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New saponin from the stem bark of Samanea Saman

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Abstract: From the stem bark of *Samanea saman* new triterpenoid glycoside have been isolated and characterized as 3β, 22β – dihydroxyolean -12-ene-24-*O*-β-D-galactopyranoside 1 by spectral data and chemical studies.

(**Keywords:** Samanea saman, new triterpenoid glycoside)

Introduction

Samanea saman (Mimosaecae) commonly called "Raintree" is a useful medicinal plant distributed from Yucatan Peninsula, Guatemala to Peru, Bolivia, Brazil, throughout the West Indies, in old world tropics and also in Southern Florida[1]. It is used as an alcoholic source[2]. It is used for cold, diarrhea, headache, intestine ailments, stomachach[3], stomach cancer[4], sore throat[5]. Earlier alkaloids, lupeol, lupeone, octaconsanolic acid, hexacosanol, flavonoids, kaempferol[6] have been isolated from the different parts of *Samanea saman.*

In continuation of our research on the chemical investigation of medicinal plants, we report herein isolation and structure elucidation of new triterpenoid glycosides from *Samanea saman*.

The water insoluble portion of the hot ethanol extract of the air-dried crushed and defatted stem barks of *S. saman* on column chromatography yield compound **1**.

Experimental section :

The stem bark of *Samanea saman* was collected in April, 1999 from the Botany Department, University of Allahabad, Allahabad, U.P., India. M.ps measured in open capillary tube and are corrected. ^{1}HNMR and ^{13}CNMR spectra were recorded on a JEOLUNK-A 500 spectrometer in $CDCl_3$ using TMS as an internal standard at 300 MHz and 100 MHz, respectively. IR spectra were run in KBr pellets. Mass spectra were recorded on JEOLSX 102/DA-6000 mass spectrophotometer.

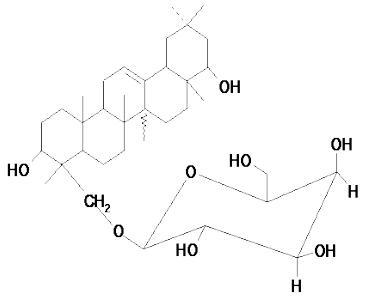
The air-dried and finely crushed stem bark (5kg) of *Samanea saman* was repeatedly extracted with boiling EtOH (4x10*l*), concentrated under reduce pressure in a rotatory evaporator and poured into an excess of ice-cold distilled water with constant stirring to give reddish brown aqueous solution (fraction-I) and light brown residue (fraction-II) were obtained. The water insoluble portion was extracted successively with different solvents of increasing polarity over a sintered Column. Elution with solvent system C_6H_6: methanol (7:3, v/v) followed by runing in preparative TLC using benzene :chloroform (8:2, v/v) marked with the help of UV lamp gave compounds **1** (0.55g).

Compound **1,** mp. 166^{o}C, yield 550 mg, homogenous on TLC, R_f 0.48 solvent C_6H_6:$CHCl_3$ (8:2, v/v); Anal. Found: C, 71.90; H, 9.83; Calcd. for $C_{36}H_{59}O_8$: C, 71.95; H, 9.89% . IR (KBr) 3450, 2923, 1630, 1100, 1050. ^{1}HNMR ($CDCl_3$, 300 MHz) : δ 0.85 (s, 3H, H-25), 0.95 (s,3H, H-26), 0.99 (s,3H, H-29), 1.03 (s,3H, H-30), 1.17 (s,3H, H-27), 1.31 (s,3H, H-28), 1.35 (s,3H, H-23), 2.59 (brs,1H, *J*=13Hz), 3.83 (dd, 1H, *J*=9.6 Hz, β-OH at C-3), 5.25 (t, 1H, *J*=3.3 Hz), 4.35 and 4.52 (d, 1H, *J*=12.1 Hz for $-CH_2OH$ group proton), 4.42 (d, 1H, *J*=7 Hz for

β linkage) and 3.52-3.88 (m, 5H, sugar protons); MS : m/z 617[M^+] 487, 438, 391, 234, 224, 219, 216, 204, 201, 175, 119; ^{13}C-NMR data are given in **Table-I**.

Table –I
^{13}C-NMR (δ) values of glycoside 1 and its aglycone 1a

Chemical Assigned	Glycoside Chemical Shift (ppm)	Aglycone Chemical shift (ppm)
C-1	38.6 (t)	39.0(t)
C-2	27.5(t)	28.4(t)
C-3	80.0 (d)	79.8(d)
C-4	43.0 (s)	43.0(s)
C-5	57.0(d)	56.3(d)
C-6	19.2(t)	19.4(t)
C-7	33.7(t)	33.5(t)
C-8	40.1(s)	39.9(s)
C-9	48.6(d)	48.1(d)
C-10	36.8(s)	37.0(s)
C-11	24.6(t)	24.0(t)
C-12	123.0(d)	122.4(d)
C-13	145.0(s)	144.9(s)
C-14	42.0(s)	42.3(s)
C-15	26.0 (t)	26.4(t)
C-16	29.4 (t)	29.0(t)
C-17	37.5 (s)	38.4 (s)
C-18	44.5(d)	44.8(d)
C-19	41.9 (t)	41.5 (t)
C-20	30.5(s)	30.0(s)
C-21	37.0(t)	37.3(t)
C-22	75.6(d)	75.6(d)
C-23	23.1(q)	23.5(q)
C-24	74.5 (t)	64.5 (t)
C-25	16.2(q)	16.3(q)
C-26	17.5(q)	17.1(q)
C-27	26.8(q)	25.5(q)
C-28	21.1(q)	21.1(q)
C-29	32.5(q)	32.0(q)
C-30	24.6(q)	24.4(q)
C-1′	106.8(q)	
C-2′	74.5 (q)	
C-3′	75.2 (d)	
C-4′	71.1 (d)	
C-5′	76.6 (d)	
C-6′	64.6(t)	



3β, 22β – dihydroxyolean -12-ene-24-*O*-β-D-galactopyranoside **1**

Results and Discussion

Compound **1** $C_{36}H_{59}O_8$, a glycoside on acid hydrolysis gave D-xylose and an aglycone **1a**. The aglycone gave colour reactions[7-10] of unsaturated pentacyclictriterpenoid. It formed triacetate on acetylation showing the presence of three hydroxyl group. The IR spectrum of **1a** showed absorption for hydroxyl (3450 cm^{-1}), trisubstituted double bond (1630 cm^{-1}), primary and secondary alcoholic group (1250, 1100, 1050 cm^{-1}) and C-methyl (2900, 1385 and 1330 cm^{-1}) groups. The ^{1}HNMR spectrum of **1a** showed signals for seven tertiary methyl groups at (δ0.85, 0.95, 0.99, 1.03, 1.17, 1.31 and 1.35 (each 3H, S) and a vinyl proton at 5.25 (1H, t, *J*=3.3Hz) all of which suggested **1a** to be an olean-12-ene derivative.

The mass spectrum of **1a** revealed important peaks at m/z 234, 224, 210, 216, 204, 201 and 175 typical of retro-Diels-Alder fragmentation of ring-C of olean-12-ene derivative containing a hydroxyl group in ring A or B[11]. Compound **1a** also gave positive Zimmermann test[12] suggesting the C-3 position for this hydroxyl group which was also biogenetically favoured. The aglycone showed signal at δ3.83 (1H, dd, *J*=9.6 Hz) with higher value of coupling constant confirming the β–orientation for hydroxyl group at C-3 (α or axial H)[13]. The ion peak at m/z 216[234-H_2O) due to ready loss of water molecule showed the position of –OH group at C-22 which was also supported by the ^{13}C-NMR signal at δ75.6(d) for C-22. The ^{1}H-NMR signals at δ4.35 and 4.52 (each 1H, d, *J*=12.1 Hz) were attributed to the methylene protons of hydroxymethyl group (AB system)[14]. Signal at δ 64.5 in ^{13}C-NMR spectrum of **1a** confirmed the presence of CH_2OH group at C-24. Thus, aglycone **1a** was identified as 3β, 22β, 24-trihydroxyolean-12-ene.

The ^{1}H-NMR spectrum of **1** showed a signal for an anomeric proton at δ 4.42 (1H, d, *J*=7.0 Hz, H-1, galactose) and sugar protons at δ3.52-3.88 (br, galactosyl protons) which was consistent with configuration of D-galactose.[15] The β-type of glycosidic linkage was also confirmed by hydrolysis of **1** with the enzyme emulsin. The position of attachment of D-galactose at C-24 hydroxyl group was confirmed by down field signal at δ74.5 (t) in the 13C-NMR of **1** for C-24. Thus the compound **1** was characterized as 3β, 22β–dihydroxyolean -12-ene-24-O-β-D-galactopyranoside.

References

1. A. James Duke, *Handbook of Energy Crop,* (1983).
2. CSIR (Council of Scientific and Industrial Research) *The Wealth of India*, New Delhi, **11**, 1948 (1976).
3. J.A. Duke, & K. K. Wain, *Medicinal Plants of the World*, **3**, (1981).
4. JL Hartwell, *Plants used against Cancer,* A survey Lloydia, **30**, 1967 (1971)**.**
5. ES Ayensu, *Medicinal Plants of the West Indies*, Reference Publications, Inc., Algonac, MI, 282 (1981).
6. PH List and L Horhamner, *Hager's hand buchderpharmazeutischen praxis*, Springer, Verlag, Berlin, 2-6, 1969 (1979).
7. C. Liebermann , *Ber Deutsch Chem. Ges.,* 1804 (1885).

8. Takahashi K, Kawashi S, NishmuraKT, Kutota K, Tanabe Y and Takani M, *Chem Pharm Bull*, **22,** 650 (1974)
9. CH Brieskorne and B. Briner, *Pharm ActaHelv,* **28,** 139 (1953)
10. L. Ruzicka and Leibigs, *Ann Chem*, **25**, 471 (1929)
11. K. Takahashi, O. Tanka and S. Shibata, *Phytochemistry*, **8**, 2345 (1969)
12. DHR Barton and De Mayo P, *J ChemSoc.,* 887 (1954)
13. OB Rogers and G. Subramony, *Phytochemistry*, **27**, 531 (1988)
14. R. Sanghi, K. Tripathi and JP Sharma JP, *Indian J Chem.***39B**, 477 (2000)
15. R. Sanghi, P. Srivastava and J. Singh, *Indian J Chem.* **41B**, 1535 (2002)

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