

The structure contains a racemic mixture of *R*- and *S*-forms of the ammonium cation (the chiral atom is C9).

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Structure of a Guaianolide Lactone Glucoside

BY ZHU YING

Instrumental Analysis and Research Centre, Lanzhou University, Lanzhou 730000, People's Republic of China

AND LI YU AND JIA ZHONG-JIAN*

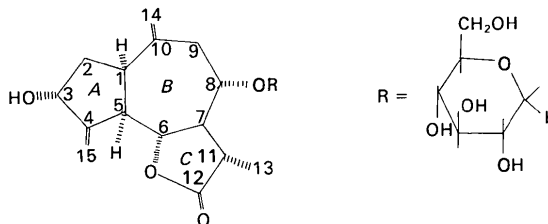
Laboratory of Applied Organic Chemistry, Department of Chemistry, Lanzhou University, Lanzhou 730000, People's Republic of China

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Abstract. (3*S*)-(3 α ,3 $\alpha\alpha$,4 α ,6 $\alpha\alpha$,8 α ,9 $\alpha\alpha$,9 β)-4-(β -D-Glucopyranosyloxy)decahydro-8-hydroxy-3-methyl-6,9-bis(methylene)azuleno[4,5-*b*]furan-2(3*H*)-one, C₂₁H₃₀O₉, m.p. = 517–518 K, *M_r* = 426.47, orthorhombic, *P*2₁2₁2₁, *a* = 7.093 (1), *b* = 13.365 (1), *c* = 21.557 (6) Å, *V* = 2043.6 (7) Å³, *Z* = 4, *D_x* = 1.386 Mg m⁻³, λ (Cu *K* α) = 1.5418, μ = 0.86 mm⁻¹, *F*(000) = 912, *T* = 293 K, final *R* = 0.031 for 1501 observed reflections. The *AB* and *BC* junctions are *cis* and *trans* respectively. C(3)—OH is substituted in the α position. The molecules are linked by hydrogen bonds.

Introduction. *Saussurea involucrata* Kar. et. Kin (Chinese name, Xue Lian Hua) is a Chinese herb, which is distributed mainly in Xinjiang province of China. It has been used for the treatment of rheumatic arthritis and gynopathy. A sesquiterpene lactone glucoside, C₂₁H₃₀O₉, was isolated from this plant. A very similar guaianolide has been characterized previously by NMR studies (Das, Baruah, Sharma, Baruah, Kulanthaivel & Hert, 1983), but this natural compound differs from ours in the configuration at C(3). Other guaianolide structures and closely related structures have also been reported (Declercq, Ger-

main, Van Meerssche, Demuynek, De Clercq & Vandewalle, 1980; McPhail & Sim, 1973; Asskawa, Taira, Toyota, Takemoto, Hert & Sakai, 1981; Hert, Watanabe & Blount, 1982; Declercq, Germain, Van Meerssche, Kok, De Clercq & Vandewalle, 1980). Though these structures differ from ours in some ways, these structural descriptions and features are helpful to further understand the structure of our compound.



Experimental. A crystal of dimensions 0.19 × 0.15 × 0.11 mm was mounted on an Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Cu *K* α radiation. Cell dimensions were obtained by least squares from 25 accurately centered reflections in the range 20–35°. A total of 1781 unique reflections (*R_{int}* = 0.021) with 4 ≤ 2 θ ≤ 120° were collected at room temperature (293 K). Experimental conditions were:

* To whom correspondence should be addressed.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

	x	y	z	B _{eq} (Å ²)
O(1g)	0.3112 (4)	0.0859 (2)	0.1125 (1)	2.25 (5)
O(2g)	0.6088 (4)	0.2998 (2)	0.1541 (1)	3.53 (6)
O(3g)	0.8094 (4)	0.2023 (2)	0.0518 (1)	3.47 (6)
O(4g)	0.5437 (4)	0.1174 (2)	-0.0372 (1)	3.18 (6)
O(6g)	0.0746 (4)	-0.0218 (2)	0.0136 (1)	4.17 (7)
O(3)	-0.3884 (4)	-0.0405 (2)	0.4130 (1)	2.99 (5)
O(6)	-0.0042 (4)	0.3010 (2)	0.3756 (1)	2.85 (5)
O(8)	0.3229 (3)	0.1740 (2)	0.2032 (1)	2.13 (4)
O(12)	0.1696 (4)	0.4360 (2)	0.3910 (1)	3.79 (6)
C(1g)	0.3517 (5)	0.1815 (3)	0.1392 (1)	2.11 (7)
C(2g)	0.5592 (5)	0.2048 (3)	0.1288 (2)	2.39 (7)
C(3g)	0.6104 (5)	0.2009 (3)	0.0602 (2)	2.41 (7)
C(4g)	0.5385 (5)	0.1066 (3)	0.0296 (1)	2.32 (7)
C(5g)	0.3343 (5)	0.0863 (3)	0.0460 (1)	2.45 (7)
C(6g)	0.2725 (6)	-0.0150 (3)	0.0229 (2)	3.46 (9)
C(1)	-0.0891 (5)	0.0307 (3)	0.3223 (2)	2.24 (7)
C(2)	-0.3058 (6)	0.0312 (3)	0.3145 (2)	2.66 (7)
C(3)	-0.3786 (5)	0.0538 (3)	0.3794 (2)	2.53 (7)
C(4)	-0.2307 (6)	0.1231 (3)	0.4066 (2)	2.62 (7)
C(5)	-0.0539 (5)	0.1218 (3)	0.3664 (1)	2.18 (7)
C(6)	-0.0221 (5)	0.2189 (3)	0.3305 (2)	2.08 (7)
C(7)	0.1642 (5)	0.2205 (2)	0.2940 (1)	1.99 (7)
C(8)	0.1363 (5)	0.1965 (3)	0.2251 (1)	2.15 (7)
C(9)	-0.0027 (5)	0.1095 (3)	0.2160 (2)	2.31 (7)
C(10)	0.0247 (5)	0.0270 (3)	0.2625 (2)	2.24 (7)
C(11)	0.2492 (5)	0.3228 (3)	0.3069 (2)	2.35 (7)
C(12)	0.1412 (6)	0.3612 (3)	0.3615 (2)	2.68 (8)
C(13)	0.4604 (6)	0.3268 (3)	0.3176 (2)	3.90 (9)
C(14)	0.1483 (6)	-0.0444 (3)	0.2529 (2)	3.26 (8)
C(15)	-0.2521 (7)	0.1738 (3)	0.4586 (2)	4.5 (1)

ω - 2θ scan, 2θ scan width $(0.52 + 0.15\tan\theta)^\circ$, aperture $(1.6 + 0.5\tan\theta)$ mm, maximum scan time 60 s, background one quarter of scan time at each of the scan limits. Three standard reflections measured at regular intervals showed no significant change in intensity. The intensity data were corrected for Lorentz and polarization effects. An empirical absorption correction was applied (maximum and minimum transmission 0.997 and 0.982 respectively). 1500 observed reflections with $I \geq 3\sigma(I)$ were used in the subsequent analysis. On the basis of systematic absences ($h00, h = 2n + 1; 0k0, k = 2n + 1; 00l, l = 2n + 1$), the space group of the compound was determined to be $P2_12_12_1$.

The structure was solved by direct methods (*MULTAN82*; Main *et al.*, 1982) and Fourier techniques and refined with 1501 observed reflections by a full-matrix least-squares technique using anisotropic thermal parameters for all 30 non-H atoms. The function minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 1$ for all observed reflections and $w = 0$ for unobserved reflections. A difference Fourier synthesis upon convergence permitted the location of 30 H atoms. In subsequent cycles of refinement the H atoms were assigned fixed isotropic thermal parameters. In the final cycle of refinement R and wR were 0.031 and 0.032 respectively (maximum shift/e.s.d. = 0.39) and the error in an observation of unit weight was 0.84. The highest peak in the final difference Fourier had a density of $0.22 \text{ e } \text{Å}^{-3}$.

Table 2. Bond distances (Å), bond angles (°) and torsion angles (°)

C(1)—C(2)	1.546 (5)	C(11)—C(13)	1.517 (5)
C(2)—C(3)	1.521 (5)	C(8)—O(8)	1.438 (4)
C(3)—C(4)	1.518 (5)	C(1g)—O(1g)	1.397 (4)
C(4)—C(5)	1.524 (5)	C(1g)—O(1g)	1.431 (4)
C(5)—C(1)	1.564 (5)	C(5g)—O(1g)	1.442 (4)
C(5)—C(6)	1.528 (5)	C(1g)—C(2g)	1.521 (4)
C(6)—C(7)	1.538 (5)	C(2g)—C(3g)	1.524 (5)
C(7)—C(8)	1.532 (4)	C(3g)—C(4g)	1.511 (5)
C(8)—C(9)	1.537 (5)	C(4g)—C(5g)	1.515 (5)
C(9)—C(10)	1.502 (5)	C(5g)—C(6g)	1.507 (6)
C(10)—C(1)	1.522 (5)	O(2g)—C(2g)	1.427 (4)
C(6)—O(6)	1.472 (4)	O(3g)—C(3g)	1.423 (5)
O(6)—C(12)	1.344 (5)	O(4g)—C(4g)	1.446 (4)
C(12)—C(11)	1.495 (5)	O(6g)—C(6g)	1.421 (5)
C(11)—C(7)	1.520 (5)	O(3)—C(3)	1.455 (4)
C(4)—C(15)	1.318 (5)	O(12)—C(12)	1.201 (4)
C(10)—C(14)	1.313 (5)		
C(2)—C(1)—C(10)	115.8 (3)	O(6)—C(6)—C(7)	104.7 (3)
C(2)—C(1)—C(5)	102.8 (3)	C(12)—O(6)—C(6)	111.3 (3)
C(1)—C(2)—C(3)	103.8 (3)	O(6)—C(2)—C(11)	111.5 (3)
C(2)—C(3)—C(4)	104.0 (3)	C(12)—C(11)—C(7)	104.5 (3)
C(3)—C(4)—C(5)	110.0 (3)	C(12)—C(11)—C(13)	111.9 (3)
C(4)—C(5)—C(1)	102.9 (3)	C(7)—C(11)—C(13)	116.8 (3)
C(4)—C(5)—C(6)	113.6 (3)	C(11)—C(7)—C(6)	105.0 (3)
C(1)—C(5)—C(6)	112.2 (3)	C(8)—O(8)—C(1g)	116.4 (3)
C(5)—C(6)—C(7)	113.5 (3)	O(8)—C(1g)—C(2g)	107.6 (3)
O(6)—C(6)—C(5)	108.1 (3)	C(2g)—C(1g)—O(1g)	108.5 (3)
C(6)—C(7)—C(8)	112.4 (3)	C(1g)—C(2g)—C(3g)	111.5 (3)
C(8)—C(7)—C(11)	114.7 (3)	C(2g)—C(3g)—C(4g)	111.8 (3)
C(7)—C(8)—C(9)	111.4 (3)	C(3g)—C(4g)—C(5g)	111.7 (3)
C(7)—C(8)—O(8)	104.1 (3)	C(4g)—C(5g)—O(1g)	109.9 (3)
C(9)—C(8)—O(8)	113.0 (3)	C(5g)—O(1g)—C(1g)	112.0 (2)
C(8)—C(9)—C(10)	112.8 (3)	C(1g)—C(2g)—O(2g)	111.4 (3)
C(9)—C(10)—C(1)	118.2 (3)	C(3g)—C(2g)—O(2g)	110.0 (3)
C(10)—C(1)—C(5)	117.1 (3)	C(2g)—C(3g)—O(3g)	111.0 (3)
C(3)—C(4)—C(15)	124.2 (4)	C(4g)—C(3g)—O(3g)	106.9 (3)
C(5)—C(4)—C(15)	125.8 (4)	C(3g)—C(4g)—O(4g)	110.1 (3)
C(1)—C(10)—C(14)	120.7 (3)	C(5g)—C(4g)—O(4g)	106.0 (3)
C(9)—C(10)—C(14)	121.0 (3)	C(4g)—C(5g)—C(6g)	111.2 (3)
O(6)—C(12)—O(12)	120.5 (3)	O(1g)—C(5g)—C(6g)	106.9 (3)
C(11)—C(12)—O(12)	128.1 (4)	C(5g)—C(6g)—O(6g)	113.0 (3)
C(2)—C(3)—O(3)	107.6 (3)	O(8)—C(1g)—O(1g)	107.7 (3)
C(4)—C(3)—O(3)	111.7 (3)		
A		C	
C(5)—C(1)—C(2)—C(3)	-41.4	C(6)—C(7)—C(11)—C(12)	15.1
C(1)—C(2)—C(3)—C(4)	33.7	C(7)—C(11)—C(12)—O(6)	-7.4
C(2)—C(3)—C(4)—C(5)	-13.5	C(11)—C(12)—O(6)—C(6)	-4.0
C(3)—C(4)—C(5)—C(1)	-11.9	C(12)—O(6)—C(6)—C(7)	13.6
C(4)—C(5)—C(1)—C(2)	32.1	O(6)—C(6)—C(7)—C(11)	-17.4
B		D	
C(10)—C(1)—C(5)—C(6)	37.8	C(2g)—C(1g)—O(1g)—C(5g)	-64.7
C(1)—C(5)—C(6)—C(7)	-68.4	O(1g)—C(1g)—C(2g)—C(3g)	56.2
C(5)—C(6)—C(7)—C(8)	99.6	C(1g)—C(2g)—C(3g)—C(4g)	-48.7
C(6)—C(7)—C(8)—C(9)	-42.4	C(2g)—C(3g)—C(4g)—C(5g)	47.2
C(7)—C(8)—C(9)—C(10)	-40.7	C(3g)—C(4g)—C(5g)—O(1g)	-53.6
C(8)—C(9)—C(10)—C(1)	91.7	C(4g)—C(5g)—O(1g)—C(1g)	63.8
C(9)—C(10)—C(1)—C(5)	-62.6		

The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72–73). All calculations were carried out on a PDP 11/44 computer using *SDP* programs.

Discussion. The final atomic coordinates of all non-H atoms and isotropic temperature factors calculated from the anisotropic temperature parameters are

listed in Table 1.* The bond distances, bond angles and torsion angles are given in Table 2.

This natural compound contains a cyclopentane ring (*A*), a cycloheptane ring (*B*), a lactone ring (*C*) and a glucosyl ring (*D*). According to the IR, MS, ^1H NMR and ^{13}C NMR spectra, the aglucone $\text{C}_{15}\text{H}_{20}\text{O}_4$ is a guaianolide with hydroxy groups at the C(3) and C(8) positions. The glucoside residue is attached at the C(8) position. The absolute configuration of the structure was not determined by X-ray diffraction techniques, but correlation with the guaianolide (*2b*) given by Das, Baruah, Sharma, Baruah, Kulanthaivel & Hert (1983) establishes the α and β designation.

Fig. 1 is an ORTEP (Johnson, 1976) view of the molecule which shows the hydroxy groups attached to C(3) and C(8) and the methyl group attached to C(11) having α configuration. The stereochemistry of the molecule shows: C(1)— αH is *cis* to C(5)— αH , C(6)— βH is *trans* to C(7)— αH , thus the *AB* and *BC*

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53773 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

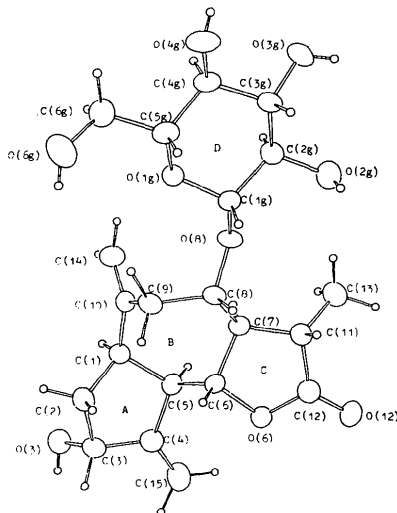


Fig. 1. Molecular structure and numbering scheme.

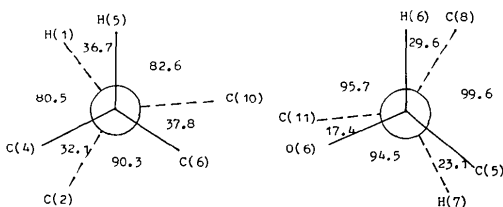


Fig. 2. Newman projection around C(1)—C(5) and C(6)—C(7).

Table 3. Ring conformation

	A (cyclopentane)	B (cycloheptane)	C (lactone)
Description	Half-chair	Twist-chair	Half-chair
Approximate symmetry	C_2	C_2	C_2
Pseudo-symmetry element	2	2	2
From atom	C(4)	C(8)	C(12)
To the midpoint of bond	C(1)—C(2)	C(1)—C(5)	C(6)—C(7)
Asymmetry parameter ΔC_2	1.6°	5.7°	2.6°

Table 4. Hydrogen bonds (Å)

A—H...B	A...B	A—H	H...B	A—H...B (°)	Position of atom B
O(2g)—H(O2g)...O(3)	3.015	0.794	2.219	176.9	$-x, \frac{1}{2} + y, \frac{1}{2} - z$
O(3g)—H(O3g)...O(4g)	2.944	0.786	2.245	148.6	$\frac{1}{2} + x, \frac{1}{2} - y, -z$
O(4g)—H(O4g)...O(3)	2.862	0.714	2.158	168.6	$\frac{1}{2} - x, -y, -\frac{1}{2} + z$
O(6g)—H(O6g)...O(12)	2.747	0.853	1.941	157.2	$-x, -\frac{1}{2} + y, \frac{1}{2} - z$
O(3)—H(O3)...O(6g)	2.677	0.876	1.833	159.8	$-\frac{1}{2} - x, -y, \frac{1}{2} + z$

ring junctions are *cis* and *trans* fused respectively. The Newman projection of Fig. 2 illustrates the stereochemistry of the junctions between ring *B* and rings *A* and *C*.

The torsion angles of the cyclopentane ring show that the ring possesses pseudorotation symmetry about an axis bisecting the C(1)—C(2) bond and passing through the C(4) atoms; therefore, the cyclopentane ring has a half-chair conformation (Duax, Weeks & Rohrer, 1974, 1976; Eliel, 1962). Because the lactone ring has pseudo C_2 symmetry, its conformation is also a half-chair.

In the cycloheptane ring, the C_2 symmetrical values of the torsion angles, *i.e.* 38, -66, 95, -41°, are similar to those (53, -76, 97, -41°) proposed by Hendrickson (1961) for a cycloheptane molecule with equal bond lengths; therefore, the cycloheptane ring is in a twist-chair conformation (Duax, Weeks & Rohrer, 1974; Dale, 1978; Hendrickson, 1961, 1963, 1976). The conformations of the three fused rings are described with some further comments in Table 3.

The average bond angle values in rings *A*, *B*, *C* and *D* are 104.7, 113.9, 107.4 and 110.9° respectively. These data suggest that the internal stress in rings *A* and *B* is larger than for rings *C* and *D*. Examination of the valence angles associated with the trigonal C atoms in rings *A*, *B* and *C* demonstrates the greater degree of strain present in the five-membered rings.

There are five intermolecular hydrogen bonds, whose geometric details are given in Table 4.

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Structure of a 2,6-Pyridinophane

BY GEORGE R. NEWKOME

Department of Chemistry, University of South Florida, Tampa, Florida 33620, USA

TOSHIO KAWATO

Laboratory of Chemistry, Kyushu University, Ropponmatsu, Fukuoka 810, Japan

AND FRANK R. FRONCZEK

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803, USA

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Abstract. 2,5,11,14-Tetraoxa-19,20-diazatricyclo-[13.3.1.1^{6,10}]jcosa-1(19),6,8,10(20),15,17-hexaene, C₁₄H₁₄N₂O₄, $M_r = 274.3$, monoclinic, $P2_1/c$, $a = 9.354$ (2), $b = 9.011$ (3), $c = 7.954$ (2) Å, $\beta = 101.75$ (2)°, $V = 656.4$ (6) Å³, $Z = 2$, $D_x = 1.388$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.97$ cm⁻¹, $F(000) = 288$, $T = 299$ K, $R = 0.035$ for 834 observations with $I > 3\sigma(I)$. The molecule lies on an inversion center and is in the *anti* or stepped conformation. The ethylene glycol chains are fully extended, and the two N—C—O—C torsion angles about the ring-O bonds are near zero, 1.6 (2) and -2.3 (2)°.

Introduction. From crystallographic and NMR studies, [2.2]metacyclophanes possess a 'stepped' (*anti*) conformation (Majestic & Newkome, 1982; Newkome, Nayak, McClure, Danish-Koshboo & Broussard-Simpson, 1977). With 3-substituted 2,6-pyridinophanes, the observed NMR data could not be accommodated with this model, thus an '*anti*, transverse' isomerization was proposed (Newkome

& Kawato, 1979) to rationalize the variable-temperature solution studies. This investigation confirms the *anti* conformation in the crystalline state (Fronczek, 1982).

Experimental. The title metacyclophane was obtained (16%) from the reaction of 2,6-dibromopyridine and sodioethyleneglycolate (Newkome *et al.*, 1977) in the form of analytically pure colorless crystals: m.p. 488–489 K, ¹H NMR (CDCl₃, 311 K) δ 4.66 (*bs*, CH₂O, 8H), 6.30 (*d*, 3,5-pyrH, $J = 8$ Hz, 4H), 7.50 (*t*, 4-pyrH, $J = 8$ Hz, 2H). Crystals were grown from cyclohexane/ethyl acetate. A single crystal of dimensions 0.18 × 0.33 × 0.60 mm was used for intensity-data collection on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. The space group was determined from systematic absences $h0l$ with l odd and $0k0$ with k odd. Cell dimensions were determined from the setting angles of 25 reflections having $13 < \theta < 15$ °. Data were collected by ω - 2θ scans designed for $I = 25\sigma(I)$, subject to a maximum scan time of 120 s.