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A New Disulfated Triterpene Glycoside from the Sea Cucumber *Mensamaria intercedens* Lampert

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Abstract: A new disulfated triterpene glycoside named intercedenside B was isolated from the sea cucumber *Mensamaria intercedens* Lampert. The structure was deduced from the spectral data.

Keywords: *Mensamaria intercedens* Lampert, intercedenside B, disulfated triterpene glycoside.

Mensamaria Intercedens Lampert, a kind of sea cucumber of Cucumariidae, is widely distributed in Southchina sea, especially in Zhaoan Gulf and Dongshan Gulf, Fujian province[1]. In this study, we report the structure elucidation of a new disulfated triterpene glycoside from this sea cucumber.

Compound **1**, $C_{55}O_{22}H_{84}S_2O_6Na_2$, a colorless amorphous powder, mp 218-220°C, $[\alpha]_D^{20}$ −14.2(c 0.5, pyridine), exhibited a pseudomolecular ion peak at *m/z* 1325 $[M+Na]^+$ in the positive-ion mode ESI-MS and a pseudomolecular ion peak at *m/z* 1279 $[M-Na]^-$ in the negative-ion mode ESI-MS. Fragment ion peaks at *m/z* 1177 $[M-Na-SO_3Na+H]^-$, and 1051 $[M-2Na-2SO_3Na+H]^-$ indicated the presence of two sulfate groups in the glycoside. In its IR spectrum, it showed the presence of hydroxyl (3502 cm^{-1}), carbonyl (1746 cm^{-1}), olefinic bond (1650 cm^{-1}) and sulfate groups (1239 cm^{-1}, 1067 cm^{-1}).

An examination of the ^1H and ^{13}C NMR spectra of compound **1**, suggested the presence of a triterpenoid aglycon with two olefinic bonds, one ester, and one lactone carbonyl group and an oligosaccharide chain composed of four sugar units. The NMR data of compound **1 (Table 1)** showed that the aglycon moiety of compound **1** was very similar to 16β-acetoxyholosta-7, 24-diene-3β−ol and the oligosaccharide chain was different from those reported sea cucumber glycosides obviously[2].

The presence of four β-monosaccharide units in compound **1** was deduced from the ^1H and ^{13}C spectrum, which showed four anomeric carbon resonances and four anomeic protons with coupling constants (J values) 7.2-7.8Hz **(Table 1)**. The presence of xylose, quinovose, and 3-O-methyl glucose in a 2:1:1 ratio was comfirmed by acid hydrolysis

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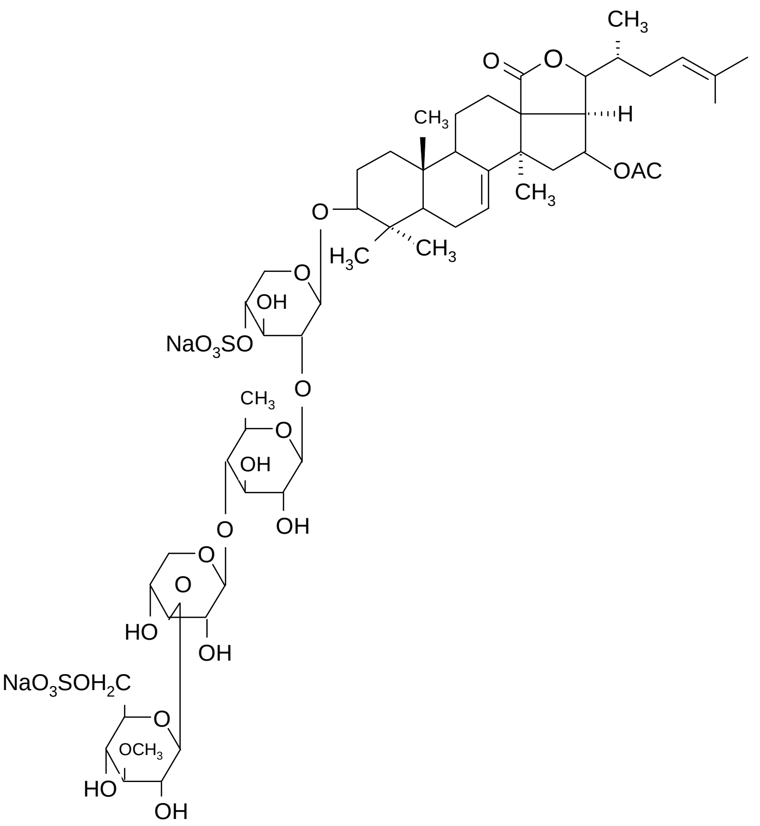
Compound **1**



with 15% H_2SO_4 followed by GC-MS analysis of the corresponding alditol peracetates. The sequence of the sugar residues in compound **1** was determined by careful analysis of HMBC correlations. Since the HMBC spectrum showed cross peaks at 4.665/88.900 (H-1′/C-3), 4.934/81.659 (H-1′′/C-2′), 4.736/85.053 (H-1′′′/C-4′′), 5.088/86.421 (H-1′′′′/C-3′′′), the sequence of the sugar residues must be 3-O-methyl-glc(1→3)-xyl (1→4)-qui(1→2)-xyl(1→3)-aglycon.

The site of linkage of the sulfate groups in the sugar units was determined by comparison of ^{13}C-NMR data of compound **1** with those reported glycosides[2]. Esterification shifts were observed at the signals of C-4′(xyl$_1$, from 68 to 76), C-6′′′′ (3-O-methyl-glc, from 61 to 67).

Therefore, the structure of compound **1** was deduced as 16β-acetoxy-3-O-{6′′′′-O-sodium sulfate-3′-O-methyl-β-D-glucopyranosyl(1→3)- β-D-xylopyranosyl(1→4)-β-D-quinovopyranosyl(1→2)-4′-O-sodium sulfate-β-D-xylopyranosyl}-holosta-7, 24-diene-3β-ol.

Table 1 ^{1}H and ^{13}C NMR data for compound **1** (in pyridine-d_5—D_2O 4:1, 600MHz)

Carbon	δ_C	δ_H (m, J in Hz)		Carbon	δ_C	δ_H (m, J in Hz)
1	35.4	1.267(2H, br)	Xy1$_1$	1	103.8	4.67 (1H, d, 7.2)
2	26.3	1.80(1H, m), 1.90(1H, m)		2	81.7	3.95 (1H, 1H, t, 7.8)
3	88.9	3.152 (1H, dd, 3.6, 12)		3	74.1	4.21(1H, t, 9)
4	38.9			4	75.8	4.89 (1H, m)
5	47.7	0.87 (1H, dd, 9.6, 12.8)		5	63.2	3.71(1H, m), 4.71 (1H,,m)
6	23.1	1.86 (2H, m)	Qui	1	104.1	4.93 (1H, d, 7.8)
7	119.9	5.57 (1H, s)		2	74.3	3.90 (1H, m)
8	145.0			3	75.0	3.80 (1H, m)
9	46.7	3.24 (1H, d, 13.8)		4	85.0	3.47(1H, t, 9)
10	34.9			5	71.1	3.62(1H, m)
11	22.0	1.44 (1H, m),1.68(1H, m)		6	17.2	1.57 (3H, s)
12	30.8	2.09 (2H, m)	Xy12	1	103.9	4.74 (1H, d, 7.8)
13	58.7			2	72.8	3.83 (1H, m)
14	47.0			3	86.4	4.02 (1H, m)
15	43.1	1.57(1H, m), 2.55 (1H, dd,		4	68.3	3.88(1H, m)
16	75.2	5.80 (1H, ddd, 7.2, 8.4,		5	65.1	3.58 (1H, m), 4.136 (1H, m)
17	53.9	2.69 (1H, d, 8.4)	3-OMeGlu	1	104.3	5.09 (1H, d, 7.8)
18	180.1			2	73.7	3.75 (1H, m)
19	23.4	1.06(3H, s)		3	85.7	3.64 (1H, m)
20	85.4			4	69.4	3.91 (1H, m)
21	27.7	1.56(3H, s)		5	74.7	4.01(1H, m)
22	38.1	1.78(1H, m), 2.36 (1H, m)		6	66.8	4.62 (1H, dd, 6, 11.4), 4.88(1H, m)
23	22.7	1.81 (1H, m), 1.98(1H, m)		OCH_3	60.8	3.84(3H, s)
24	123.3	4.95(1H, d, 7.8)				
25	132.0					
26	25.0	1.57 (3H, s)				
27	17.4	1.59 (3H, s)				
30	16.8	0.96 (3H, s)				
31	28.2	1.13 (3H, s)				
32	31.8	1.14 (3H, s)				
CH_3COO	170.8					
CH_3COO	31.8	1.95 (3H, s)				

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