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TERPENOIDS FROM CASSIA SIAMEA

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SUMMARY

2-oxo-1β, 3β, 19α-trihydroxyurs-12-ene-28-oic acid-β-D-glucopyranoside and 1β, 2α, 3β, 19α tetrahydroxy urs-12-ene-28-oate-3-0-β-D-glucopyranoside are two new triterpenoids isolated from Cassia siamea.

KEYWORDS*:* Cassia siamea,stem bark, triterpenoids, structure elucidation.

Cassia siamea, a member of fabaceae family is found abundantly in Nothern India,It is a large tree bearing yellow flowers.Investigation of this plant revealed the presence of anthraquinones, terpenoids, alkaloids and flavonoids (1&4).

Cassia siamea has recently been shown to have antimicrobial, antimalarial, antidiabetic, anticancer, hypotensive, diuretic, antioxidant,laxatie, antiinflammatory, analgesic, antipyretic, anxiolytic, antidepressant & sedative activities. The decoction of the stem bark is used against diabetes. The decoction is used as a mild, pleasant & safe purgative, scabies, urogenital diseases, herpes & rhinitis**.**

Compound 1: M^+ 502 was isolated as platelet crystals mp 140-142^{0}C. The IR spectrum showed the presence of hydroxy groups (3455 cm^{-1}) ester carbonyl (1740 cm^{-1}), carbonyl (1715 cm^{-1}) and double bond (1655 cm^{-1}). It gave positive liebermann-Burchard and Molish tests. On acid hydrolysis it gave D glucose. Comparision of the ^{13}CNMR and DEPT spectral data with those 2-oxopolmolic acid showed that their structure are very similar except that compound 1 had OH aditional on gp at C-1 and gluosyl moiety (δ 95.6) at C-28. The ^{1}HNMR spectrum also showed that glusoyl moeity was linked with the aglycone in the β -configuration (δ 6.23, d, J 7.9 Hz, 1H).

The ^{1}HNMR spectrum of compound I showed signals for 6 methyl gps as singlets δ 0.70, 0.86, 1.00 1.28 1.30. Signals at 2.57 (1H, 18-β-H) together with secondary methyl group (δ 0.95, d,3H J 7.0Hz) and olefinic proton (δ 5.35, t, 1H, J 3.6 Hz). The α orientation and tertiary hydroxy gp at C-19 was suggested by the chemical shift of Me 29 (δ 1.30) and Me 27 (1.20) signals, which were of the order reported for germinal (Δδ = 0.35) and vicinal (Δδ = 0.14), deshieldings in similar structural environments.(5) The ^{13} CNMR exhibited the presence of a methyl gp as a doublet which shows that compound 1 was 19α hydroxy urs -12-ene type of terpenoid.

The mass spectral analysis of compound 1a (aglycone) exhibited the peak at m/z 264 derived from the D/E ring and that at m/z 237 from the A/B ring formed through the characteristic retero Diels Alder fragmentation of C indicated the presence of 2 hydroxyl gp in A/B ring system and one hydroxy gp. D/E ring system.(2)

Compound 2 : C_{36} H_{58} O_{10} mp - 138° on acid hydrolysis with 7% H_2SO_4 it gave aglycone. 2a. The sugar was identified as D-glucose on the basis of co-paper chromatography with an authentic sample. IR spectra of compound 2a showed absorption due to hydroxyl 3400 cm^{-1} , ester carbonyl 1712 cm^{-1} and trisubstituted double bond 1520 cm^{-1} . The mass spectra of compound 2a M^+ 518. Two main fragments at m/z 279 and m/z 240 due to retro Diels Alder fragmentation of ring C. commonly found in spectra urs-12-ene derivatives possessing a hydroxyl and a carbonyl groups in rings D/E. This was confirmed by peaks at m/z 261 [279-H_2O]$^+$, 220 [279-COO CH_3]$^+$ and 201 [264 – H_2O COO CH_2]$^+$, other important mass fragment of ions at m/z 179 and 196 were due to tertiary hydroxyl functions on C-19 in an urs –12-ene skeleton (2) The fragmentation pattern (m/z 240) also indicated that three secondary hydroxyl groups were located in the A/B ring portion of the compound.

On acetylation, compound 2a showed signals for three acetoxymethine protons at δ4.78 (d, 1H,J 10.5 Hz) 4.86 (d,1H, J 9.3 Hz) and 5.15 (dd, 1H, J_1 10.5 Hz, J_2 9.3 Hz) which were asignable to C-1 (or C-3), C-3 (or C-1) and C-2 respectively. The J values of these signals indicated transdiaxial correlated protons, hence the hydroxyl groups must be equatorial (3) The structure of compound 2a is also supported by^{13} CNMR spectrum

Extraction of Isolation : Air dried and crushed material was extracted with boiling ethanol. The extract was chromatographed over a dry flash column (silica gel G) yielded compound 1 (0.3g) hexane: benzene (7:3/v/v) and compound 2 (0.4g) with benzene.

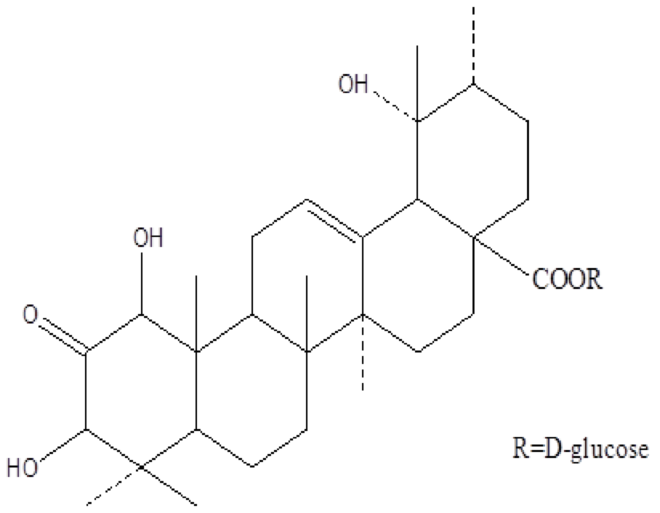
Compound 1: mp 140-142° IR bands (KBr) : 3455, 1740, 1715, 1665, 1635, 1235, 1180, 1160, 1010, 940, 835 cm^{-1} ; ^{1}HNMR (CDCl$_3$ 400 MHz): δ 6.23 (d J 7.9 Hz 1H, H-1 of glu), 5.35 (br.s, 1H, H-12), 5.18 (s, 1H, H-3α), 2.84 (s, 1H, H-18), 0.70, 0.86, 1.00, 1.20, 1.30 (s, 6x 3H, H-23, 24, 25, 26, 27, 29) 1.06 (J 6.3 Hz, 3H, H-30) 4.41 – 4.00 (m, 6H, sugar H) 4.80 (d, 1H, J 4 Hz, H-1) ^{13}CNMR : see table 1, EIMS m/z 502 [M-Glc]$^+$ 264, 237, 246, 214, 201.

Compound 2 : mp – 138^{0}C IR bands (KBr : 3450, 1718, 1510, 1375 cm^{-1}, ^{1}H NMR (CDCl$_3$, 200 MHz): δ 0.67 – 1.20 (each 6x Me) 0.90 (d, 3H, J 6.4 Hz, Me – 30) 2.55 (brs 1H, 18-β-H) 3.64 (s. 3H-COOMe), 3.0 – 3.7 (m sugar proton), MS 636 (M$^+$) 279, 261, 240, 218, 179, 146, ^{13} CNMR CDCl$_3$, 200 MHz Table 1.

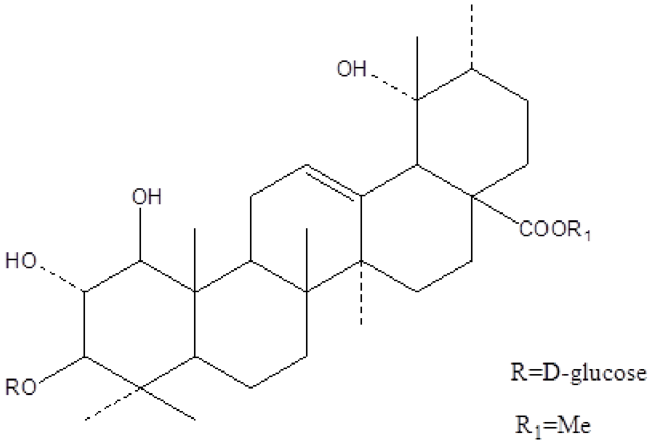
Table 1 ^{13}CNMR spectral data of compound 1 and 2 (200 MHz δ in ppm from TMS).

C	1	DEPT	2	DEPT
1.	68.0	CH	68.2	CH
2.	210.7	C	71.0	CH
3.	83.2	CH	88.8	CH
4.	45.6	C	37.6	C
5.	54.7	CH	56.0	CH
6.	19.0	CH_2	18.2	CH_2
7.	33.0	CH_2	33.6	CH_2

8.	40.5	C	40.5	C
9.	47.2	CH	47.9	CH
10.	43.5	C	38.3	C
11.	23.7	CH_2	24.4	CH_2
12.	127.6	CH	128.2	CH
13.	139.3	C	139.2	C
14.	42.0	C	42.4	C
15.	29.9	CH_2	29.1	CH_2
16.	25.8	CH_2	26.6	CH_2
17.	48.6	C	48.5	C
18.	54.3	CH	54.5	CH
19.	72.4	C	72.5	C
20.	42.2	CH	42.0	CH
21.	26.4	CH_2	26.1	CH_2
22.	37.4	CH_2	37.5	CH_2
23.	29.5	CH_3	28.7	CH_3
24.	17.2	CH_3	17.6	CH_3
25.	16.3	CH_3	16.7	CH_3
26.	16.7	CH_3	17.2	CH_3
27.	24.5	CH_3	24.5	CH_3
28.	178.9	C	176.5	C
29.	26.7	CH_3	26.9	CH_3
30.	16.3	CH_3	15.5	CH_3
COOMe			53.5	CH_3



COMPOUND 1



COMPOUND-2

Sugar moieties

	glc-28		glc-3	
1.	95.6	CH	108.1	CH
2.	73.8	CH	73.9	CH
3.	78.6	CH	78.6	CH
4.	71.1	CH	72.0	CH
5.	78.9	CH	79.1	CH
6.	62.2	CH_2	62.3	CH_2

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