Chapter 5

HIBISCUS ROSA-SINENSIS L.

REVIEW OF LITERATURE
**HIBISCUS ROSA-SINENSIS LINN. (MALVACEAE)**

Introduction

*Hibiscus* Linn. is a large genus of herbs, shrubs and trees widely distributed in the tropical and subtropical regions of the world. Out of 160 species about 40 occur in India. Many Hibiscus species are valued as ornamental plants and are cultivated in gardens. Some yield fibre.

*H. cannabinus* and *H. sabdariffa* are important sources of commercial fibre. Some species are useful as food: Yet others are medicinal.

Vernacular names

Sanskrit - Japa, Java, Rudra pushpam.
Hindi - Jasut, Jasun, Gudhal
Bengali - Joba
Marathi - Dasindacha phula, Jasavanda
Gujarati - Jasuva
Telugu - Java pushapamu, Dasana
Tamil - Separuthi
Kannada - Dasavala
Malayalam - Chembarathi
Oriya - Mondaro
Assami - Joba
Punjabi - Jasum

Distribution

*H. rosa-sinensis* is cultivated all over India as an ornamental plant. Probably it is a native of China.
Plate 5.1: Plant of *Hibiscus rosa-sinensis* L.

Plate 5.2: Leaves of *Hibiscus rosa-sinensis* L.
Morphology

Arborescent; stem without prickles. Leaves short-petioled, ovate or ovate-lanceolate, more or less acuminate, irregularly and coarsely serrate towards the top, entire near the base, glabrous on both sides or with a few minute stellate hairs on the nerves beneath; stipules lanceolate-subulate, glabrous. Pedicels axillary, solitary very long, as long as, or longer than the leaves, jointed above the middle. Involucral bracts 5-7, about half as long as the calyx, lanceolate, glabrous. Calyx divided almost to the middle, purberulous with very minute stellate hairs, lobes 2 cm long, lanceolate. Corolla 7.5 cm diam, tubular below, red; petals thrice as long as the calyx. Staminal tube exerted far beyond the petals.

An extremely variable species. There are single and double forms and so as to colour the flowers may be orange, yellow, crimson, bright red, magenta, and parti-cloured. - No fruits produced in India (Kirtikar and Basu, 1993).

Description

It is grown as an ornamental plant in gardens throughout India and often planted as a hedge or fence plant. It can be cultivated with advantage in group planting of shrubs or for beautifying parks and grassy plots. Numerous types adopt to sunny, semi-shady and shady locations and with single and double flowers of red, yellow, white, magenta and cherry colours.

The plant thrives in any type of soil, but good results are obtained in well prepared, manured and irrigated soils. It can be propagated by cuttings, preferably from mature wood of current growth. It blossoms almost throughout the year and seldom sets seeds under cultivation.
Chemical Constituents

A number of compounds have been reported from *H. rosa-sinensis*. Vitamins, thiamine, riboflavin, niacin and ascorbic acid have been reported from its flowers (Intengan et al., 1955). Crushed red and magenta flower varieties yield dark-purplish dye. An anthocyanin pigment, cyanidin diglucoside, is also reported in its flowers. (Wealth of India, 1997). Deep yellow flowers of its variety furnished quercetin-3-diglucoside, quercetin 3,7-diglucoside, cyanidin-3,5-diglucoside and cyanidin-3-sophoroside-5-glucoside where as ivory white flowers of its variety afforded kaempferol-3-xylosyglucoside in addition to above mentioned compounds (Subramanian and Nair, 1972), two cyclopeptide alkaloids I & II isolated from flowers of Pakistani plant *H. rosa sinensis* (Khokhar et al., 1992), a sterol, β-rosasterol has been isolated from Chinese species (*H. rosa sinensis*) (Yu et al., 1991).

The leaves contain carotene and two compounds namely taraxeryl acetate and β-sitosterol were isolated from leaves and stem bark of the plant (Aggarwal and Rastogi, 1971). An acidic polysaccharide, composed of rhamnose, glucose, galacturonic acid and glucuronic acid in molar ratio of 5.0: 8.0: 3.0: 2.0 respectively, reported in the mucilage called Hibiscus mucilage RL, was isolated from the leaves of *Hibiscus rosa sinensis* (Shimizu et al., 1993).

8-Nonynoic and 9-decynoic acids and their methyl esters were isolated and synthesized from stem bark (Nakatani et al., 1985).

The aliphatic esters, namely methyl 10-oxo-11-octadecynoate, methyl 8-oxo-9-octadecynoate, and methyl 9-methylene-8-oxoheptadecanoate, were
isolated from stem bark (Nakatani et al., 1986). Methyl (R) 2-hydroxysterculcate along with methyl sterculate and methyl malvalate, isolated from roots, bark (Nakatani et al., 1991). Two aliphatic enone ethers isolated from roots and characterized as methyl (E) 11-methoxy 9-oxo-10-nonadecenoate and (E) 10-methoxy-8-oxo-9-octadecenoate along with three cyclopropenoids, methyl sterculate, methyl malvalate and methyl (R)-2-hydroxysterculcate (Nakatani et al., 1994). Campesterol, sitosterol, stigmasterol and a sterol ester have been reported from this plant (Chouhan and Shukla, 1984).

The isolated compounds, namely 8-nonynoic and 9-decynoic acids and their esters, methyl esters of 10-oxo-11-octadecynoic, 8-oxo-9-octadecynoic acids from this plant have been reported for their inhibitory action on lettuce seeds germination. 8-Nonynoic, 9-decynoic acids and their methyl esters showed 50% inhibition at 1mg/ml of water whereas methyl esters of 10-oxo-11-octadecynoic and 8-oxo-9-octadecynoic acids showed moderate inhibitory activity on lettuce seed germination when compared with a well known acetylenic growth inhibitors, cis-dehydromatricaric acid. Reynolds (1977, 1978) showed that inhibitory activity increased with carbon chain length and the main structure - activity correlation was with lipophilic properties. Increased lipophilicity usually led to increased inhibitory activity, thus methyl esters showed increased activities (Nakatani et al., 1985; 1986).
Me(CH₂)₇C === C(CH₂)₇COOH

Sterculic acid

Me(CH₂)₇C === C(CH₂)₆COOH

Malvalic acid

Me-(CH₂)₇C === C-(CH₂)₇CO₂Me

Methyl sterculate

Me-(CH₂)₇C === C-(CH₂)₆CO₂Me

Methyl malvalate

Me-(CH₂)₇C === C-(CH₂)₆CH(OH)CO₂Me

Methyl (R) 2-hydroxy sterculate

I  Me(CH₂)₅C === CCO(CH₂)₆COOMe

Methyl 10-oxo-11-octadecynoate

II  Me(CH₂)₇C === CCO(CH₂)₆COOMe

Methyl 8-oxo-9-octadecynoate

III  Me(CH₂)₇C(=CH₂)CO(CH₂)₆COOMe

Methyl 9-methylene-8-oxo hepta decanoate

IV  Me(CH₂)₇C(=CH₂)CO(CH₂)₇COOMe

Methyl 10-methylene-9-oxo octa decanoate
Cyclo peptide alkaloids

I. $R = H, R' = Me$

II. $R = Me, R' = H$

Chemical constituents reported from *Hibiscus rosa sinensis*
Medicinal uses

The flowers are considered as demulcent, emollient, refrigerant, aphrodisiac and emmenagogue. Paste of the flowers are applied to swellings and boils. A decoction of the flowers is given in bronchial catarrh. Ghee fried flowers are beneficial in menorrhagia. Crushed flowers yield a dark purplish dye which was formally employed for blackening shoes. In China, the dye is used for colouring hair, eyebrows, foods and liquors (Kirtikar and Basu, 1993; Wealth of India, 1997).

In Indian medicinal system the flowers of H. rosa-sinensis have been quoted for their antifertility efficacy and to treat menstrual disorders (Kholkute et al., 1976; 1977). The flower buds are prescribed in the treatment of vaginal and uterine discharges, whereas stalk extract is applied to sore eyes.

Alcoholic (50%) extract of aerial parts showed antispasmodic action on isolated smooth muscles due to the presence of cholinergic and papaverine-like substances, CNS depressant and hypotensive action, whereas alcoholic extract of leaves displayed antipyretic, analgesic and anti-inflammatory action. The leaves are also emollient, aperient, anodyne, laxative and a decoction of leaves and stem bark is used for abortion. In Malaya, a decoction of the leaves with Vemonia cinerea Less juice is recommended to stimulate expulsion after child birth. Various parts of plant are used in urinary complaints and in hysteria. In Samoa roots and other parts are taken as remedies for gonorrhoea, vomiting of blood and in stomach trouble. Hibiscus-mucilage RL, isolated from the leaves of H. rosa-sinensis, showed considerable anticomplementary activity (Shimizu et al., 1993). Roots are also used in certain cattle's diseases in India. Now a days Hibiscus rosa sinensis is widely used in herbal cosmetics. Aqueous and alcoholic extract of leaves are recommended to colour the hair and beard (Ambasta, 1986). Its aqueous and alcoholic extracts are also used as an antidandruff, antiinfective, prophylactic against skin diseases and in allergic conditions. It also checks hair-loss, stimulates hair growth and darken the hair (Folicon).
GENERAL NOTES ON EXPERIMENTAL WORK

All melting points were determined in centigrade scale in one-end open capillary on Perfit melting point apparatus and are uncorrected.

U.V Spectra were recorded on Shimadzu Beckmann DU - 64 model, spectrophotometer. Values are in nm.

IR spectra were recorded on Hitachi, model - 270.

All optical rotations were recorded on Abbe Polarimeter in chloroform at 28°C.

¹H and ¹³C-NMR Spectra were scanned on Bruker Spectrospin NMR (100, and 300 MHz) instruments in CDC13, DMSO-d6, TFA, D₂O (unless otherwise stated) using tetramethylsilane (TMS) and CDCl₃ as the internal standards. Chemical shifts are expressed in δ ppm with respect to the internal standard and coupling constants (J values) are expressed in Hertz (Hz). Notations used for spin-coupling pattern throughout the manuscripts designated as s = singlet; d = doublet; dd = doublet of doublet; ddd = doublet of double doublet; t = triplet; q = quartet; m = multiplet, brs = unresolved broad singlet; wₓₜ = half - width.

Mass Spectra were recorded by effecting electron impact ionization at 70-ev on a Jeol D - 300 (EI/Ci) mass spectrometer equipped with direct inlet probe system. the m/z values of the more intense peaks are mentioned and the figures in bracket attached to each m/z values indicated relative intensities with respect to the base peak.

The solvents used were of Qualigens AR grade (Hexane, Petroleum-ether 60°-80°, Chloroform, and Methanol.

Silicagel (Quaigens- 60-80 mesh) was used for column chromatography. The column was (75%) filled with petroleum ether, silicagel added in small
portions and allowed to settle down gently to attain necessary length of packing. All the air bubbles were allowed to escape by running the column blank thrice with solvent.

Previously prepared air dried silicagel slurry of the plant extract was packed in the column. The column was eluted with successive series of solvents in various combinations, viz., petroleum ether, petroleum ether: Chloroform (9:1, 3:1, 1:1, 1:3 v/v), Chloroform, and Chloroform: methanol (99.9: 0.1, 99.5: 0.5, 99:1, 98:2, 95:5, 9:1, 3:1 and 1:1 v/v). The completion of elution of the components, was confirmed via evaporating a small fraction of the eluent.

The fractions collected were subjected to thin layer chromatography to check homogeneity of various fractions. Chromatographically identical fractions were combined and concentrated.

TLC was performed on plates coated with Silica gel G (Qualigens). The dried plates were activated in an electric oven at 110° C for two hours. The dissolved fractions were spotted on the plates with the fine capillary tubes and then air dried. The spotted plates were kept in chromatographic chambers containing the solvent mixture and covered with greased glass plates. The TLC plates were visualized under UV light or exposure of plates to iodine vapours or spraying with ceric ammonium sulphate solution in 65% H₂SO₄ followed by keeping at 120° C for 10-15 minutes in an oven.

Anhydrous sodium sulphate was used for drying all the solvents those were used during the research work.

All the drugs used during research work were authenticated by Dr. M.P. Sharma, Reader, Department of Botany, Faculty of Science, Jamia Hamdard, New Delhi.

Well resolved spectral data chart of the compounds are attached.
Chapter 5

**HIBISCUS ROSA-SINENSIS L.**

**EXPERIMENTAL, ISOLATION AND STRUCTURE ELUCIDATION**
Extraction And Isolation Of Chemical Constituents From The Leaves of Hibiscus rosa-sinensis Linn.

Plant Material

The leaves of *H. rosa-sinensis* were collected from the campus, Jamia Hamdard, New Delhi-110062.

Extraction

The air dried and coarsely powdered material (2.045 kg) was exhaustively extracted with EtOH (95%) in a Soxhlet apparatus for 72 hours. The combined extracts were dried under reduced pressure to obtain a dark brownish coloured residue (550 g) (26.89%).

Isolation of Chemical Constituents

The dried ethanolic extracts (375 g) was dissolved in minimum quantity of MeOH and absorbed on silica gel to form a slurry. The slurry was air dried and subjected to silica gel column chromatography. The column was eluted with petroleum ether, petroleum ether - chloroform (99:1, 98:2, 95:5, 9:1, 3:1, 1:1, 1:3) chloroform, chloroform - methanol (9:1, 8:2, 3:1, 1:1) to isolate the following compounds.

*n*-pentacosane (1)

Elution of the column with petroleum ether (fraction 1) furnished colourless amorphous powder of 1, purified by preparative chromatography, 263 mg (0.0128 % yield), mp 58 - 59°, *R*<sub>f</sub> 0.76 (petroleum ether: hexane : chloroform : methanol; 18: 1.5: 1.5: 0.5).

IR *ν*<sub>max</sub> 2920, 2835, 1455, 1370, 1250, 1020, 710 cm<sup>-1</sup>.
\(^1\)H NMR (CDCl\textsubscript{3}) : \(\delta 1.25\) (50H, brs, 25 x CH\textsubscript{2}), 0.88 (3H, t, J = 6.0 Hz, Me -1), 0.85 (3H, t, J = 6.5 Hz, Me -27).

\(^{13}\)C NMR : \(\delta 31.94\) (CH\textsubscript{2}), 29.71 (23 x CH\textsubscript{2}), 29.37 (CH\textsubscript{2}), 22.7 (Me - 27), 14.10 (Me-1).

EI/MS \(m/z\) (rel. int.) 380 [M]\textsuperscript{+} (C\textsubscript{27}H\textsubscript{56}) (6.1), 365 (5.8), 351 (5.6), 337 (55.0), 309 (5.3), 295 (5.0), 281(5.0), 267 (5.0), 253 (5.1), 239 (5.1), 225 (5.2), 211 (5.3), 197 (5.2), 183 (7.9), 169 (8.3), 155 (10.9), 141 (11.0), 127 (15.1), 113 (20.3), 99 (31.5), 85 (51.2), 71 (63.8), 57 (100), 43 (83.6)

D- 3-\textit{epi} - Taraxeryl acetate (2)

Elution of the column with petroleum ether - chloroform (95:5) (fractions 2-10) afforded colourless amorphous powder 2, recrystallized from chloroform : methanol (1:1). 230 mg (0.01125% yield), mp 300 - 301\textdegree, R\textsubscript{f} 0.75 (petroleum ether : hexane : chloroform : methanol; 6:2:2:3)

\([\alpha]\text{D}\textsuperscript{28}(+) 152.6\textdegree \) (C = 0.05, CHCl\textsubscript{3})

UV \(\lambda_{\text{max}} 240\text{ nm (log }\varepsilon 4.5)\)

IR \(\nu_{\text{max}} 2965, 2870, 1730, 1645, 1475, 1450, 1380, 1255, 1110, 1030, 1000, 910, 820\text{ cm}^{-1}\).

\(^1\)H NMR - Table 5.1

\(^{13}\)C NMR - Table 5.1

EI/MS \(m/z\) (rel. int) 468 [M]\textsuperscript{+} (C\textsubscript{32}H\textsubscript{52}O\textsubscript{2}) (20.3), 453 (9.5), 408 (10.2), 393 (8.2), 345 (34.2), 330 (17.2), 286 (16.7), 270 (16.7), 258 (9.1), 250 (5.2), 218 (20.2), 207 (9.9), 206 (34.1), 205 (93.9), 204 (10.1), 190 (22.5), 182 (3.2), 176 (10.6),
Hydrolysis of 2. The acetate 2 (50 mg) was refluxed with 10% ethanolic KOH (20 ml) for 3 hours. The reaction mixture was extracted with chloroform. The chloroform solution, after washing with water and drying over anhydrous Na₂SO₄ was evaporated to yield D-3-epi-taraxerol (2a), mp 310 - 311°. IR ν⁺ 3420, 1640, 890 cm⁻¹. EIMS m/z (rel. int.) 426 [M]⁺ (C₃₀H₄₅O₄).

Oxidation of D-3-epi-taraxerol (2a). Compound (2a) (25 mg) was dissolved in Me₂CO (40 ml) and treated with freshly prepared Jone's reagent (5 ml). The reaction mixture was stirred at room temperature till the reaction was completed (TLC monitoring). It was diluted with water and extracted with Et₂O. Removal of the solvent from the Et₂O extract produced D-3-epi-taraxesone (2b), mp 155°. IR ν⁺ 1705, 1640, 885 cm⁻¹. EIMS m/z 424 [M]⁺ (C₃₀H₄₅O) (5.2).

Reduction of D-3-epi-taraxesone (2b)

The compound (2b) (15 mg) was dissolved in MeOH (5 ml) and NaBH₄ (0.5 mg) was then added with stirring (1 hour). After dilution with water, the mixture was extracted with chloroform and the chloroform layer was washed with water, dried and evaporated to obtain taraxerol (2c), mp and mmp 289 - 290°, Co-TLC comparable.

Taraxeryl acetate (3).

Further elution of the column with petroleum ether-chloroform (95:5) (fractions 11 to 20) furnished colourless mass of 3, recrystallized from chloroform- methanol (1:1) to yield off-white plates, 218 mg (0.0106 % yield), mp 303 - 305°. Rᵣ 0.82 (petroleum ether : hexane : chloroform : methanol; 6:2:2:3).
[α]$_D^{23}$  (+) 10.5° (C =1.5, CHCl$_3$)

UV $\lambda_{max}$  210, 215, 222 nm (log $\varepsilon$ 4.2, 5.3, 5.0)

IR $\nu_{max}$  2950, 2865, 1740, 1390, 1265, 1205, 1195, 1175, 1045, 810,
730 cm$^{-1}$.

$^1$H NMR - Table 5.2

$^{13}$C NMR - Table 5.2

EI-MS $m/z$ (rel. int.) 468[M]$^+$ (C$_{32}$H$_{52}$O$_2$) (25.4), 453 (7.8), 408 (5.2), 393 (5.3),
345 (33.4), 330 (13.9), 286 (6.4), 270 (13.3), 258 (8.7), 218 (21.8), 207 (12.5),
206 (41.4), 205 (68.0), 204 (10.4), 190 (20.2), 176 (12.0), 162 (13.9), 150
(22.1), 136 (35.0), 134 (26.6), 124 (20.9), 122 (30.4), 107 (31.5), 95 (35.8), 81
(30.7), 69 (53.2), 55 (53.9), 43 (100).

Alkaline hydrolysis of 3. Compound 3 (50 mg) was refluxed with 10%
ethanolic KOH (20 ml) for 3 hours. The reaction mixture was neutralized with
dilute HCl and extracted with chloroform. The organic phase was washed
with water, dried over Na$_2$SO$_4$ and evaporated. The residue was crystallized
from chloroform - methanol (1:1) to yield taxaxerol (D - Friedolean - 14-en-
3β-ol) (2c), mp and mmp 283-285°. [M]$^+$ $m/z$ 426 (C$_{30}$H$_{50}$O) (11.6).

Aliphatic alcohol (4)

Elution of the column with petroleum ether-chloroform (9:1)
(fractions 28 -40) afforded colourless amorphous powder of 4, recrystallized
from chloroform - methanol (1:1), 238 mg (0.0116 % yield), mp 74-76°, $R_f$
0.38 (petroleum ether : hexane: chloroform: methanol; 14:4:12:9).

UV $\lambda_{max}$  212 nm (log $\varepsilon$ 3.1).
IR $\nu_{\text{max}}$ (KBr) 3350, 2955, 2875, 1480, 1395, 1285, 1140, 1080, 820, 740, 730 cm$^{-1}$.

$^1$H NMR (CDCl$_3$): $\delta$ 3.66 (1H, d, $J = 6.05$ Hz, H-1a), 3.61 (1H, d, $J = 6.32$ Hz, H-1b), 1.56 (2H, m, H-2'), 1.25 (60H, brs, 30xCH$_2$), 0.88 (3H, t, $J = 5.89$ Hz, Me-33).

$^{13}$C NMR: $\delta$ 63.10 (C-1), 32.82 (C-2), 31.92 (CH$_2$), 29.68 (27x CH$_2$), 25.73 (CH$_2$), 22.67 (CH$_2$), 14.09 (Me-33).

EIMS $m/z$ (rel. int.) 480 [M$^+$ (C$_{33}$H$_{66}$O)] (1.3), 449 (12.0), 435 (1.1), 421 (21.1), 407 (4.3), 393 (8.6), 379 (2.3), 365 (2.7), 351 (2.3), 337 (2.5), 323 (2.6), 309 (2.8), 295 (3.1), 281 (4.3), 267 (4.8), 253 (8.1), 239 (8.3), 225 (8.4), 211 (8.6), 197 (8.7), 183 (7.4), 169 (8.7), 155 (9.6), 141 (10.0), 127 (16.3), 113 (26.2), 99 (47.1), 85 (49.8), 71 (51.8), 69 (63.8), 57 (100), 43 (93.9).

Hibiscusyl ester (5)

Elution of the column with petroleum ether-chloroform (3:1) (fractions 42-52) yielded colourless amorphous powder of 5, recrystallized from chloroform-methanol (1:1), 290 mg (0.0141 % yield), mp 90-91°C, Rf 0.35 (petroleum ether : hexane : chloroform :methanol; 13:3:14:10).

IR $\nu_{\text{max}}$ (KBr) 2940, 2850, 1735, 1460, 1255, 1090, 1010, 800, 790, 725, 710 cm$^{-1}$.

$^1$H NMR (CDCl$_3$): $\delta$ 3.57 (1H, d, $J = 6.51$ Hz, H-1'), 3.53 (1H, d, $J = 6.57$Hz, H-1b'), 1.97 (1H, m, H-2'), 1.49 (2H, m, CH$_2$), 1.19 (50H, brs, 25x CH$_2$), 0.81 (3H, t, $J = 9.30$ Hz, Me-24)
$^{13}$C NMR (CDCl$_3$): 173.02(CO-1'), 63.09 (CH$_2$O-1), 39.79 (CH-2'), 32.93(CH$_2$), 31.94 (CH$_2$), 29.70 (24 x CH$_2$), 29.35 (CH$_2$), 25.82 (CH$_2$), 22.66 (CH$_2$), 13.98 (Me - 24).

EI/MS m/z (rel. int.) 450 [M]$^+$ [ C$_{30}$H$_{50}$O$_2$] (2.1), 423 (16.7), 409 (5.2), 395 (16.3), 381 (2.1), 351 (1.9), 309 (2.1), 283 (2.5), 253 (2.7), 239 (2.6), 25 (6.3), 197 (8.0), 183 (10.0), 169 (13.7), 155 (10.0), 141 (13.6), 127 (18.1), 113 (91.8), 97 (100), 85 (51.3), 71 (32.6), 69 (53.2), 57 (82.6), 43 (81.2).

Hibiscusol (6)

Elution of the column with petroleum ether-chloroform (3:1), (fractions 53-65) yielded dull white powder of 6, recrystallized from chloroform-methanol (1:1), 208 mg (0.01 % yield), mp 80-81°, R$_f$ 0.34 (petroleum ether : Hexane : chloroform : methanol; 13: 3: 14: 10).

IR $\nu_{\text{max}}$ 3300, 2945, 2855, 1460, 1250, 1095, 1015, 805, 790, 720, 705 cm$^{-1}$.

$^1$H NMR (CDCl$_3$): $\delta$ 3.55 (1H, dd, J = 6.54, 6.57 Hz, H-5 $\alpha$), 1.59 (1H, m, H - 27), 1.19 (56 H, brs, 28x CH$_2$), 0.81 (3H, t, J = 6.21 Hz, Me-1).

$^{13}$C NMR (CDCl$_3$) : $\delta$ 63.07 (CHOH), 32.90 (C- 27), 31.92 (CH$_2$), 29.68 (7x CH$_2$), 29.45 (11x CH$_2$), 29.33 (7 x CH$_2$), 25.78 (CH$_2$), 22.65 (CH$_2$), 13.99 (CH$_3$-1).

EI/MS m/z (rel. int.) 450 [M]$^+$ (C$_{31}$H$_{62}$O) (3.1), 421(10.7), 407 (5.2), 393 (11.4), 365 (5.2), 309 (4.3), 295 (4.4), 281 (4.5), 267 (4.6), 253 (5.0), 239 (5.1), 225 (5.2), 211 (6.4), 197 (6.7), 183 (7.1), 169 (11.3), 155 (11.4), 141 (12.3), 127 (21.6), 111 (38.5), 97 (71.8), 83 (73.2), 69 (71.1), 57 (100).
DISCUSSIONS

Compound 1

Compound 1, an aliphatic hydrocarbon ($M^+ 380$, $C_{27}H_{56}$), did not show any characteristic IR absorption bands for functional groups. Its mass spectrum showed ion fragments relating to $C_nH_{2n+1}$, $C_nH_{2n}$ and $C_nH_{2n-1}$ and most of the peaks were separated by 14 mass units and decreased in abundance with increasing molecular weight of long chain aliphatic compounds. (Budzikiewicz et al., 1965). More intense ion peaks corresponding to $C_nH_{2n+1}$ (m/z 71, 85, 99, 113, 127 etc.) in comparison to that relating to $C_nH_{2n-1}$ (m/z 69, 83, 97, 111, 125, etc) supported the acyclic and saturated nature of the molecule. The $^1H$ NMR spectrum of 1 displayed two three-proton triplets at $\delta$ 0.88 (J = 6.0 Hz) and 0.85 (J = 6.5 Hz) assigned to terminal primary methyl protons and a broad signal at $\delta$ 1.25 (50 H) for 25 methylene signals. The $^{13}C$ NMR spectrum of 1 exhibited carbon signals for methyl carbons (δ 22.70 and 14.10). The absence of any signals beyond δ 1.25 in the $^1H$ NMR and δ 31.94 in the $^{13}C$ NMR spectrum confirmed the saturated nature of the molecule and ruled out the location of any functional group. On the basis of these spectral data analyses the structure of 1 has been established as $n$-pentacosane.

Compound 2

Compound 2, named D-3-epi-tetraxeryl acetate, was obtained as colourless amorphous powder from petroleum ether-chloroform (95:5). It responded positively to Liebermann-Burchard test and exhibited a strong IR absorption band at 1730 cm$^{-1}$ characteristic to an ester group. It had a molecular ion peak at m/z 468 corresponding to triterpenic ester $C_{32}H_{52}O_2$. The mass spectrum of 2 demonstrated the existence of characteristic ion fragments at
m/z 453 [M - Me] +, 408 [M - AcOH]+ and 393 [408-Me]+. The important ion fragment were observed at m/z 122 [C_{5,6} - C_{9,10} fission, 182 - AcOH]+, 286 [M-182]+, 258 [C_{7,8} - C_{9,10} fission]+, 250 [C_{8,14} - C_{9,11} fission]+, 218 [M - 250]+, 207 [250-Ac]+, 190[250-AcOH]+, 204[C_{8,14} - C_{11,12} fission]+ and 190 [C_{8,14} - C_{12,13} fission]+ suggesting saturated nature of the rings A, B and C (Budczikiewicz et al., 1965) (Scheme 5.1)

The ¹H NMR spectrum of 2 (Table 5.1) displayed a one proton double doublet at δ 5.50 (J = 3.0, 3.03 Hz) for a vinyl proton assigned to H-15 of a taraxerol type molecule. A one proton carbinol proton appeared as a double doublet at δ 4.45 ascribeable to C-3 acetoxyl methine proton. On the basis of biogenetic grounds and its coupling interactions of 5.74 and 6.48 Hz (H-3 eq-H-2 eq, H-3 eq-H_{2ax}) indicated α-orientation of C-3 acetoxyl group. Two one-proton doublets at δ 1.95 (J = 3.0 Hz) and 1.90 (J = 3.03 Hz) were accounted to C-16 methylene protons adjacent to the vinylic linkage. A one proton double doublet at δ 1.54 (J = 5.60, 9.82 Hz) was associated with C-18 β proton. Four three - proton broad signals at 1.10, 0.88, 0.86 and 0.83 were accounted to C-23, C-24, C-27 and C-28 methyl protons respectively. Two six-proton broad signals at δ 0.95 and 0.91 were attributed to C-25, C-26 and C-29, C-30 methyl protons, respectively. A three -proton broad signal at δ 2.02 was due C-3 acetoxyl group. The existence of these methyl signals between δ 1.10 - 0.83 indicated location of all the methyl functionalities at saturated carbons. The ¹³C NMR spectrum of 2 showed the presence of 32 carbon atoms. The assignment of the proton and carbon chemical shifts were made by comparison with the δ values of the corresponding positions of taraxeranes (Carpenter et al., 1980; McLean et al., 1987; Merfort et al., 1992; Persons et al., 1993; Sakuri et al., 1987).

Alkaline hydrolysis of the D-3- epi-taraxeryl acetate (2) produced free alcohol 2a.
Oxidation of 2a with Johnes' reagent gave the 3-oxo derivative 2b which responded positively to Zimmermann test (Barton and Mayo, 1954) for 3-oxo terpenoids suggesting the presence of the secondary hydroxyl group at C-3. The NaBH₄ reduction of 2b, regenerated taraxerol 2c, confirming axial orientation of the hydroxyl group in 2a and, hence, acetoxy group in 2. Based on these evidences the structure of 2 has been elucidated as D-friedo-olean-14-en-3α-yl acetate. This is a new friedelane type triterpene isolated from *H. rosa-sinensis* and other related species. The presence of taraxeryl acetate has already been reported from this plant (Aggrawal and Rastogi, 1971).

Table 5.1. ¹H NMR and ¹³C NMR Spectral data of 3-epi-Taraxeryl acetate (2) (CDCl₃)

<table>
<thead>
<tr>
<th>Position</th>
<th>¹H NMR</th>
<th>¹³C NMR</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>α</td>
<td>β</td>
</tr>
<tr>
<td>1</td>
<td>1.37 dddd (16.12, 9.75, 5.32, 3.42)</td>
<td>1.64 dddd (5.31, 11.23, 4.21, 9.36)</td>
</tr>
<tr>
<td>2</td>
<td>1.67 m</td>
<td>1.62 m</td>
</tr>
<tr>
<td>3</td>
<td>-</td>
<td>4.45 dd (5.74, 6.48)</td>
</tr>
<tr>
<td>4</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>1.46 dd (5.26, 6.13)</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>1.33 m</td>
<td>1.26 m</td>
</tr>
<tr>
<td>7</td>
<td>1.62 m</td>
<td>1.24 m</td>
</tr>
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</tr>
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<td>1.51 m</td>
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</tr>
<tr>
<td>10</td>
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</tr>
<tr>
<td>11</td>
<td>1.62 m</td>
<td>1.28 m</td>
</tr>
<tr>
<td></td>
<td>1.0 dddd (11.61, 9.36, 5.34, 6.08)</td>
<td>1.59 dddd (5.34, 10.38, 12.43, 6.14)</td>
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</tr>
<tr>
<td>13</td>
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</tr>
<tr>
<td>14</td>
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</tr>
<tr>
<td>15</td>
<td>5.50 dd (3.00, 3.03)</td>
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<tr>
<td>16</td>
<td>1.95 d (3.0)</td>
<td>1.90 d (3.03)</td>
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<tr>
<td>17</td>
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<td>-</td>
</tr>
<tr>
<td>18</td>
<td>-</td>
<td>1.54 dd (5.60, 9.82)</td>
</tr>
<tr>
<td>19</td>
<td>1.58 d (5.62)</td>
<td>1.40 d (9.84)</td>
</tr>
<tr>
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<td>-</td>
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<tr>
<td>21</td>
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<td>25</td>
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<tr>
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<tr>
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</tr>
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<td>28</td>
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</tr>
<tr>
<td>29</td>
<td>0.91 brs</td>
<td>-</td>
</tr>
<tr>
<td>30</td>
<td>0.91 brs</td>
<td>-</td>
</tr>
<tr>
<td>OAc</td>
<td>2.02 s</td>
<td>-</td>
</tr>
</tbody>
</table>

Coupling constants in Hertz are provided in parentheses.
Scheme 5.1. Mass spectral fragmentation pattern of 3-epi-taraxerol (2)
Compound 3, taraxeryl acetate ([M]⁺ 468, C₃₂H₅₂O₉), had an IR absorption band at 1740 cm⁻¹ (ester) and identical fragmentation pattern to that of 2. In the ¹H NMR spectrum the C-3 carbinol proton appeared at δ 4.38 (dd, J = 8.49, 5.35 Hz) (Table 5.2). The ¹³C NMR (Table 5.2) carbon values were compared with taraxerol (Parson et al., 1993). Alkaline hydrolysis of 2 yielded taraxerol (2c). On the basis of identical physical and spectral data the compound 3 has been formulated as D-friedo-olean-14-en-3β-ol.

Table 5.2. ¹H and ¹³C NMR spectral data of Taraxeryl acetate (3) (CDCl₃).

<table>
<thead>
<tr>
<th>Position</th>
<th>¹H NMR</th>
<th>¹³C NMR</th>
</tr>
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<tr>
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<td>α</td>
<td>β</td>
</tr>
<tr>
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<td>4.38 dd (8.49, 5.35)</td>
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</tr>
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<td>5</td>
<td>1.47 dd (5.32, 6.15)</td>
<td>-</td>
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<tr>
<td>6</td>
<td>1.33 m</td>
<td>1.26 m</td>
</tr>
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<td>1.54 m</td>
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</tr>
<tr>
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<td>1.44 m</td>
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</tr>
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<td>1.08 m</td>
<td>1.44 m</td>
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<tr>
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<td>5.46 dd (3.03, 3.09)</td>
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<tr>
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<td>1.87 d (3.03)</td>
<td>1.82 (3.10)</td>
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<tr>
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<td>17</td>
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<td>1.52 dd (5.52, 9.60)</td>
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<tr>
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<td>1.39 d (5.5)</td>
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<td>19</td>
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</tr>
<tr>
<td>20</td>
<td>1.29 m</td>
<td>1.11 m</td>
</tr>
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<td>21</td>
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</tr>
<tr>
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<td>0.83 brs</td>
<td>-</td>
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<tr>
<td>30</td>
<td>1.96 brs</td>
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</tr>
<tr>
<td>OAc</td>
<td>1.96 brs</td>
<td>-</td>
</tr>
</tbody>
</table>

Coupling constants in Hertz are provided parentheses.
Compound 4

Compound 4, an aliphatic alcohol, was obtained as a colourless amorphous powder from petroleum ether-chloroform (9:1) eluents. It showed characteristic IR absorption bands for hydroxyl (3350 cm\(^{-1}\)) and long aliphatic chain (820, 740, 730 cm\(^{-1}\)). Its mass spectrum had a molecular ion peak at \(m/z\) 480 relating to a molecular formula of aliphatic alcohol, \(C_{33}H_{56}O\) along with \(C_nH_{2n+1}\), \(C_nH_{2n}\), and \(C_nH_{2n-1}\) ions. Most of the ion peaks were separated by 14 mass units and decreased in abundance with increasing molecular weight of long straight chain hydrocarbon. More intense clusters of peaks corresponding to \(C_nH_{2n+1}\) (\(m/z\) 57, 71, 85, 99, 113, 127, 141, etc.) in comparison to \(C_nH_{2n-1}\) (\(m/z\) 69, 83, 97, 111, 125, 139, etc.) supported the acyclic and saturated nature of the compound (Budzikiewicz et al., 1965). The \(^1\)H NMR spectrum of 4 displayed two one-proton doublets at 3.66 (\(J= 6.05\) Hz) and 3.61 (\(J = 6.32\) Hz) assigned to oxygenated C-1 methylene proton. A three-proton triplet at \(\delta\) 0.88 (\(J = 5.89\) Hz) was ascribed to C-33 terminal methyl group. The remaining methylene protons resonated as a multiplet at \(\delta\) 1.56 (2H) and as a broad signal at \(\delta\) 1.25 (60 H). The \(^{13}\)C NMR spectrum of 4 exhibited a signal at \(\delta\) 63.10 assigned to oxygenated methylene carbon. The absence of signals beyond \(\delta\) 3.66 in the \(^1\)H NMR spectrum and \(\delta\) 63.10 in the \(^{13}\)C NMR spectrum confirmed the saturated nature of the molecule. Based on these evidences the structure of 4 has been formulated as \(n\)-tritriacontan -1-ol and isolated for the first time from \(H. rosa-sinensis\).

Compound 5

Compound 5, designated \(h\)ibiscus\(y\)l ester, was obtained as a colourless amorphous powder from petroleum ether - chloroform (3:1) eluents. Its IR spectrum showed the presence of ester group (1735 cm\(^{-1}\)) and long aliphatic chain (790, 725, 710 cm\(^{-1}\)). It had a molecular ion peak at \(m/z\) 450 in its mass
spectrum corresponding to a long aliphatic ester \( C_{30}H_{58}O_2 \). The mass spectrum of 5 gave \( C_nH_{2n+1}, C_nH_{2n} \) and \( C_nH_{2n-1} \) ions with higher abundance for lower fragments. Most of the ion peaks were separated by 14 mass units and the abundance decreased with increasing molecular weight of the long aliphatic chain. The generation of the base peak at \( m/z \) 98 due to cleavage of ester linkage indicated cyclopentane carboxylic ester at one of the terminal of the chain. The \(^1H\) NMR spectrum of 5 displayed two one-proton doublets at \( \delta \) 3.57 (J= 6.51 Hz) and 3.53 (J= 6.57 Hz) assigned to C-1 oxygenated methylene protons. A one-proton multiplet at \( \delta \) 1.97 was ascribed to C-2' methine proton adjacent to carbonyl group. A three - proton triplet at \( \delta \) 0.81 (J=9.30 Hz) was associated with C-24 primary methyl group. The remaining methylene protons appeared at \( \delta \) 1.49 (2 H) and 1.19 (50 H). The \(^{13}C\) NMR spectrum of 5 exhibited important carbon signals at \( \delta \) 173.02 (ester CO), 63.09 (O-CH\(_2\)) and 13.98 (Me- 24). The absence of any signal beyond \( \delta \) 3.57 in the \(^1H\) NMR spectrum and between \( \delta \) 173.02 - 63.09 in the \(^{13}C\) NMR spectrum confirmed the saturated nature of the molecule. On the basis of these spectral data analyses the structure of 5 was formulated as cyclopentanyi carboxy-\( n\)-tetracosane. This is an unreported aliphatic ester isolated from \( H. rosa-sinensis \) for the first time.

**Compound 6**

Compound 6, named *hibiscusol*, was obtained as a dull white powder from petroleum ether-chloroform (3:1) eluents. Its IR spectrum showed absorption bands for hydroxyl group (3300 cm\(^{-1}\)) and long aliphatic chain (790, 720, 705 cm\(^{-1}\)). The mass spectrum of 6 exhibited a molecular ion peak at \( m/z \) 450
corresponding to a long chain alcohol with a cyclic ring $C_{31}H_{62}O$. The prominent ion peaks observed in the mass spectrum at $m/z \ 57[C_4H_9]^+$. 393 $[M-C_4H_9]^+$ and 69 [cyclopentanyl ring]$^+$ indicated the existence of the hydroxyl group at C-5 and cyclopentanyl ring at one of the terminal of the carbon chain (Scheme 5.2). The mass spectrum had $C_nH_{2n+1}$, $C_nH_{2n}$ and $C_nH_{2n-1}$ ions and most of the fragments were separated by 14 mass units. The intensity of the ion peaks decreased with increasing molecular weight of the long straight chain hydrocarbon. The absence of [M$^+$- Me] ion suggested its straight chain nature (Stoianova - Ivanova et al., 1969). The $^1H$ NMR spectrum of 6 showed a one-proton double doublet at $\delta \ 3.55 (J = 6.54, \ 6.57 \ Hz)$ assigned to $\alpha$ orientated C-5 carbinol proton. A one-proton multiplet at $\delta \ 1.59$ was associated with C-27 methine proton. A three-proton triplet at $\delta \ 0.81 (J=6.21 \ Hz)$ was ascribed to C-1 primary methyl group. The remaining methylene protons appeared as a broad signal at $\delta \ 1.19$. The $^{13}C$ NMR spectrum of 6 displayed carbon signals for carbinol ($\delta \ 63.07$), methyl ($\delta \ 13.99$), methine ($\delta \ 32.90$) and methylene carbons between $\delta \ 31.92 - 22.65$. The absence of any signal beyond $\delta \ 3.55$ in the $^1H$ NMR spectrum and $\delta \ 63.07$ in the $^{13}C$ NMR spectrum confirmed the saturated nature of the molecule. On the basis of these evidences the structure of 6 has been elucidated as 26-cyclopentyl n-hexacosan-5-ol. This is a new aliphatic constituents isolated from a natural or synthetic source for the first time.
Scheme 5.2. Mass fragmentation pattern of Hibiscusol (6)
Table 5.3. Chemical Constituents isolated from the leaves of *Hibiscus rosa sinensis* Linn.

<table>
<thead>
<tr>
<th>Code</th>
<th>Name</th>
<th>Column eluent</th>
<th>Rf, mobile phase</th>
<th>Yield (% w/w) by preparative chromatography</th>
<th>m.p°C</th>
<th>Mol. Wt. [M]* m/z [Mol. For.]</th>
<th>Nomenclature</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Aliphatic hydrocarbon</td>
<td>A</td>
<td>0.76 E</td>
<td>0.0128</td>
<td>58-59°C</td>
<td>380 C_{27}H_{56}</td>
<td>n-Pentacosane</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>3-epi-Taraxeryl acetate</td>
<td>A-B 95:5</td>
<td>0.75 F</td>
<td>0.011</td>
<td>300-301°C (+) 152.6 (C=0.05)</td>
<td>468 C_{32}H_{52}O_{2}</td>
<td>D-Friedo-olean-14-en-3a-yl acetate (New)</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Taraxeryl acetate</td>
<td>A-B 95:5</td>
<td>0.82 F</td>
<td>0.011</td>
<td>303-305°C (+) 10.5 (C=1.5) at 23°C</td>
<td>468 C_{32}H_{52}O_{2}</td>
<td>D-Friedo-olean-14-en-3β-yl acetate</td>
</tr>
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<tr>
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<td>Aliphatic alcohol</td>
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<td>0.38 G</td>
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<td>74-78°C</td>
<td>576 C_{33}H_{68}O</td>
<td>n-Tritriacontan-1-ol (New)</td>
</tr>
<tr>
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</tr>
<tr>
<td>5</td>
<td>Hibiscusyl ester</td>
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<td>0.35 H</td>
<td>0.014</td>
<td>90-91°C</td>
<td>450 C_{30}H_{58}O_{2}</td>
<td>Cyclopentanyl carboxy-n-tetracosane (New)</td>
</tr>
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<td></td>
</tr>
<tr>
<td>6</td>
<td>Hibiscusol</td>
<td>A-B 3:1</td>
<td>0.34 H</td>
<td>0.01</td>
<td>80-81°C</td>
<td>450 C_{31}H_{52}O</td>
<td>26-cyclopentyl n-hexacosan-5-ol (New)</td>
</tr>
</tbody>
</table>

A = Petroleum ether, B = Chloroform, C= Hexane, D= Methanol

E = Mobile phase (petroleum ether : hexane : chloroform : methanol ; 18:1.5 : 1.5:0.5),

F= Mobile phase (petroleum ether: hexane: chloroform : methanol ; 6:2:2:3)

G= Mobile phase ( petroleum ether : hexane : chloroform : methanol; 14:4:12:9)

H= Mobile phase (petroleum ether: hexane: chloroform : methanol ; 13:3:14:10)
Chemical constituents isolated from the leaves of *Hibiscus rosa sinensis*

1. n-Pentacosane

2. 3-epi-tetrahydro acetate

3. Taraxeryl acetate

4. Aliphatic alcohol

5. Hibiscusyl ester

6. Hibiscusol
Chapter 5

HIBISCUS ROSA-SINENSIS L.

SPECTRA AND REFERENCES
EIMS of n-Pentacosane (1)
EIMS of 3-epi-taraxeryl acetate (2)

${}^1$H NMR of 3-epi-Taraxeryl acetate (2)
$^{13}$C NMR of 3-epi-Taraxeryl acetate (2)

EIMS of Taraxeryl acetate (3)
\textbf{H NMR of Teraxeryl acetate (3)}

\textbf{\textsuperscript{13}C NMR of Taraxeryl acetate (3)}
CH$_3$(CH$_2$)$_{31}$ CH$_2$OH

EIMS of Aliphatic alcohol (4)

CH$_3$(CH$_2$)$_{31}$ CH$_2$OH

$^1$H NMR of Aliphatic alcohol (4)
NMR of Aliphatic alcohol (4)

\[ \text{CH}_3(\text{CH}_2)_{31} \text{CH}_2\text{OH} \]

\[ 1^3\text{C NMR of Aliphatic alcohol (4)} \]

\[ \text{Hibiscusyl ester (5)} \]

\[ \text{CH}_3(\text{CH}_2)_{22}\text{CH}_2\text{O}^\text{2'}\text{CO}^\text{2'} \]

\[ \text{1H NMR of Hibiscusyl ester (5)} \]
EIMS of Hibiscusyl ester(5)
$^{13}$C NMR of Hibiscusyl ester (5)

$^1$H NMR of Hibiscusol (6)
EIMS of Hibiscusol (6)
$\text{CH}_2(\text{CH}_2)_{20}^\text{CH}-(\text{CH}_2)_3^\text{OH}$
REFERENCES


Maheswari, P and Sigh, U. *Dictionary of economic plants of India*, ICAR, New Delhi, p .82, 1965.


