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Higher Fatty Acids From Pleurospermum Densiflorum

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ABSTRACT

Fatty Aicds have been isolated from the petrolium ether, benzene and Chlaroforum fraction of the areal part of P. densiflorum and identified by means of ^{1}HNMR, ^{13}CNMR, I. R. Spectrums, Mass Spectrum data as well as by colour reactions. Alkanes, Fatty acids β-sitosterol have been found.

Key words – Fatty Acids, P.densiflorum, Apiaceae, β-Sitosterol, Fatty Ester, Coumarins, High Altitude Himalayan herbs.

Plant Material

The plant Pleurospermum densiflorum (Apiaceae) was collected in the month of September at an altitude of 17500-18000 ft. along the snow lines, from Millam glaciers of the Kumaon Himalaya, Uttranchal, India. The plant was identified in the Department of Botany, Kumaon University, Nainital, well as Forest Research Institute, Dehradun.

Experimental

Extraction and isolation - Shade dried aerial parts of P. densiflorum were pulverised and 950 gm powder material extracted in Soxhlet apparatus with 90% MeOH for 120 hrs. After complete extraction it was concentrated under reduced pressure in a rotatory vacuum evaporator.

The concentrated MeOH residue was further extracted and fractionated with petroleum ether (60-80°C), benzene (78-81°C), chloroform (40-60°C), ethyl acetate and lastly with MeOH. The petroleum ether extract. benzene extract, chloroform extract, ethyl acetate extract and methanol extract were concentrated under reduced pressure in a rotatory vacuum evaporator and strored for analysis.

ANALYSIS OF FRACTION 248

The benzene and chloroform extracts of P. densiflorum were mixed together and subjected to silica gel G. chromotography. The cloumn was eluted with 100% benzene and each fraction was checked on TLC and HPLC. **Fraction no. (243-248)** were similar in TLC and Rf values. These were mixed up, a light yellow coloured residue was obtained. Which on further column chromatography afforded the following white coloured compounds, when the silica gel G. CC was eluted with increasing the polarity. Identification of these **compound1, 2, 3 and4** have been done on the basis of **^{1}H NMR, ^{13}C NMR** Spectra as well as by GC-MS analysis and literature search.

IDENTIFICATION OF COMPOUND-1

1. Molecular formula :: $C_{25}H_{50}O_2$

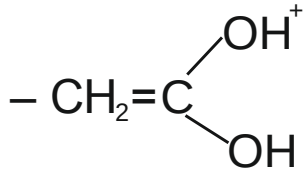
2. Molecular Weight :: 382

3. Melting Point :: 86°C (Lit.)

4. Mass Spectra :: M^+ (382), 365, 322, 60, 45

Fragmentation Patterns :: The main fragments identified were as follows-

(i) CH_3-$(CH_2)_{23}$– C=0^+ m/z = 365 (ii) $CH_3-(\overset{+}{C}H_2)_{22}-CH_2$ m/z= 322

(iii)  m/z = 60 (iv) – COOH m/z = 45

SPECTRAL STUDIES

^{1}H NMR Spectra :: CDCI, and TMS as an internal standards

ppm		
δ 10.3		$CH_3-(CH_2)_{21}-CH_2-CH_2-COO\underline{H}$
δ 2.32	t, j = 7.5 Hz	$CH_3-(CH_2)_{21}-CH_2-C\underline{H}_2-COOH$
δ 1.61	t, t, j=7.5, 7.5 Hz	$CH_3-(CH_2)_{21}-C\underline{H}_2-CH_2-COOH$
δ 1.35-1.20	m	$C\underline{H}_3-(CH_2)_{21}-CH_2-CH_2-COOH$
δ 0.86	t, j = 7.0 Hz	$CH_3-(CH_2)_{21}-CH_2-CH_2-COO\underline{H}$

^{13}C NMR Spectra :: ($125MH_7$)

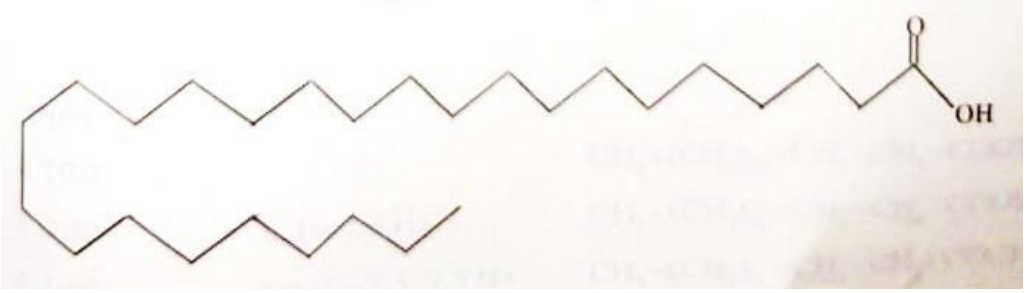
CDCI, and TMS as an internal standards

ppm	
δ 178.9 and 178.6	$CH_3-CH_2-CH_2-(CH_2)_{19}-CH_2-CH_2-\underline{C}OOH$
δ 33.8	$CH_3-CH_2-CH_2-(CH_2)_{19}-CH_2-\underline{C}H_2-COOH$
δ 31.8	$CH_3-CH_2-CH_2-(CH_2)_{19}-\underline{C}H_2-CH_2-COOH$
δ 29.6 – 29.0	$CH_3-CH_2-CH_2-(\underline{C}H_2)_{19}-CH_2-CH_2-COOH$

δ 24.6	$CH_3 - CH_2 - \underline{C}H_2-(CH_2)_{19} - CH_2 - CH_2 - COOH$
δ 22.6	$CH_3 - \underline{C}H_2 - CH_2-(CH_2)_{19} - CH_2 - CH_2 - COOH$
δ 14.0	$\underline{C}H_3 - CH_2 - CH_2-(CH_2)_{19} - CH_2 - CH_2 - COOH$

On the basis of above spectral studies and literature search it was **identified as Pentacosanoic acid $CH_3 - (CH_2)_{23} - COOH$ or Cerotic acid.**

STRUCTURE

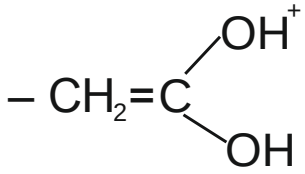


COMPOUND 1

IDENTIFICATION OF COMPOUND 2

1.	**Molecular formula**	**::**	$C_{26}H_{52}O_2$
2.	**Molecular Weight**	**::**	396
3.	**Melting Point**	**::**	87°C (lit.)
4.	**Mass Spectra**	**::**	M^+ (396), 379, 336, 60, 45
	Fragmentation Patterns	**::**	The main fragments identified were as follows-

(i) CH_3-$(CH_2)_{24}$ – C = 0^+ m/z = 379 (ii) $CH_3 - (\overset{+}{C}H_2)_{23} - CH_2$ m/z = 336

(iii)  m/z = 60 (iv) – COOH m/z = 45

SPECTRAL STUDIES

^{1}H NMR Spectra	::	$CDCl_3$ and TMS as an internal standards
ppm		
δ 10.3		$CH_3 - (CH_2)_{22} - CH_2 - CH_2 - COO\underline{H}$
δ 2.32	t, j = 7.5 Hz	$CH_3 - (CH_2)_{22} - CH_2 - C\underline{H}_2 - COOH$
δ 1.61	t, t, j=7.5, 7.5 Hz	$CH_3 - (CH_2)_{22} - C\underline{H}_2 - CH_2 - COOH$
δ 1.35-1.20	m	$C\underline{H}_3 - (C\underline{H}_2)_{22} - CH_2 - CH_2 - COOH$
δ 0.86	t, j = 7.0 Hz	$C\underline{H}_3 - (CH_2)_{22} - CH_2 - CH_2 - COOH$

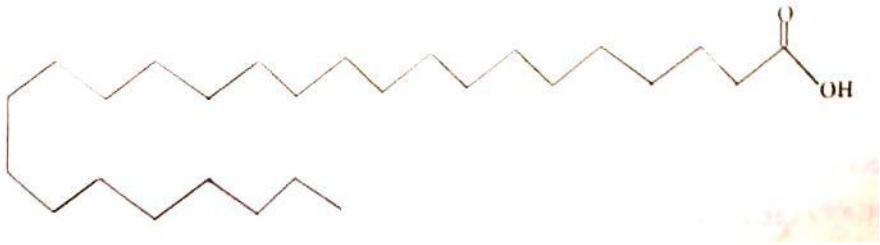
^{13}C NMR Spectra :: ($125MH_7$)

$CDCI_3$ and TMS as an internal standards

ppm

δ 178.9 and 178.6	$CH_3 – CH_2 – CH_2 –(CH_2)_{20} – CH_2 –CH_2 – \underline{C}OOH$
δ 33.8	$CH_3 – CH_2 – CH_2–(CH_2)_{20} – CH_2 –\underline{C}H_2 – COOH$
δ 31.8	$CH_3 – CH_2 – CH_2–(CH_2)_{20} – \underline{C}H_2 –CH_2 – COOH$
δ 29.6 – 29.0	$CH_3 – CH_2 – CH_2–(\underline{C}H_2)_{20} – CH_2 –CH_2 – COOH$
δ 24.6	$CH_3 – CH_2 – \underline{C}H_2–(CH_2)_{20} – CH_2 –CH_2 – COOH$
δ 22.6	$CH_3 – \underline{C}H_2 – CH_2–(CH_2)_{20} – CH_2 –CH_2 – COOH$
δ 14.0	$\underline{C}H_3 – CH_2 – CH_2–(CH_2)_{20} – CH_2 –CH_2 – COOH$

On the basis of above spectral studies and literature search it was **identifiedas Hexacosanoic acid $CH_3 – (CH_2)_{24} – COOH$.**



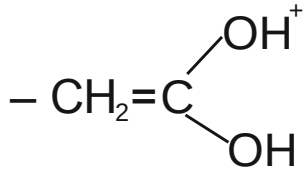
COMPOUND 2

IDENTIFICATION OF COMPOUND 3

1.	**Molecular formula**	**::**	$C_{27}H_{54}O_2$
2.	**Molecular Weight**	**::**	410
3.	**Melting Point**	**::**	87°C (lit.)
4.	**Mass Spectra**	**::**	M^+ (410), 393, 350, 60, 45
	Fragmentation Patterns	**::**	The main fragments

identified were as follows-

(i) CH_3-$(CH_2)_{25}$ – C = 0^+ m/z = 393 (ii) $CH_3 – (\overset{+}{C}H_2)_{24} – CH_2$ m/z = 350

(iii)  m/z = 60 (iv) – COOH m/z = 45

SPECTRAL STUDIES

^{1}H NMR Spectra **::** $CDCI_3$ and TMS as an internal standards

ppm

δ 10.3		$CH_3 – (CH_2)_{23} – CH_2 – CH_2 – COOH$
δ 2.32	t, j = 7.5 Hz	$CH_3 – (CH_2)_{23} – CH_2 – CH_2 – COOH$
δ 1.61	t, t, j=7.5, 7.5 Hz	$CH_3 – (CH_2)_{23} – CH_2 – CH_2 – COOH$
δ 1.35-1.20	m	$CH_3 – (CH_2)_{23} – CH_2 – CH_2 – COOH$
δ 0.86	t, j = 7.0 Hz	$CH_3 – (CH_2)_{23} – CH_2 – CH_2 – COOH$

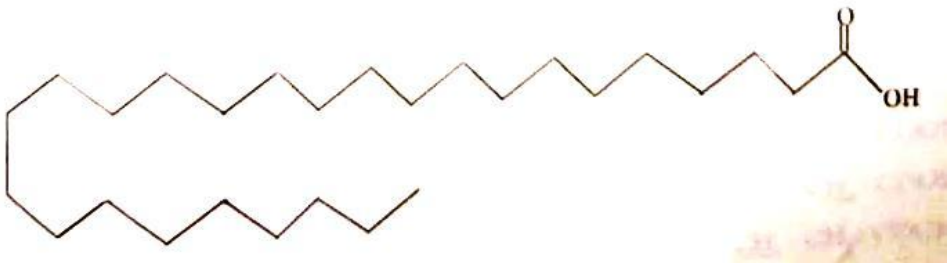
^{13}C NMR Spectra **::** $(125MH_7)$

$CDCI_3$ and TMS as an internal standards

ppm

δ 178.9 and 178.6	$CH_3 – CH_2 – CH_2 –(CH_2)_{21} – CH_2 –CH_2 – COOH$
δ 33.8	$CH_3 – CH_2 – CH_2–(CH_2)_{21} – CH_2 –CH_2 – COOH$
δ 31.8	$CH_3 – CH_2 – CH_2–(CH_2)_{21} – CH_2 –CH_2 – COOH$
δ 29.6 – 29.0	$CH_3 – CH_2 – CH_2–(CH_2)_{21} – CH_2 –CH_2 – COOH$
δ 24.6	$CH_3 – CH_2 – CH_2–(CH_2)_{21} – CH_2 –CH_2 – COOH$
δ 22.6	$CH_3 – CH_2 – CH_2–(CH_2)_{21} – CH_2 –CH_2 – COOH$
δ 14.0	$CH_3 – CH_2 – CH_2–(CH_2)_{21} – CH_2 –CH_2 – COOH$

On the basis of above spectral studies and literature search it was **identified as Heptacosanoic acid $CH_3 – (CH_2)_{25} – COOH$.**



COMPOUND 3

IDENTIFICATION OF COMPOUND 4

1.	**Molecular formula**	**::**	$C_{28}H_{56}O_2$
2.	**Molecular Weight**	**::**	424
3.	**Melting Point**	**::**	92°C (lit.)
4.	**Mass Spectra**	**::**	M^+ (424), 407, 364, 60, 45
	Fragmentation Patterns	**::**	The main fragments identified were as follows-

(i) $CH_3 – (CH_2)_{26} – C = 0^+$ m/z = 407 (ii) $CH_3 – (\overset{+}{C}H_2)_{25} – CH_2$ m/z = 364

$– CH_2 = C\begin{matrix} OH^+ \\ \diagdown \\ \diagup \\ OH \end{matrix}$

(iii) m/z = 60 (iv) – COOH m/z = 45

SPECTRAL STUDIES

^{1}H NMR Spectra :: $CDCl_3$ and TMS as an internal standards

ppm		
δ 10.3		$CH_3 – (CH_2)_{24} – CH_2 – CH_2 – COO\underline{H}$
δ 2.32	t, j = 7.5 Hz	$CH_3 – (CH_2)_{24} – CH_2 – C\underline{H}_2 – COOH$
δ 1.61	t, t, j=7.5, 7.5 Hz	$CH_3 – (CH_2)_{24} – C\underline{H}_2 – CH_2 – COOH$
δ 1.35-1.20	m	$C\underline{H}_3 – (C\underline{H}_2)_{24} – CH_2 – CH_2 – COOH$
δ 0.86	t, j = 7.0 Hz	$C\underline{H}_3 – (CH_2)_{24} – CH_2 – CH_2 – COOH$

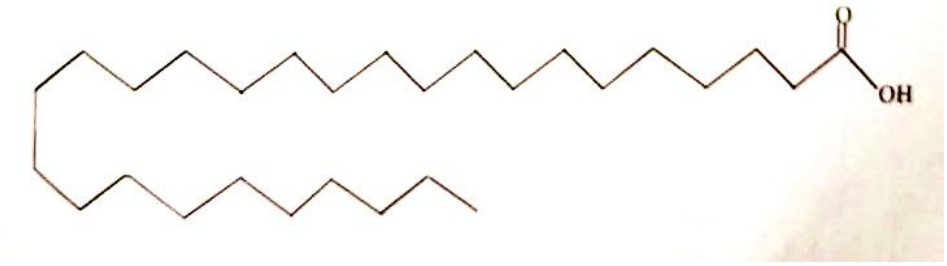
^{13}C NMR Spectra :: ($125MH_7$)

$CDCl_3$ and TMS as an internal standards

ppm	
δ 178.9 and 178.6	$CH_3 – CH_2 – CH_2 –(CH_2)_{22} – CH_2 –CH_2 – \underline{C}OOH$
δ 33.8	$CH_3 – CH_2 – CH_2–(CH_2)_{22} – CH_2 –\underline{C}H_2 – COOH$
δ 31.8	$CH_3 – CH_2 – CH_2–(CH_2)_{22} – \underline{C}H_2 –CH_2 – COOH$
δ 29.6 – 29.0	$CH_3 – CH_2 – CH_2–(\underline{C}H_2)_{22} – CH_2 –CH_2 – COOH$
δ 24.6	$CH_3 – CH_2 – \underline{C}H_2–(CH_2)_{22} – CH_2 –CH_2 – COOH$
δ 22.6	$CH_3 – \underline{C}H_2 – CH_2–(CH_2)_{22} – CH_2 –CH_2 – COOH$
δ 14.0	$\underline{C}H_3 – CH_2 – CH_2–(CH_2)_{22} – CH_2 –CH_2 – COOH$

On the basis of above spectral studies and literature search it was **identified as Octacosanoic acid $CH_3 – (CH_2)_{26} – COOH$.**

STRUCTURE



COMPOUND 4

Acknowledgement – The Author are grateful to CDRI Lucknow, RSIC for MS, ^{1}HNMR, ^{13}CNMR IR Spectral Results and Prof. K. S. KhetralNainital Department of Chemistry, Kumaon University

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