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Research Article

Phytoconstituents: Isolation and Characterization from Root Bark of *Shorea robusta* Plant

*Sharma T. C., Sharma A. K., Sharma K. K., Payal P., Sharma M. C., Dobhal M. P.

Natural Products Laboratory, Department of Chemistry, University of Rajasthan, Jaipur-302004, India

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ABSTRACT

Phytochemical examination on the petroleum ether extract of *Shorea robusta* root bark led to the isolation of Asiatic acid (1), 3,25-epoxy-1,2,3,11-etrahydroxyurs-12-en-28-oic Acid (2), 3,25-epoxy-1,2,3-trihydroxyurs-l2-en-28-oic Acid (3), Phayomphenol (4) and 3,7-dihydroxy-8-methoxyflavone 7-O-α-L-rhamnopyranosyl-(1→4)-α-L-rhamnopyranosyl-(1→6)-β-D-glucopyranoside (5). The structure of these compounds was elucidated on the basis of different spectroscopic techniques.

Keywords: *Shorea robusta*, Resin, *Shorea* genus, Biological activity, Natural compounds, Phytochemicals,

INTRODUCTION

The Dipterocarpaceae is a relatively large family of tropical plants consisting of 16 genera and approximately 600 species[1]. *Shorea* is the largest and economically most important genus of this family out of which 59 species are found in Peninsular Malaysia and nearly 167 species in Borneo and Sumatra. This genus is widely distributed in the Southeast Asia region, especially in Malaysia and Indonesia[2]. The plants belonging to this family have been known to produce oligostilbenoid compounds[3-5]. They include di-, tri-, tetra-, hexa-, hepta- and octastilbenoids, containing various molecular frameworks as a result of different condensation of the resveratrol monomer. Some of these compounds show interesting biological activities, such as antibacterial, antiviral and cytotoxic[4,5]. *Shorea robusta* Gaertn., commonly known as 'Saku' in Hindi is distributed in forest areas over a large part of the country. Its bark is used for the treatment of ulcers, wounds and piles[6]. Flowers are good source of honey and fruits which are used in diarrhea, leprosy and gonorrhea and the seed oil is used as good remedy for skin diseases and scabies[6-8]. The man-made or natural cuts in the bark of stem or branches lead to the exudation of a resin which is trivially called 'saal ki raal, it is produced in large quantities in India and constitutes one of the resins of commerce. It occurs in rough, brittle pieces having a faint resinous, balsamic odour and is widely used as incense in Indian religious ceremonies as on burning it emits copious white fumes. This resin is widely used in the indigenous system of medicine as an astringent and an ingredient in ointments for skin diseases and in ear troubles[9]. Earlier work on this resin reported the isolation of several known triterpenoids[10]. Also, the resin is used for domestic as well as economic purpose such as varnish glues, torch fuel, detergent, medicine for skin diseases, dysentery, astringent, gonorrhea, in ear troubles and cosmetics[11-13]. Along with Bee wax it act as an ointment base for foot cracks, psoriasis, wounds, ulcers, burns, chronic skin diseases and ear and eye troubles while seeds are used for pus forming wounds[14]. A combination of oleoresin with cow ghee is claimed to control burning sensation of hemorrhoids, pain and swelling[15]. A recent study with methanol extract of mature leaves reported anti-inflammatory and antinociceptive activities[16].

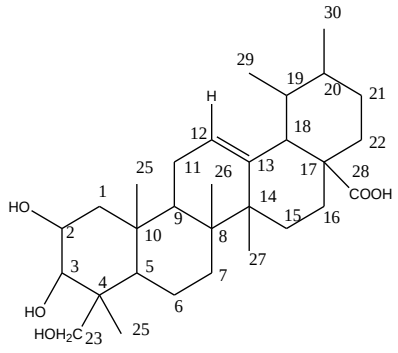
EXPERIMENTAL

General Experimental Procedure**:** Melting points were determined in soft glass capillaries in an electrothermal melting point apparatus. Qualitative TLC was conducted on aluminium sheet Kieselgel 60 F254 (E. Merck). Silica gel (E. Merck, 60-120 mesh, 500 gm) used for column (1.0m × 4.0cm) chromatography. The IR spectra were recorded on FTIR SHIMADZU 8400S spectrometer with KBr pellets. The ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ at 300 MHz and 75 MHz on a Brucker NMR instrument, respectively, using TMS as internal standard. FAB mass spectra were recorded on JEOL SX 102 /DA-6000 mass spectrometer using Argon /Xenon as FAB gas.

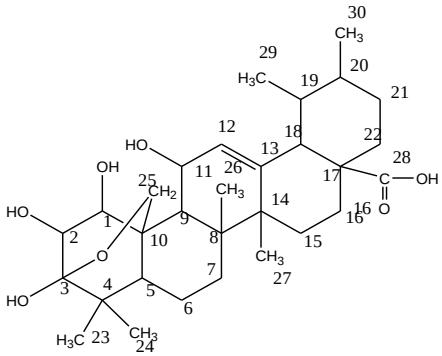
Plant material: The plant material *Shorea robusta* (Part: Root bark) was collected from Uttarakhand (India) and the authenticity of the plant was confirmed by Prof. N. J. Sarna, Department of Botany, University of Rajasthan, Jaipur.

Extraction and Isolation of the Constituents: The shade dried root bark (6 kg) was finely powdered and extracted with petroleum ether for 12×3 hrs on water bath. The extract was filtered and solvent was removed under reduced pressure. To avoid unwanted fat, the extract was treated with acetonitrile and aliquot of acetonitrile phase was transferred into a centrifuge tube and stored in a freezer for two days. Where, the major part of fat (40g) was precipitated. The precipitate was separated and dried. Acetonitrile phase was also combined filtered and evaporated to dryness where a semi-solid, yellowish mass

*Author for correspondence



Compound 1



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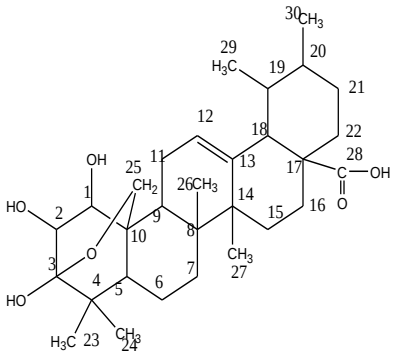
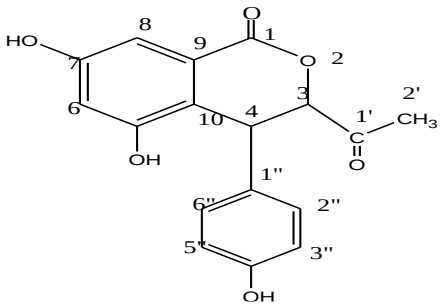
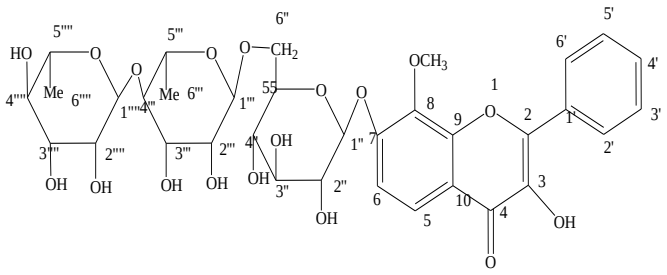


Compound 3



Compound 4



Compound 5

(30g) was obtained. Fat free extract after removal of solvent was chromatographed over silica gel column which afforded five compounds (1 to 5). For this purpose, a column of 1.0 m in height with 4 cm in diameter was used and it was charged with 500 g silica gel for column (60-120 mesh) chromatography. The column was eluted with different solvents in order of increasing polarity where following compounds were isolated, purified and characterized.

Asiatic acid **(1)** : Compound 1 was obtained when column was eluted with petroleum ether. After removal of solvent, a white solid mass was obtained which on crystallization with ethyl acetate yielded shiny white crystals. It showed single spot on TLC examination (R_f = 0.35) in benzene as a mobile phase. The melting point of this compound was found to be 322-324°C. IR (KBr, cm^{-1}) 3414 (-OH stretching), 2927 (-OH streatching in -CO<u>OH</u>), 1695 (-CO streatching in -<u>CO</u>OH), 1455(C=C streatching), 1383, 1247 and 1046; ^{1}H NMR (δppm, $CDCl_3$) 0.71 (3H, s, H-24), 0.83 (3H, s, H-26), 0.89 (3H, d, *J=6.5* Hz, H-29), 0.96 (3H, d, *J=6.0* Hz, H-30), 1.06 (3H, s, H-25), 1.15 (3H, s, H-27), 1.35 (2H, s,-C<u>H</u>$_2$OH), 2.01 (2H, d, H-11), 3.35 (1H, dt, *J=9.6* Hz, H-2), 3.70 (1H, d, H-3), 5.25 (1H, t, H-12), 10.98 (1H, S, -COO<u>H</u>), 1.21-2.20 (m, for remaining 22 protons); ^{13}C NMR (δppm, $CDCl_3$) 181.7 (C-28), 139.9 (C-13), 126.7 (C-12), 78.3 (C-3), 69.7 (C-2), 66.3(C-23), 54.4 (C-18), 48.9 (C-5 and C-17), 48.4 (C-1), 48.3 (C-9), 44.2 (C-4), 43.4 (C-14), 40.8 (C-8), 40.5 (C-19 and C-20), 39.0 (C-10), 38.2 (C-22), 33.7 (C-7), 31.8 (C-21), 29.2 (C-15), 25.4 (C-16), 24.2 (C-11 and C-27), 21.6 (C-30),19.1 (C-6), 17.9 (C-26), 17.7 (C-25 and C-29), 14.4 (C-24). MS(m/z) 487.27 $[M-H]^+$; Molecular formula calculated as $C_{30}H_{48}O_5$.

3,25-epoxy-1,2,3,11-etrahydroxyurs-12-en-28-oic acid **(2)** : A white solid mass was obtained when column was eluted with petroleum ether and benzene in ratio 3:1. After removal of the solvent it was redissolved in acetone and crystallized. Its R_f value was found to be 0.73 in benzene and chloroform (8:2) as a solvent system. IR (KBr, cm^{-1}) 3600-3200 (OH, COOH), 3000-2910, 1693 (COOH), 1645 (C=C), 1120, 1050 (C-O-C); ^{1}H NMR (δppm, $CDCl_3$) 10.98 (s, 1H, -COO<u>H</u>), 6.01 (d, 1H, H-12), 3.98 (dd, 1H, H-11), 3.52 (d, 2H, H-25), 1.25 (s, 3H, H-23), 1.16 (s, 3H, H-24), 1.07 (s, 3H, H-29), 1.03 (s, 3H, H-30), 0.98 (s, 3H, H-27), 0.71 (s, 3H, H-26), 1.32-2.41 (m, remaining 23 protons); ^{13}C NMR (δppm, $CDCl_3$) 180.3 (C-28), 149.0 (C-13), 122.8 (C-12), 112.1 (C-3), 78.7 (C-2), 70.3 (C-1), 65.1 (C-11), 62.5 (C-25), 45.1 (C-14), 44.5 (C-17), 41.7 (C-9), 40.5 (C-18), 39.0 (C-5), 36.5 (C-4), 34.0 (C-8), 31.9 (C-20), 31.2 (C-15), 30.2 (C-7), 29.5 (C-10), 29.5 (C-16), 28.9 (C-19), 26.9 (C-21), 26.0 (C-22), 19.0 (C-6), 18.8 (C-26), 18.1 (C-27), 17.7 (C-30) 15.8 (C-29), 13.2 (C-23), 12.6 (C-24). MS(m/z) 518 $[M]^+$. Molecular formula calculated as $C_{30}H_{46}O_7$.

3,25-epoxy-1,2,3-trihydroxyurs-l2-en-28-oic Acid **(3)** : Compound 3 was obtained when column was eluted with petroleum ether and benzene in the ratio of 1: 1. After removal of solvent colorless viscous mass was obtained. It showed single spot on TLC examination (R_f = 0.86) in benzene and chloroform (7: 3) as a solvent system. IR (KBr, cm^{-1}) 3600-3200 (OH, CO<u>OH</u>), 3000-2900, 1747, 1710 (COOH), 1645 (C=C), 1235, 1110, 1044 (C-O-C); ^{1}H NMR (δppm, $CDCl_3$) 11.9 (s, 1H, -COO<u>H</u>), 5.93 (t, 1H, H-12), 3.13 (d, 2H, H-25), 2.62 (dd, 2H, H-11), 1.13 (s, 3H, H-24), 1.11 (s, 3H, H-23), 1.06 (s, 3H, H-29), 1.03 (s, 3H, H-30), 0.96 (s, 3H, H-27), 0.73 (s, 3H, H-26), 1.30-2.39 (m, remaining 22 protons); ^{13}C NMR (δppm, $CDCl_3$) 183.9 (C-28), 148.1 (C-13), 123.7 (C-12), 113.2 (C-3), 77.2 (C-2), 68.0 (C-1), 60.8 (C-25), 45.3 (C-14), 43.3 (C-17), 41.7 (C-18), 39.6 (C-8), 39.3 (C-5), 37.8 (C-9), 37.6 (C-4), 32.8 (C-15), 32.4 (C-10), 30.3 (C-20), 29.1 (C-16), 28.4 (C-7), 27.4 (C-21), 27.3 (C-19), 26.3 (C-11), 24.4 (C-23), 24.0 (C-22), 19.8 (C-27), 19.6 (C-26), 18.4 (C-6), 15.6

(C-30), 14.2 (C-29), 12.4 (C-23), 11.9 (C-24). MS(m/z) 502 $[M]^+$. Molecular formula calculated as $C_{30}H_{46}O_6$.

Phayomphenol (4): It was isolated as white powder when elution of column by petroleum ether with benzene in the ratio 1: 3 and it showed single spot on TLC examination. Its R_f value was observed at 0.63 (benzene as mobile phase). IR (KBr, cm^{-1}) 3482 (-OH, stretching), 1728 (C=O, COOH), 1692, 1613, 1514, 1468, 1352, 1196, 1125 (-C-O-C-); ^{1}H NMR (δppm, $CDCl_3$) 6.99 (1H, *d*, H-8), 6.96 (2H, *d*, H-2'',6''), 6.71 (2H, *d*, H-3'',5''), 6.55 (1H, *s*, H-6), 5.28 (1H, *d*, H-3), 4.80 (1H, *br s*, H-4), 2.23 (3H, *s*, H-2'); ^{13}C NMR (δppm, $CDCl_3$) 205.4 (C-1'), 166.7 (C-1), 159.3 (C-7), 157.6 (C-4'') 156.7 (C-5), 132.4 (C-1''), 129.8 (C-2'',6''), 127.8(C-9), 117.5 (C-10), 116.3 (C-3'',5''), 109.2 (C-6), 107.5 (C-8), 89.1 (C-3), 38.5 (C-4), 26.0 (C-2'). MS(m/z) 337 $[M\text{-}Na]^+$. Molecular formula calculated as $C_{17}H_{14}O_6$.

3,7-dihydroxy-8-methoxyflavone 7-O-α-L-rhamnopyranosyl-(1→4)-α-L-rhamnopyranosyl-(1→6)-β-D-glucopyranoside **(5)** : This compound was isolated when column was eluted with benzene. It showed single spot on TLC examination (R_f = 0.72) in benzene and chloroform (7: 3) as a solvent system. Melting point of this compound (Pale Yellow solid) was found to be 238°C. IR (KBr, cm^{-1}) 3278 (-OH stretching), 2842, 1680 (C=O stretching.), 1532 (-C-0-C-); ^{1}HNMR (δppm, $CDCl_3$) 9.6 (1H, *s*, OH-3), 8.5 (2H, *m*, H-2',6'), 7.5 (3H, m, H-3', H-4', H-5'), 7.4 (1H, *d*, *J* = 9.5 Hz, H-5), 7.1(1H, *d*, J = 9.2 Hz, H-6), 3.8 (3H, *s*, OMe-8), 1.05 (3H, *s*, $-CH_3$), 4.4-5.3 (*m*, remaining protons of rhamnose rings); ^{13}CNMR (δppm, $CDCl_3$) 177.5 (C-4), 165.1 (C-7), 156.7 (C-2), 156.4 (C-9), 136.2 (C-3), 133.6 (C-2'), 133.4 (C-6'), 125.4 (C-8), 120.80 (C-1'), 115.8 (C-3'), 114.1 (C-4'), 113.4 (C-5'), 104.9 (C-10), 102.28 (C-1''), 102.15 (C-1'''), 100.21 (C-1''''), 100.6 (C-5), 98.3 (C-6), 78.15 (C-3''), 73.8 (C-5''), 73.5 (C-2''), 71.15 (C-4''), 70.90 (C-2'''), 70.50 (C-3'''), 70.4 (C-5'''), 70.00 (C-2''''), 68.53 (C-3''''), 68.15 (C-5''''), 66.6 (C-6''), 60.24 (C-4''''), 56.0 (OMe), 17.7 (C-6'''), 17.6 (C-6''''). MS(m/z) 738 $[M]^+$. Molecular formula calculated as $C_{34}H_{42}O_{18}$.

RESULTS AND DISCUSSION

Compound 1 *(Asiatic acid) [(4α)-2α, 3β, 23-Trihydroxy-urs-12-en-28-oic acid)]*: In the mass spectrum of compound 1, prominent signal was observed at m/z 487.27 $[M^+{+}H]$. The ^{1}H NMR and ^{13}C NMR spectrum indicated that it contain forty eight protons and thirty carbon atoms in the skeleton. On the basis of these observations the molecular formula of compound 1 was calculated as $C_{30}H_{48}O_5$. The IR spectrum (cm^{-1}, KBr) shows strong absorption at 3414 (br), indicated the presence of hydroxyl group. Absorption at 2927 confirmed the presence of –COOH group. The absorption at 1695 was due to carbonyl group whereas, the absorption at 1455 confirmed the presence of olefinic (>C=C<) stretching. In the ^{1}H NMR spectrum (δppm, $CDCl_3$) presence of six singlets at 1.15, 1.06, 0.96, 0.89, 0.83 and 0.71 for three protons each were assigned to methyl groups attached at position C-27, C-25, C-30, C-29 C-26 and C-24, respectively. A sharp singlet was observed at 10.98 and assigned to –COOH group. A triplet observed at 5.25 was attributed to olefinic proton at C-12 position. Oxygenated proton at C-3 and C-2 afforded a doublet and a double triplet at 3.70 and 3.35. A doublet observed at 2.01 was due to the presence of two protons at C-11 position. A sharp singlet for two protons at 1.35 confirmed the presence of hydroxymethyl group CH_2OH. The remaining 22 protons were appeared as multiplates in the range of 1.21-2.20. In ^{13}C NMR (δppm, $CDCl_3$) spectrum the peak observed at 181.07 confirmed the presence of –COOH group. The absorptions observed at 139.9 and 126.7 were due to the presence of olefinic carbons (C-13 and C-12). The absorptions at 69.7 and 78.3 showed the presence of two hydroxyl groups in the molecule and their position was assigned as C-2 and C-3 positions, respectively. The signals appeared at 24.2, 21.6, 17.9, 17.7, 17.5 and 14.4 were assigned for six methyl groups attached at C-27, C-30, C-26, C-29, C-25 and C-24 respectively. The remaining signals appeared at 66.3, 54.4, 48.9, 48.9, 48.4, 48.3, 40.8, 40.5, 40.5, 38.2, 33.7, 31.8, 29.2, 25.4, 24.2 and19.1 and assigned to C-23, C-18, C-5, C-17, C-2, C-9, C-8, C-19, C-20, C-22, C-7, C-21, C-15, C-16, C-11 and C-6, respectively. On the basis of above observation and discussion compound 1 was identified as Asiatic acid ((4α)-2α, 3β, 23-trihydroxy-urs-12-en-28-oic acid)[17,18].

Compound 2 *(3,25-Epoxy-1,2,3,11-tetrahydroxyurs-12-en-28-oic acid)*: The mass spectrum of compound 2 showed a molecular ion peak at m/z 518$[M]^+$ and the molecular formula was determined as $C_{30}H_{46}O_7$ with the help of ^{1}H NMR and ^{13}C NMR spectral analysis. The IR spectrum (cm^{-1}, KBr) of compound 2 confirmed the presence of hydroxy group/groups by showing a broad absorption between 3600 - 3200. The presence of carboxyl acid group was established by the absorption at 3000 - 2910. An absorption at 1693 confirmed the presence of C=0 group of -COOH. An absorption at 1645 confirmed the presence of unsaturation (>C=C<) in the molecue. The ^{1}H NMR spectrum (δppm, $CDCl_3$) showed a sharp singlet for one proton of- COOH group at 10.98. Presence of a doublet at 6.01 was assigned to olefinic proton at the position C-12. The double doublet observed at 3.98 was assigned to one proton attached at C-11. Presence of a doublet at 3.52 was assigned for two protons which are attached to epoxy oxygen atom ($-O-CH_2-$) at C-25. Presence of six sharp singlets at 1.25, 1.16, 1.07, 1.03, 0.98 and 0.71 for three protons each, were assigned for methyl group at position C-23, C-24, C-29, C-30, C-27 and C-26, respectively. The remaining 23 protons were appeared as multiplates in the range of 1.32-2.41. The ^{13}C NMR spectrum (δppm, $CDCl_3$) showed absorption at 180.3 due to presence of C=O group of –COOH (C-28). The peaks were observed at 149.0 and 122.8 due to the presence of olefinic carbons >C=C< (C-13 and C-12). Absorptions were observed at 112.1, 78.7, and 70.3 and 65.1 due to carbons attached with hydroxyl groups at C-3, C-2, C-1 and C-11, respectively. The carbon atom (C-25) attached to epoxy oxygen atom ($-O-CH_2-$) showed absorption at 62.5. The signal at 45.1 was confirmed for C-14.The absorptions at 44.5, 41.7, 40.5, 39.0, 36.5, and 34.0 confirmed the position of C-17, C-9, C-18, C-5, C-4 and

C-8, respectively[17]. The absorptions at 29.5 confirmed the position of (C-21) and (C-16). Absorption were observed at 28.9, 26.9, 26, and 19.0 were assigned for (C-19), (C-21), (C-22) and (C-6), respectively. The absorption at 18.8 indicates the presence of methyl group attached at C-26. On the basis of the above spectral studies the structure for compound 2 was established as 3,25-epoxy-1,2,3,11-tetrahydroxyurs-12-en-28-oic acid.

Compound 3 *(3,25-epoxy-1,2,3-trihydroxyurs-l2-en-28-oic acid)*: The mass spectrum of compound 3 exhibited the molecular ion peak at 502 [M^+]. On the basis of spectral analysis the molecular formula was calculated as $C_{30}H_{46}O_6$. The IR spectrum (cm^{-1}, KBr) displayed the significant broad absorption at 3600-3200 for hydroxyl group and 3000-2900 and 1747 for carboxylic group. The unsaturation in the molecule was confirmed as the absorption at 1645. Other signals at 1235, 1110, 1044 gives evidence of -C-O-C- linkage On the basis of these observations, compound C seems to be a unsaturated aliphatic acid having epoxy group with multi ring system. The ^{1}H NMR spectrum (δppm, $CDCl_3$) showed a sharp singlet at 11.90 which corresponded to proton of –COOH group. A triplet was observed at 5.93 confirmed the presence of an olefinic proton. A doublet was observed at 3.13 due to two protons of C-25. Presence of a double doublet at 2 .62 was assigned as the two protons at C-11 position. Sharp singlets at 1.13, 1.11, 1.06, 1.03, 0.96 and 0.73 for three protons each were assigned for six methyl groups present at position C-24, C-23, C-29, C-30, C-27 and C-26, respectively. The presence of remaining twenty two protons was observed as multipletes from 1.30-2.39 in the spectrum. In the ^{13}C NMR spectrum (δppm, $CDCl_3$), the presence of carboxyl group (–COOH) was confirmed by the absorption at 183.9. Signals at 148.1 and 123.7 were assigned as olefinic carbons i.e. C-13 and C-12 respectively. The signals observed at 113.2, 77.2 and 68.0 was assigned to the carbons attached with -OH group C-3, C-2 and C-1, respectively. The carbon atom (C-25) attached to epoxy oxygen atom (-O-CH_2-) showed a absorption at 60.8. The absorptions at 43.3, 41.7, 39.6, 39.3, 37.8, 37.6, 32.8 and 32.4 confirmed the position of C-17, C-18, C-8, C-5, C-9, C-4, C-15 and C-10, respectively. C-20, C-16 and C-7 positions were confirmed by the absorptions at 30.3, 29.1 and 28.4, respectively. On the basis of the above spectral studies the structure for compound 3 was established as 3,25-epoxy-1,2,3-trihydroxyurs-l2-en-28-oic acid[17].

Compound 4 *(Phayomphenol)*: The mass spectrum of compound 4 displayed [M^++H] at 337 and [M^+] at m/z 336. In the IR spectrum [cm^{-1}, KBr] characteristic strong absorption was observed at 3482 suggested the presence of a hydroxy group. Signal observed at 1728 confirmed the presence of carbonyl group. The signal observed at 1125 strongly indicates the presence of -C-O-C- linkage. The ^{1}H NMR spectrum [δppm, $CDCl_3$], the signals at 6.99 (*s*, 1H), 6.96 (*d*, 2H), 6.71 (*d*, 2H) and 6.55 (*s*, 1H) were observed for C-8, (C-2", C-6"), (C-3", C-5") and C-6 protons, respectively. The signals at 5.28 (*d*, 1H) was assigned for C-3 proton. A broad singlet was observed at 4.80 due to C-4 proton. A singlet was observed at 2.23 which assigned to methyl protons of acetyl group C-2'. In ^{13}C NMR spectrum [δppm, $CDCl_3$] an absorptions at 205.4 and 166.7 were confirmed the presence of two ketonic groups at C-1' and C-1 position, respectively. Other absorptions at 159.3, 157.6 and 156.7 were due to three hydroxyl groups and their positions were assigned as C-7, C-4" and C-5, respectively. The assignments of olefinic carbon atoms and their position were established as 132.4 (C-1"), 129.8 (C-2"and C-6"), 127.8 (C-9), 117.5 (C-10), 116.3 (C-3" and C-5") 109.2 (C-6), 107.5 (C-8) by comparing the data with the literature values. Signals at 89.1 and 38.5 were observed corresponding to C-3 and C-4, respectively. Thus position of all seventeen carbons was established on the behalf of ^{13}C NMR spectrum of the compound and the identity of the title compound was further confirmed by comparing the spectral data with reported values[19,20]. On the basis of above spectral analysis compound 4 was characterized as Phayomphenol.

Compound 5 *(3,7-dihydroxy-8-methoxyflavone 7-O-α-L-rhamnopyranosyl-(1→4)-α-L-rhamnopyranosyl-(1→6)-β-D-glucopyranoside):* The molecular formula of compound 5 was found to be $C_{34}H_{42}O_{18}$ by mass spectral studies 738 [$M]^+$. The IR spectrum [cm^{-1}, KBr] showed absorption peak at 3278 which was assigned for -OH group (C-3). A signal at 2842 was assigned as methoxy group (C-8). An intense peak was observed at 1680 due to presence of carbonyl group (C-4). Absorption at 1532 was interpreted as double bond system in Skelton. Other complicated signals were assigned as presence of rhamnose and pyranose like rings. The ^{1}H NMR spectrums (δppm, $CDCl_3$) showed a sharp singlet for hydroxyl group at 9.6 (*s*, 1H, C-3). A multiplet was observed at 8.5 due to two protons, one each at C-2'and C-6'. The position of H-3', H-4' and H-5' was assigned by the multiplate at 7.5 corresponding to three protons one on each carbon. The signals at 7.4 (1H, d, *J = 9.5* Hz, H-5) and 7.1 were assigned for C-5 and C-6 positions, respectively. In the range 4.4-5.3 a multiplet pattern was observed to rhamnose ring wich was attached at (C-7) position. In ^{13}C NMR spectrum [δppm, $CDCl_3$] an absorptions at 177.5 was assigned to (C-4) of ketonic group. The position of aromatic carbon (C-7) was confirmed due to peak at 165.1. Double bonded carbons were indicated at 156.7 and 136.2 for (C-2) and (C-3) respectively. ^{13}C NMR values at 133.4 (C-6'), 113.4(C-5'), 114.1(C-4'), 115.8 (C-3'), 133.6 (C-2'), 120.8 (C-1') were assigned for Aromatic skelton attached to (C-2). Aromatic carbons were observed at 104.9 (C-10), 100.6 (c-5), 98.3 (C-6), 165.1 (C-7), 125.4 (C-8), 156.4 (C-9). A owerlapping set of singlets in the region of δ 78.15 to 17.6 belongs to the carbons of rhamnose ring. On the basis of above spectral studies compound 5 was 3,7-dihydroxy-8-methoxyflavone 7-O-α-L-rhamnopyranosyl-(1→4)-α- L-rhamnopyranosyl-(1→6)-β-D-glucopyranoside[21].

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