

NOTE

A Rare Homoisoflavonoid Cambodianol from *Sansevieria roxburghiana*

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A homoisoflavonoid and a triterpene were isolated from the carbon tetrachloride soluble fraction of a methanol extract of the whole plants of *Sansevieria roxburghiana* (Agavaceae). The structures of the isolated compounds were elucidated as cambodianol (**1**) and lupeol (**2**) by extensive spectroscopic studies, including high field NMR analyses.

Key Words: Agavaceae, Cambodianol, homoisoflavonoid, *Sansevieria roxburghiana*.

Sansevieria roxburghiana (Family-Agavaceae), called Murva in Sanskrit and Hindi, Indian bowstring hemp in English is a herbaceous perennial plant with short fleshy stem and stout rootstock, occurring in eastern coastal region of India, as well as in Sri Lanka, Indonesia and tropical Africa^{1,2}. In India, this plant has been traditionally used for several medicinal purposes. The whole plant is traditionally used as a cardiogenic, expectorant, febrifuge, purgative, tonic, in glandular enlargement and rheumatism³⁻⁵. The rhizomes are mucilaginous and used in consumptive complaints, long lasting chronic persistent coughs, for quick relief of common cough and cold, in ear pain, etc.^{2,5-7}. The juice of tender shoots is administered to children for clearing viscid phlegm from throats. The roots are used as febrifuge in snake bite and hemorrhoids^{6,7}.

The occurrence of steroidal saponins in the genus *Sansevieria* is well documented⁸⁻¹⁰. However, a survey of the literature showed that no chemical studies have been carried out on the constituents of *Sansevieria roxburghiana* (Agavaceae). We, herein, report the isolation of cambodianol (**1**) and lupeol (**2**) from the carbon tetrachloride soluble fraction of a methanol extract of *S. roxburghiana*.

The ¹H NMR spectra were recorded using a Bruker AMX-400 (400 MHz) instrument and the spectra were referenced to the residual nondeuterated solvent signal. PTLC was carried out using Merck Si gel 60 F₂₅₄ on glass plates (20 cm X 20 cm) at a thickness of 0.5 mm. TLC was conducted on normal-phase Merck Si gel 60 F₂₅₄ on glass plates and spots on TLC and PTLC plates were visualized under UV light at 254 nm as

well as by spraying with vanillin sulfuric acid followed by heating for 5 min at 110 °C.

Collection of plant materials: The whole plant sample of *S. roxburghiana* was collected from Dhaka, Bangladesh in the month of October 2009. A voucher specimen for this collection has been deposited in the herbarium of the Department of Botany, University of Dhaka. The samples were cut into small pieces and sun dried for 7 days followed by oven drying for 24 h at 40 °C to facilitate grinding.

Extraction and isolation: The powdered material (533 g) was soaked in 2.5 L of methanol in a large conical flask for 7 days with occasional shaking and stirring. The whole mixture was then filtered off through a cotton plug followed by Whatman filter paper no.1 and the filtrate thus obtained was concentrated at 40 °C with a rotary evaporator. A portion (10 g) of the concentrated methanol extract was fractionated by the modified Kupchan partitioning protocol¹¹ which afforded pet-ether (4.0 g), carbon tetrachloride (2.1 g), dichloromethane (2.1 g) and aqueous (0.8 g) soluble materials.

An aliquot of the carbon tetrachloride soluble partitionate (2.1 g) was fractionated by gel filtration chromatography (GFC) over Sephadex LC-20 using of a mixed solvent system of pet-ether: dichloromethane: methanol (2:5:1) followed by mixtures of methanol and dichloromethane of increasing polarity and finally only with methanol. A total of 100 fractions were collected, each 2 mL. Preparative thin layer chromatography (PTLC) of column fractions eluted with 2:5:1 *n*-hexane:dichloromethane:methanol (column fractions 39-42) over silica gel using toluene-ethyl acetate (65:35) afforded

compound **1** (3.5 mg) and the yield value was 0.07 %. Again, PTLC of column fractions 91 to 96 eluted with dichloromethane: methanol (9:1) over silica gel using 85 % toluene in 15 % ethyl acetate as the developing solvent gave compound **2** (7.0 mg), having the yield value 0.14 %.

Cambodianol (1): Yellowish amorphous crystal; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 11.27 (1H, s, OH-5), 7.06 (1H, $J = 8.4$ Hz, H-2', 4'), 6.75 (1H, $J = 8.4$ Hz, H-3', 5'), 6.06 (1H, s, H-8), 4.97 (1H, s, OH-3), 4.19/4.04 (2H, m, $J = 11.2$ Hz, H-2), 3.87 (3H, s, OMe-4'), 2.94/2.90 (2H, m, $J = 14.0$ Hz, H-9), 2.0 (3H, s, Me-6); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): 72.0 (C-2), 27.2 (C-2), 72.28 (C-3), 198.35 (C-4), 160.19 (C-5), 106.55 (C-6), 166.52 (C-7), 91.01 (C-8), 40.84 (C-9), 100.21 (C-4a), 161.17 (C-8a), 6.91 (C-6Me), 126.40 (C-1'), 131.81 (C-2'), 115.26 (C-3'), 154.84 (C-4'), 115.26 (C-5'), 131.8 (C-6'), 55.98 (C-4'-OMe).

Lupeol (2): Colourless gum; δ 4.68 (1H, br. s, H_a -29), δ 4.57 (1H_b, br. s, H_b -29), δ 3.18 (1H, dd, $J = 11.2, 5.2$ Hz, H-3), δ 1.67 (3H, s, Me-30), δ 1.02 (3H, s, Me-27), δ 0.96 (3H, s, Me-26), δ 0.94 (3H, s, Me-25), δ 0.82 (3H, s, Me-24), δ 0.78 (3H, s, Me-23), δ 0.75 (3H, s, Me-28).

Repeated chromatographic separation and purification of the carbon tetrachloride soluble partitionate of a methanol extract of the whole plant of *S. roxburghiana* provided two compounds, the structures of which were determined by analysis of $^1\text{H NMR}$ and $^{13}\text{C NMR}$ spectral as well as by comparison with previously reported values.

After comparing the $^1\text{H NMR}$ and $^{13}\text{C NMR}$ data of compound **1** (Fig. 1) with those existing in the literature indicated that the compound is a homoisoflavonoid [(3R)-3,5,7-trihydroxy-6-methyl-3-(4'-methoxybenzyl)-4-chromanone] namely cambodianol¹².

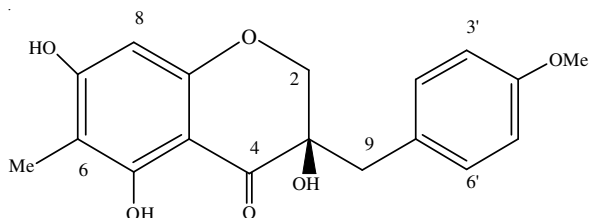


Fig. 1. Compound **1**, Cambodianol

In the $^1\text{H NMR}$ spectrum four proton signals at δ 4.19, 4.04, 2.94 and 2.90 and four carbon signals in the $^{13}\text{C NMR}$ spectrum at δ 71.99, 72.28, 198.35 and 40.84 indicated the presence of 3-hydroxy-3-benzyl-4-chromanone.

Four proton signals were observed in the $^1\text{H NMR}$ spectrum at δ 4.19 (1H, d, $J = 11.2$ Hz, H_a -2), 4.04 (1H, d, $J = 11.2$ Hz, H_b -2), 2.94 (1H, d, $J = 14.0$ Hz, H_a -9) and 2.90 (1H, d, $J = 14.0$ Hz, H_b -9). $^{13}\text{C NMR}$ spectrum showed four carbon signals at δ 71.99 (t, C-2), 72.28 (s, C-3), 198.35 (s, C-4) and 40.84 (t, C-9). A AA:BB: spin system at δ 7.06 (2H, d, $J = 8.4$ Hz, H-2) and 6.75 (2H, d, $J = 8.4$ Hz, H-3) in the $^1\text{H NMR}$ spectrum indicated the C-4 in ring B was oxygenated. In addition, an aromatic proton at δ 6.06 (1H, s, H-8), a methyl signal δ 2.00 (3H, s, H-6) and a methoxyl signal δ 3.87 (3H, s, H-4) were observed in the $^1\text{H NMR}$ spectrum. Their locations were deduced by HMBC experiment. The methoxy group was

connected with C-4 on the basis of HMBC correlations between the 3H singlet (δ 3.87) and the carbon at δ 154.84 (C-4). The other three aromatic carbons with oxygen function were observed at δ 160.19 (C-5), 166.52 (C-7), 161.17 (C-8a) and in the $^{13}\text{C NMR}$ spectrum, which indicated that ring A was a phloro-glucinol ring.

In the HMBC spectrum, both the aromatic proton (δ 6.06, s, 1H) and the two protons of C-2 showed correlations with the carbon at δ 161.17 (C-8a), which indicated that the aromatic proton in ring A belonged to C-8; therefore, the methyl group (δ 2.00, s, 3H) was attached to C-6. The configuration at C-3 was proposed to be R, similar to the homoisoflavonoid Cambodianol. Homoisoflavonoids are less frequently reported from plant origin. Cambodianol is a new homoisoflavonoid which was previously once reported from *Dracaena cambodiana* of Agavaceae family. This is the second report of its occurrence from any plant and first report from *S. roxburghiana*.

On the basis of comparison of $^1\text{H NMR}$ data with previously reported values allowed to characterize compound **2** as lupeol¹³. Again the identity of **2** as lupeol was further confirmed by co-TLC with an authentic sample.

Conclusion

The present phytochemical study of the carbon tetrachloride soluble fraction of the methanol extract of *S. roxburghiana* afforded an isoflavone and one triterpene, the structure of which were established as cambodianol (**1**) and lupeol by extensive spectroscopic studies as well as by comparison with published result.

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