O2	0.3985(10)	0.3615(11)	0.6787(2)	0.140(3)
H2C	0.399112	0.353521	0.703612	0.210*
H2D	0.501712	0.340087	0.669635	0.210*
O3	0.7165(7)	0.0845(6)	0.65439(17)	0.103(2)
H3A	0.671064	0.144882	0.670080	0.155*
H3B	0.794275	0.035038	0.666554	0.155*
O4	0.2214(6)	0.1986(5)	0.73934(19)	0.0949(18)
H4C	0.184391	0.257624	0.756404	0.142*
H4D	0.141026	0.136044	0.735452	0.142*
O5	0.7937(4)	0.3702(4)	0.65035(9)	0.0456(9)

Experimental details

The crystal structure was solved by direct methods and refined 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 methyl.

Comment

Studies show that *S. hookeriana* has many biological activities, such as cholinesterase inhibitory effect [5], immunosuppression and liver protection [6], and antibacterial effect [7]. Its main chemical component is pregnenic-type steroidal alkaloids [8]. *S. hookeriana* has been used as folk medicine in Asia areas for a long time to treat stomach diseases and so on.

The title compound crystallizes with one molecule in the asymmetric unit (see the Figure). Geometric parameters of the title structure are in the usual ranges [9, 10]. The compound contains three six membered rings, one five membered ring, and one oxirene moiety. The double bond was confirmed by the distance of 1.3375(6) Å (C5–C6). The oxirene was determined by the distances of 1.461(5) Å, 1.459(5) Å (C16–O1 and C17–O1), respectively. The intermolecular connection was achieved by van der Waals interactions only.

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