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STUDIES ON *VIBURNUM NERVOSUM* HOOK CHEMISTRY AND SPECTROSCOPY OF BERGENIN AND ITS DERIVATIVES

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ABSTRACT

Bergenin is reported to occur in *Viburnum nervosum* (Capprifoliaceae). This is the first report of its occurrence in genus Viburnum. Derivative of Bergenin *viz* Bergenin diethyl ether, its penta acetate and diethyl ether triacetate have been prepared. Their method of preparation and characterization by spectroscopy is reported for the first time.

INTRODUCTION [1, 2, 3, 4]: *Viburnum nervosum* Hook (Capprifoliaceae) known in Hindi as "Shirporna- jaya" is used in the Indian system of medicine as astringent and emmenagogue. It yields as essential oil, which is used as a potential perfumery material. In Kashmir its roots are used in treatment of acute furunculosis.

Although several known triterpenes, alcohols and flavones have been isolated from this plant, we are the first to report the occurrence of Bergenin (I) from the roots of this plant [2]. Compound (I) is already known to occur in genus *Bergenia* (Saxifragaceae) and has been found to possess use in the treatment of hypercholestraemia, kidney stones, fever, diarrhoea, pulmonary infection, as an antioxidant and anticancer agents.

This paper deals with the isolation preparation and spectroscopy of Bergenin and some of its derivatives.

MATERIAL AND METHODS:

Plant material: The roots of *Viburnum nervosum* were collected from the hilly area Of Srinagar Kashmir and was authenticated by Prof.Shantosha Saraf, Deptt of Botany, Kashmir University. A Voucher Specimen (RLK/VN/77) is preserved in Department of Pharmaceutics, Banaras Hindu University, Varanasi.

Extraction and isolation: The roots were air-dried, powdered to moderately coarse powder and defatted with the petroleum ether (60^{o}-80^{o}C) in a soxhlet apparatus. It was extracted thoroughly with the rectified spirit.

The extract was dried and Chromatographed over SiO_2, eluting the column with $CHCl_3$:MeOH (5:1) as the solvent system, which resulted with separation of homogenous white solid, Bergenin (I), m.p 144^{o}C, soluble in water, alcohol, pyridine, insoluble in organic solvents; crystallizing as colourless rhombohedrons from methanol; $[\alpha]_D$ -47^{o} (H_2O); M^+ 328; found C (%), 51.53; H, 4.47; $C_{14}H_{16}O_9$ requires C, 50.12; H, 4.87; compound is phenolic in nature (ferric chloride bluish green; phosphomolybdic acid & ammonia vapors blue); numbers of double bond equivalents calculated is seven; UV (methanol, nm) 219, 275; IR (KBr, cm^{-1}) 3450,3270(broad, -OH); 2894, 2850(CH_2); 1720(α, β unsaturated δ lactone); 1610, 1530 (aromatic C-O); 1345, 1235 (aromatic C - O), 858(C-H); ^{1}H

NMR(Pyridine-d_5), δ, 3.98 (3H, s , Ar-OCH_3); 4.50-5.81(Complex m , methines, methylenes and hydroxyls); 7.52 (1H, s, Isolated Ar - H). Refluxing Bergenin with ethyl iodide in dry acetone in presence of anhydrous K_2CO_3 furnished di ethyl ether of Bergenin (II), m.p.1760; ^{1}H NMR (pyridine-d_5), δ 1.42,1.50 (2 x 3H, t, each), 4.30 (2 x 2H, q overlapped).

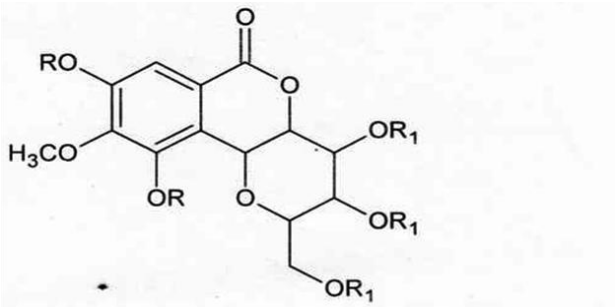
Confirming the presence of two phenolic hydroxyls groups. Bergenin in acetic anhydride and tri-ethylamine at room temperature furnished a penta-acetate (III), m.p. 134^{o}C, ^{1}H NMR ($CDCl_3$) δ, 2.15(6H, s, for two additional acetoxy methyls) proving the presence of three additional nonphenolic secondary alcoholic group in (I). Treatment of (II) with acetic anhydride and tryethylamine at room temperature furnished diethyl ether tri -acetate of *Bergenin* (IV), m.p 170^0, M^+ 510.

Mass spectra of (I) show M^+ at m/z 328 with base peek at m/z 208(V), which corroborates with the correctness of tricyclic structure of *Bergenin*.

Experimental: *Bergenin Diethyl ether* (II) Compound I (20mg) in dry acetone (50ml) with anhydrous K_2CO_3 (5 gms) was refluxed with ethyl Iodide (3ml) for 12 hours contents, were cooled, filtered and acetone was evaporated from the filtrate, the residue was crystallized from methanol as grey colourled needles, m.p 176^0; I.R (KBr, cm^{-1}) 3200–3300 (broad, -OH), 1725 (α β unsaturated - δ- lactone); ^{1}H NMR (Pyridine-d_5) δ, 3.98 (3H, s) Ar-OCH_3; 1.42, 1.50(2x3H, t, each) 4.30 (2x2H, q overlapped) for two ethoxy functions; 4.55-5.75 complex m methines, methylenes and hydroxyls); 7.52 (1H, s, isolated Ar-H).

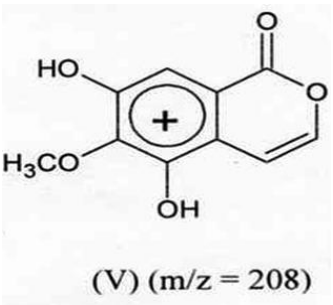
Bergenin penta-acetate (III) A mixture of compound I (10mg), acetic anhydride (3ml) and try ethylamine (1ml) was kept at the room temperature overnight and then poured into crushed ice till the decomposition of acetic anhydride was complete. It was then extracted with chloroform, chloroform layer was washed first with aqueous sodium bicarbonate and subsequently with distilled water. Chloroform extract was dried over anhydrous sodium sulphate and evaporated to dryness to give a residue, which crystallized from methanol as yellowish shinning needles m.p. 134-35^{o}C. I.R (KBr) indicates absence of hydroxyl groups, peaks at cm, $^{-1}$ 1720(α, β unsaturated δ lactone, 1740 (broad, ester carbonyl); PMR ($CDCl_3$ δ) signales at 2.08, 2.12, 2.14 (3H, s each) and 2.15(6H, s) for five acetoxy methyl proton, 3.98(3H, s , Ar-OCH_3); 7.52(1H , s , Isolated Ar-H).

Bergenin di-ethyl ether triacetate (IV) compound II was acetylated by method as given under compound (III), the product crystallizing as white flakes from methanol m.p. 170^{o}C; M^+ 510; found (%) C, 55.76; H 5.62; $C_{24}H_{30}O_{12}$ requires C 56.47; H 5.88; I.R (KBr cm^{-1}) 1720(α, β unsaturated carbonyl), 1740(broad, ester carbonyl); ^{1}H NMR (pyridine-d_5) δ, 2.10, 2.12, 2.13(3H, s, each for the acetoxy methyl 4.00(3H, S, Ar-OCH_3); 4.50-5.82(complex m methines and methylens); 7.53(1H, s, Isolated ArH); mass fragment ions at m/z 264 in figure V, (two, phenolic hydroxyl are replaced by two ethoxy functions), m/z 335(VI).

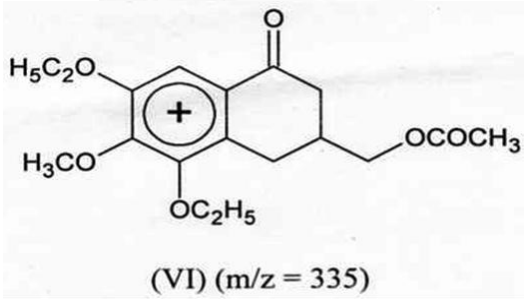


(I) R = R_1 = H (M^+ = 328)
(II) R = C_2H_5, R_1 = H
(III) R = R_1 = -CO-CH_3
(IV) R = C_2H_5, R_1 = -CO-CH_3 (M^+ = 510)

FIGURE OF COMPOUNDS (I-IV)



(V) (m/z = 208)



(VI) (m/z = 335)

FIGURE OF COMPOUND (V) & (VI)

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