INTERNATIONAL JOURNAL OF RESEARCH IN COMMERCE, IT, ENGINEERING AND SOCIAL SCIENCES (IJRCIESS) ISSN: 2349-7793
Vol. 3 No. 1 (2009): January
Double-Blind Peer Reviewed Refereed Open Access International Journal

Esters and Fatty acids from Pleurospermumdensiflorum

P. S. Rajpoot
Reader
Department of Chemistry
Govt. P. G. College, Hardoi, UP 241001 India

Abstract

Esters and fatty acids have been isolated from the Petroleum ether, benzene and chloroform fraction of the areal parts of pleurospermumdensiflorum and identified by means of ^{1}HNMR, ^{13}CNMR, Mass spectral data as well as by hydrolytic studies. Two linear Furanocoumarins, Psoralen and bergapten along with β-Sitosterol have been identified. Bergabten has been found to be present as a major constituent.

Keyword: Pleurospermumdensiflorum Apiceae High altitude Himalayan herbs, Esters, Fatty acids. Linear Coumains, Bergapten

Introduction

Pleuraspermumdensiflorum belonging to the family Apiceae is an annual herb growing at an altitude of 17500-18000 ft. along the snow lines of the Kumaon Himalayan glaciers in Uttaranchal, India. It is traditionally used as one of the best herbal perfumes in the region and is known for its long lasting pleasant smell. It is used as an incense in the talk of the Himalayan region. A literature search revealed that a preliminary chemical screening report (1), an ester and coumains (2). Earlier this plant has neither been investigated chemically nor even recorded as a traditional perfume. In a programme to search useful high altitude Himalayan herbs this plant was collected and chemically investigated. Some Higher esters of fatty acids and alcohols **1-4**

and Higher fatty acids **5-10** were isolated and identified along with β-sitosterol, psoralen and bergapten. P **densiflorum** has been found to be rich in coumains and bergapten was a major constituent present in the plant.

Results and discussion

The benzene and Chloroform extracts were mixed together and subjected to silica gel G. CC. On eluting the coloumn with Pet. Either : benzene (97.5 : 2.5) afforded compound (1-4). ^{1}HNMR, ^{13}CNMR, MS as wellas hydrolytic studies indicated the presence of esters of saturated acids and a saturated primary alcohol moiety.

The uniform loss of 14mu between a number of ionpeaks in its mass sloectoum showed the presence of longaliphatic chain. ^{1}HNMR spectrum of compound showed a triplet at δ 0.86ppm (J = 7.1 Hz) for six protons of two terminal methyl groups. A Triplet of δ 4.03 ppm (J = 6.7 Hz) was due to protons of methylene groups attached to oxygen atom of ester group (COO CH_2). Another triplet at δ 2.27 ppm (J = 7.6 Hz) was due to methylene protons adjacent to α carbonyl group (CH_2 COO). These two Triplets δ 4.03 and 2.27 indicated the presence of ester linkage in the compound. The integral for the triplet at δ 1.56 (J = 7.6 Hz) was accounted for by two protons assignable to CH_2CH_2CO and the broad clustered singlet at δ125 (1.36 – 1.16) integrating for methylene protons of acid and alcohol moieties present in the identical environment.

^{13}CNMR spectrum showed different signals which were assigned to different carbons of **1-4**. The singlet of δ 173.9 ppm was assigned to the ester carbonyl group. A peak at δ 64.3 ppm and at δ 34.3 ppm assigned to the carbons of O CH_2 and CH_2CO respectively.

The carbons adjacent to ester group were assigned by peak at δ 31.8, 28.5, 25.5, 24.9, 22.6 ppm respectively. The clustered signal at δ 29.6 – 29.1 ppm was due to remaining methylene carbons present in molecule and a peak at 14.0 ppm showed the presence of terminal methyl group. The structure of molecule as an ester was further supported be the demonstration that on saponification it afforded an acid A $C_{15}H_{31}COOH$ $[M^+]$ = m/z = 256 and an alcohol B $C_{22}H_{45}$ OH $[M^+]$ = m/z = 326.

These results as well as the published ^{13}CNMR values of similar type of compound (5) and ^{1}HNMR values of similar type of compound. It concluded that the compound **1** is docosanylhexadecanoate.

The other compounds **2**$C_{42}H_{84}O_2$ $[M^+]$(620), on hydrolysis afforded an acid $G_7H_{35}COOH$ $[M^+]$ = 284 and an alchol$C_{24}H_{29}OH$ $[M^+]$ = 354. The ^{1}HNMR and ^{13}CNMR values were interpreted ain of compound 1. Thus 2 identified as Tetracosanyloctadecanoate.

Compound **3**$C_{46}H_{92}O_2[M^+]$ (676), on hydrolysis afforded an acid $C_{19}S_{39}COOH$ $[M^+]$312 and an alcohol$C_{26}H_{53}$ O_H $[M^+]$ 382. The ^{1}HNMR and ^{13}CNMR values of compound were interpreted as on case of compound **1**. Thus **3** was identified as hexacosanoleicosanoate, compound **4**$C_{50}H_{100}O_2[M^+]$ (732), on hydrolysis afforded an acid $C_{21}H_{43}COOH[M^+]$ = m/z 340 and an alcohol $C_{28}H_{57}$ O_H $[M^+]$ 410. The ^{1}HNMR and ^{13}CNMR spectral values of compound

were also interpreted as in case of compound **1**. Thus compound **4** was identified as octaconsanyldocosanoate.

The benzene and chloroform mixed fraction column of P. densiflorum further on elution with 100% benzene afforded a light yellow coloured residue which on further column chromatography Silicagel GCC, on elution with increasing polarity afforded the compound **5-10**. Which were identified by means of ^{1}HNMR and ^{13}CNMR spectral results and mass spectral methods.

The GC-MS results as well as ^{1}HNMR and ^{13}CNMR spectral values established that compound **5-10** were identified higher fatty acids.

Thus – compound **5**$C_{23}H_{46}O_2$MS 354 [M^+] 294, 60, 45 get was identified as Tricosanoic acid.

Compound **6**$C_{24}H_{48}O_2$[M^+] 368, identified as Tetra Cosanoic acid, compound **7**$C_{25}H_{50}O_2$[M^+] 382, identified asTentaCosanoic compound **8**$C_{26}H_{52}O_2$[M^+] 596 identified as Hecacasanoic acid and Compound **9** and **10**, $C_{27}H_{54}O_2$[M^+] 410 and $C_{28}H_{56}O_2$[M^+] 424 were identified HeptaCosanoic acid and OctaCosanoic acid respectively.

Plant material

Pleurospermumdensiflorum (Apiaceae) was collected in the month of September from Milamglacius of Kumaon Himalaya in Uttaranchal, India, growing at an altitude of 17500-18000 ft. along the snow lines. It was identified in the Department of Botany, Kumaon University, Nainital where the voucher specimen is stored.

Separation

Major fraction were separated in Silica gel G (Glaxo 60 × 120 mesh), CC and purified by TLC and HPLC water associates HPLC fitted with variable wavelength (190-750 nm) UV detector 6000 psi and M-45 dueal pump system and Z-module be BondapakC_{18} Cartridges or steel column were used to checking the purity of compounds at a constant flow rate of 1.5 ml per minute using HPLC, TLC Zones were visualized either by exposourtoI_2vapours, 15% H_2SO_4, with Iron (III) chloride or under long range UV light (365 nm).

Extraction and isolation

The shade dried whole plant was pulverized and soxhlet extracted with 90% MeOH for 72 hrs. The alcoholic extract was concentrated in vacuum. The concentrate was then reextractedfractionated with petroleum either, benzene, chloroform ethylacetate and mostly with MeOH each factions was concentrated in vacuum. The defatted benzene and chloroform mixed residue was chromatographed (CC) on silica gel G. Elution was carried out with Pet either, benzene ethyl acetate in different proportions. These collections afforded **1-10** which were repeated purified by TLC and HPLC method.

Acknowledgement

The authors are grateful to RSIC, CDRILucknow for MS ^{1}HNMR and ^{13}CNMR spectral results and Prof. K. S. Khetwal Department of Chemistry Kumaon University Nainital.

References:

1. Khetwal, K. S. and Verma, D. L. 1990, Indian Drugs 28.
2. K. S., Khetwal, A. A. Rirvi and SunitaPandey, Phytochemistry, Vol. 35, No. 4 1033-1035, 1994.
3. Khetwal, K. S. and Verma, D. L., Pathak, R. P. Manral. K., Tandon, A. and Joshi Manju, Indian drugs, 1985, 23 (3) p- 123-128.
4. Neerja Pant, D. L. Jain & R. S. Bhakuni Indian Journal of Chemistry, Vol. 41B, Sept. 2003, pp. 1980-1986.
5. Kuo – Hsiung Lee and T. O. Soine, Journal of pharmancautical Sciences, Vol. 58, No. 6, June 1969 (681-683)
6. K. Na Kanihsc, Infrared Absorption Spectrescopy Holden day, June, San Francisco, Calif., 1962, p. 52.
7. Murray, RD. H., Ménde, Z. J. and Brown, S. A. (1982) The Natural Coumarins, Occurrence, Chemistry and Biochemistry, John Wiley, New York.
8. Murrey, RD. H. and Jorge, Z. D. (1984) Phytochemistry 23, 697.
9. Conzalez, A. G., Diaz, C. E., Lopez Dorta, H., Luis, J. R. and Rodriguez, L. F. (1977) An. Quim. 73, 430.
10. Khetwal, K. S. and Harbola, S. (1989) J. Nat. Prod. 52, 837.
11. Medina, R. J. and Rodriguez, L F (1976) An. Quin 72, 60.