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QUINONES AND OTHER CONSTITUENTS FROM *PHYLLARTHRON COMORENSE*

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Key Word Index—*Phyllarthron comorense*; Bignoniaceae; quinones; lapachol; sesamin; tectol.

Plant. Phyllarthron comorense DC. Voucher specimen No. 11039 deposited in the R.U.B.L. Herbarium. *Previous work.* No work has been reported on this species. On sister species *P. madagascariensis.*[1]

Present work. The coarsely powdered heartwood (2 kg) was completely exhausted with hot petrol. (60–80°). The extract was concentrated to dryness, taken in Et_2O, extracted with 2 N Na_2CO_3 and acidified with 2 N HCl.[2]

Acidic component. The acidic fraction was chromatographed over silica gel. Benzene–light petrol. (9:1) gave compound I, fine yellow needles, m.p. 139–140° (benzene); (Found: C, 74·30; H, 5·77. Calc. for $C_{15}H_{14}O_3$: C, 74·38; H, 5·78%); ν_{max} (Nujol) 3350, 1660, 1630, 1580 cm^{-1}. These data suggested that compound I was lapachol and this was confirmed by comparison with an authentic specimen (co-TLC, IR and m.m.p.).

Neutral components. The neutral fraction was chromatographed over deactivated alumina and afforded compounds II, III, IV, V, VI, VII, VIII and IX.

Compound II. From light petrol. (100%), blue green crystals, m.p. 194–195° (MeOH); (Found: C, 79·80; H, 6·03, Calc. for $C_{30}H_{24}O_4$: C, 80·35; H, 5·35%); ν_{max} (Nujol) 1645 (C = O) cm^{-1}; λ_{max} (EtOH) 271, 340 nm. From the above data, compound II appeared to be dehydrotectol and this was confirmed (co-TLC, IR and m.m.p.).

Compound III. From light petrol.–benzene (9:1), orange needles, m.p. 142–143° (50% benzene and light petrol.); (Found: C, 74·92; H, 4·87. Calc. for $C_{15}H_{12}O_3$: C, 75·00; H, 5·00%); ν_{max} (Nujol) 1690, 1640, 1632, 1598 cm^{-1}; M^+ 240. These data indicated that compound III was dehydro-α-lapachone and this was confirmed (co-TLC, IR and m.m.p.).

Compound IV. From light petrol.–benzene (4:1), orange needles, m.p. 154–155° (benzene); (Found: C, 74·65; H, 5·30. Calc. for $C_{15}H_{14}O_3$: C, 74·38; H, 5·78%); ν_{max} (Nujol) 1690, 1632, 1598 cm^{-1}; λ_{max} (EtOH) 256, 282, 330, 431 nm. From the above data, compound IV seemed to be β-lapachone which was confirmed (co-TLC, IR and m.m.p.).

Compound V. From light petrol.–benzene (3:2), colourless prisms, m.p. 122–123° (50% benzene and light petrol.); (Found: C, 68·14; H, 5·20. Calc. for $C_{20}H_{18}O_6$: C, 67·80; H, 5·09%); ν_{max} (Nujol) 1610, 1255, 1185, 1100, 1060, 1040, 930, 855, 790 cm^{-1}. These data indicated compound V to be D-sesamin (co-TLC, IR and m.m.p.).

Compound VI. From light petrol.–benzene (1:1), colourless granules, m.p. 216–218° (MeOH); (Found: C, 79·06; H, 5·92, Calc. for $C_{30}H_{26}O_4$: C, 79·64; H, 5·77%); ν_{max}

[1] R. PERNET, *Bull. Acad. Malgache (Tananarive, Madagascar)* **34**, 47 (1956).
[2] PAHUP SINGH, LALIT PRAKASH and KRISHNA C. JOSHI, *Phytochem.* **11**, 1498 (1972).

(Nujol) 3430, 1560, 1225, 1180, 1135; 1070, 1010, 770, 690 cm^{-1}. From the above facts compound VI appeared to be tectol and this was confirmed (co-TLC, IR and m.m.p.).

Compound VII. From light petrol.–benzene (2:3), colourless prisms, m.p. 84–85° (MeOH); (Found: C, 62·70; H, 5·45. Calc. for $C_{21}H_{22}O_8$: C, 62·68; H, 5·51%); ν_{max} (Nujol) 3200–3500, 1610, 1260, 1240, 1040, 940, 815, 750 cm^{-1}. These data suggested that compound VII was paulownin methanolate which was confirmed (co-TLC, IR and m.m.p.).

Compound VIII. From light petrol.–benzene (2:3), colourless needles, m.p. 104–105° (50% benzene and light petrol.); $C_{20}H_{18}O_7$; ν_{max} (Nujol) 3300 (br), 1610, 1260, 1240, 1040, 940, 815, 750 cm^{-1}. These data indicated that compound VIII was paulownin this was confirmed with an authentic specimen (co-TLC, IR and m.m.p.).

Compound IX. From benzene (100%), colourless flakes, m.p. 136–137° (MeOH); $C_{29}H_{50}O$; ν_{max} (Nujol) 3400 (OH) cm^{-1}; acetate (pyridine–Ac_2O), colourless needles, m.p. 126–127° (50% MeOH and $CHCl_3$). From the above data, compound IX was sitosterol.

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TRITERPENOIDS OF *BUXUS PAPILLOSA*

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Plant. Buxus papillosa C.K. Schn. *Uses.* None. *Previous work.* Several alkaloids[1] and unidentified steroids[2] have been isolated from the leaves and stems of the plant.

Present work. Leaves and stems of the plant have been examined. Extraction: The shade dried whole plant (4 kg) was extd with 95% EtOH (30 l.) at room temp. The concentrated extract was macerated with 5% HOAc and kept at 2° for 2 days and filtered. The residue was macerated with $CHCl_3$ and treated with charcoal to remove chlorophyll. The solution on concentration deposited lightly coloured crystalline mass which crystallized from MeOH, but showed the presence of small amounts of an impurity on TLC which was removed by column chromatography over alumina (50 g, B.D.H.) in $CHCl_3$. The product obtained crystallized from EtOH to give needles, $C_{30}H_{50}O$, m.p. 215°, $[\alpha]_D$ +27·0° (*c* 5·0, $CHCl_3$) (Found: C, 84·51; H, 11·79 Calc. for $C_{30}H_{50}O$; C, 84·44, H, 11·91%). IR (Nujol) ν 3390,

[1] M. IKRAM, G. A. MIANA, F. SULTANA and F. MAHMUD, *Pak. J. Sci. Ind. Res.* **11**, 488 (1968).
[2] M. IKRAM, G. A. MIANA, F. MAHMUD and M. ISRAR KHAN, *Pak. J. Sci. Ind. Res.* **12**, 201 (1970).