Structural Analysis of Norviburtinal Obtained from the Root Barks of Kigelia Pinnata

Manoj Kumar^{1*}, Nempal Singh², L.P. Singh¹, Sachin Kumar² and Nishi²

¹Dept. of Chemistry, R.B.S. College Agra, U.P ²Dept. of Applied Science, Indraprastha Inst. of Technology, Amroha, U.P E mail: drmschemistry@gmail.com, npaluna@gmail.com

Abstract

In this paper we have reported the phytochemical investigation of Norviburtinal which is a Benzene extracted constituent from the root barks of kigelia pinnata. The crushed plant material has been extracted with different organic solvent with increasing polarity. The extract is then subjected to column chromatography in order to get pure organic constituents. The purity of compounds has been checked by qualitative TLC and the pure compound is then subjected to structural characterization to establish their structures.

Keywords: Kigelia Pinnata, Phytochemical, Bignoniaceae etc.

INTRODUCTION

Phytochemistry plays an important role in medicinal chemistry. In India, extreme variations of climate and geographic conditions, provide rich vegetation comprising 1,30,000 species of plants belonging to about 120 families and at least 24,000 of these plants have been used in indigenous Ayurvedic and Unani systems of medicine. Isolation of active principles which possess anti-cancer, anti-inflammation, antileprosy, antifertility and wide range of biological properties from aromatic and medicinal plants and their successful utilization to alleviate human suffering have encouraged research workers to continue investigation of new drugs from the plant kingdom with an ever increasing zeal. As a result, phytochemical research has made tremendous progress in recent times and a very large number of new secondary metabolites viz. alkaloids, terpenoids, steroids, flavonoids and related phenolic compounds, tannins, cumarins, quinones etc. have been discovered.

The present work was, therefore, undertaken with a view to isolate and characterise the active principles of some medicinally important plants. The plant of

Kigelia pinnata has the following specifications:

Family	_	Bignoniaceae	
Genus	-	Kigelia	
Species	-	Pinnata	
Vern	_	English – Sausage tree	
		Hindi – Jhar Phanoos	

Kigelia pinnata is a medium-sized spreading tree with pendulous recemes of dull liver-coloured flowers and long stalked large gourd-like fruits. It is cultivated as an ornamental and road side tree. The bark is greyish brown and rough; the leaves imparinnate and the flowers deep chocolate-red.

Its fruit is used in Africa as dressing for ulcer and for treatment of syphilis and rheumatism and as a purgative. The bark is used in rheumatism, dysentery and venereal diseases.

The leaves and flowers have been examined earlier. The present investigation leads to the isolation of five constituents obtained from its roots-bark through benzene extract namely-

- 1. Norviburtinal (A new compound)
- 2. Pinnatal (A new compound) 3-Methoxy caffeic acid (Ferulic acid)
- 3. 3,4-Dimethoxy caffeic acid
- 4. β-sitosterol

In this paper we have limited our studies to the Norviburtinal, a benzene extacted compound from the root bark of Kigelia Pinnata.

EXPERIMENTAL DETAILS

The plant material was collected from R.B.S. College Agra (India) and voucher specimen was deposited in the Herbarium of the Botanical Laboratories, Dr. B.R. Ambedkar University, Agra.

Extraction and Isolation of Constituents from the Roots-Bark

Air dried and finely powdered roots-bark (3 kg) was extracted exhaustively with petroleum ether ($60-80^{\circ}$) for 3 x 12 hrs followed by benzene. In each case, the extract was concentrated under reduced pressure and the concentrated mass subjected to preliminary examination by TLC. As the two extracts showed similar TLC behaviour, they were mixed up.

Column Chromatography

The dark green semisolid (40 g) was chromatographed over a column of silica gel. Elution was carried out with solvents of increasing polarity, the fractions was collected and worked up. Fractions showing similar TLC behaviour were mixed together and crystallized.

Characterization of Norviburtinal

The compound with yellow shining needles, m.p. 58°, sublimes at ambient temperature and co-distilled with solvent, gave single spot on silica gel TLC plate (petroleum ether : benzene 1 : 1). Elemental analysis and mass measurement (M^+ 146) showed C₉H₆O₂ as its molecular formula. It gave no response to Liebermann-Burchard and Noller's reagents. However, it reduced Tollen's reagent suggesting the presence of carbonyl function.

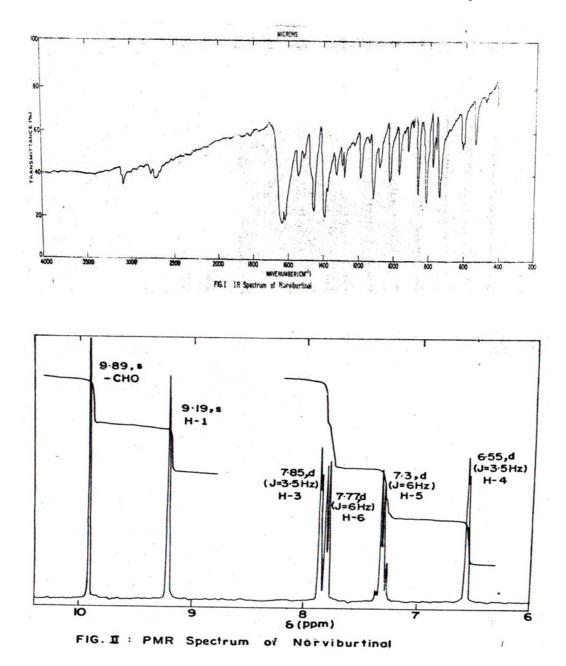
The ultraviolet spectrum of compound exhibited absorption at λ_{max}^{MeOH} 228, 242, 293, 424 nm.

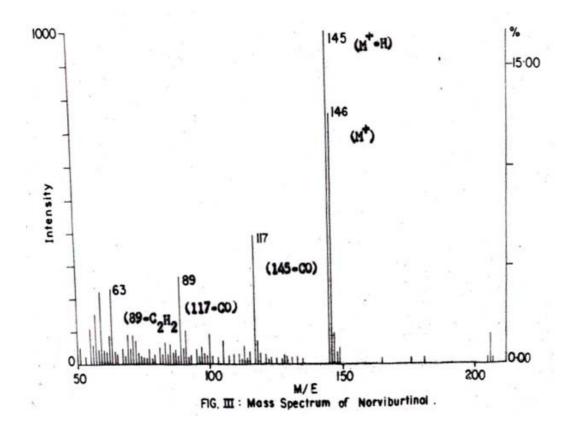
Infrared spectrum (Fig. I) showed important absorption peaks at v_{Max}^{KBr} 1635 (C=O), 1618, 1452, 1391, 1118, 1020, 970, 862, 740 cm⁻¹. The PMR values in CDCl₃ are listed below in Table I.

In the above PMR (Fig. – II) data, a singlet at 9.89 is due to an aldehyde group. A singlet at δ 9.19 appeared due to uncoupled proton present at position – 1. Doublet at δ 7.85 (J = 3.5 Hz) appeared due to a proton present at position – 3 which coupled with proton at position – 4. The proton at position – 4 also gave a doublet (J = 3.5 Hz) at δ 6.55 due to coupling with proton present at position – 3. Further, proton present at position – 5 coupled with vicinal proton at position – 6 giving a doublet at δ 7.30 (J = 6 Hz) and similarly proton at position – 6 coupled with proton present at position – 5 giving a doublet at δ 7.77 (J = 6 Hz). These observations suggested the cyclopenta [c] pyran structure.

A peak at 145 (Fig. – III) was shown due to loss of H from molecular ion peak. Loss of CO molecule from 145 resulted in a peak at 117. Further loss of CO molecular gave a peak at 83 which lost a C_2H_2 molecule yielding another peak at 63. The PMR spectrum is in good agreement with that reported for baldrinal.

Position δ ppm	Multiplicity	No. of Protons	Assignments
9.89	S	1	-CHO
9.19	S	1	H – 1
7.85	d (J = 3.5 Hz)	1	H – 3
7.77	d (J = 6 Hz)	1	H – 6
7.30	d (J = 6 Hz)	1	H – 5
6.55	d (J = 3.5 Hz)	1	H-4





RESULTS AND DISCUSSION

Evaporation of solvent from fraction nos. 16-20 gave a yellow solid (250 mg) which was found to be impure on silica gel TLC plate. It was therefore again chromatographed over a column of silica gel, elution being made with various mixtures of light petroleum and benzene. Successive removal of solvent from light petroleum-benzene (1:1) fraction, followed by crystallization from light petroleum, yielded a compound as yellow shining needles (200mg), m.p. 58° which showed single spot on TLC plate [solvent: light petroleum-benzene (1:3)]. It reduced Tollen's reagent indicating the presence of carbonyl function. It gave no response to Liebermann-Burchard and Noller's tests.

Its characterization as a now tarpenoid aldehyde was based on the following spectral studies –

Analysis : Found C, 74.0: H, 4.2%

C₉H₆O₂ requires C, 74.0: H, 4.1%

UV : λ_{max} MeOH 228, 242, 293, 424 nm

IR : V_{max} KBr 1635, 1618, 1452, 1391, 1118, 1020, 970, 862, 740 cm⁻¹

PMR (220 MHz, CDCl₃) δ 9.89 (1H, s, CHO), 9.19 (1H, s, H-1), 7.85 (1H, d, J=3.5 Hz, H-3), 7.77 (1H, d, J=6 Hz, H-6), 7.30 (IH, d, J=6 Hz H-5), 6.55 (1H, d, J=3.5 Hz, H-4)

MS m/e 146(M⁺, 79%), 145 (100), 117 (14), 89 (10), 63 (10), 59 (9).

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