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"Coumarins from Pleurospermum Densiflorum"

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ABSTRACT

Coumarins have been isolated from benzene and Chlaroform fraction of the P. densiflorum and identified by means of ^{1}HNMR, ^{13}CNMR, I. R. Spectrums, Mass Spectrum data as well as by colour reactions. Fatty acids, Fatty Ester.

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

INTRODUCTION

The plant Pleurospermumdensiflorum (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.

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RESULT AND DISCUSSION

The benzene and chloroform extracts of P. densiflorumwere mixed together and subjected to silica gel G. chromatography. On eluting column with benzene: ethyl acetate (95: 5 v/v) gave **fraction No.(268-274)**were light greenish blue coloured. After checking purity of thesefractions on TLC, similar fractions were mixed up. It was further purified with petroleum ether and acetone on TLC and HPLC and a white coloured pure **compound 1** was obtained. It was crystallised from acetone. It was soluble in chloroform & benzene. It appeared as sky blue fluorscent spot on silica gel TLC plate under the long range UV light (365 nm.). It was identifiedas follows.

IDENTIFICATION

1.	**Melting point**	**::**	162°C (160-161°C lit.)
2.	**Molecular formula**	**::**	C_{11} H_6 O_3
3.	**Molecular weight**	**::**	186
4.	**Elemental Analysis**	**::**	
	i. Found Values		C=70.98%, H=3.29%
	ii. Required Values		C=70.97%, H= 3.25%

COLOUR REACTIONS

1. It appeared as sky blue fluorscent spot on silica gel TLC plate under the long range UV light (365 nm.).
2. It gavedark brown colour on exposour to I_2 vapours.
3. It gave L.B. test positive and gave pinkish brown spot.
4. Compound gave violet colour on spraying the TLC with hydroxylamine and Fe Cl_3 solution.
5. The compound formed addition product with sodium bisulphate.
6. The compound formed dibromo derivatives with bromine water which on treatment with alkali gave coumarillic acid.
7. The compound was treated with 10% methanolic potassium hydrox ide solution, gave yellow solution, indicating the presence coumarin nucleus.

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SPECTRAL STUDIES

1. UV Spectra ::

λ_{Max}^{MeoH} (nm) 298, 262, 249, 242

The absorption bands of the compound in MeoH are characteristics of furano-coumarin structure. Shift reagent like H_3BO_3 and $AICI_3$ had no effect.

2. Mass spectra :: M^+ (186), 171, 158, 143, 128.171(M-CH,), 158 (M-CO).

3. IR Spectra :: υ_{max} (KBr) cm^{-1}: 3100, 1730, 1592,1493 1065, 930, 842.

υ(Cm^{-3})	Stretching
3100	due to Aromatic C-H- stretching.
1592	due to-C=C- stretching.
1730	due to-C=0- stretching.
852	due to furan ring

IR spectra support the compound to be a furanocoumarin.

4.^{1}H NMR Spectra :: ($CDCI_3$ and TMS as an internal standard).

δ (ppm)	proton	δ (ppm)	proton
6.4	IH of H-3	6.8	IH of H-4
7.2	IH of H-5	7.5	IH of H-8
7.7	1H of H-2`		

On the basis of colour reactions and spectral studiescompound has a furanocoumarin nucleus.

Based on UV spectra, mass spectra, **^{1}H NMR,** IR spectra, reactions and literature search (Sadler 1978; Murray, 1984).**Compound 1** was identified as and structure of the compoundassigned as follows.

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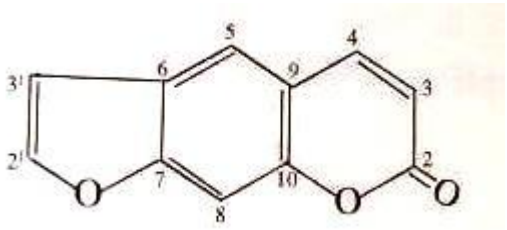
Structure



Psoralen

The structure of the compound was further confirmed by Co-chromatography with an authentic sample and mixed melting point.

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