ISOLATION OF 3,4,3′,5′-TETRAHYDROXYSTILBENE (PICEATANNOL) FROM *CASSIA MARGINATA* HEARTWOOD*

By V. S. Sundara Rao† and S. Rajadurai†

Cassia marginata Roxb., of the family Leguminosae (vakai: Tamil; kadakonna: Malayalam), is a small tree grown in the forests from South Arcot to Travancore (India) and often planted for ornament.[1] The present communication describes the isolation and identification of 3,4,3′,5′-tetrahydroxystilbene for the first time from *Cassia marginata* heartwood. Earlier this stilbene was isolated by King *et al.*[2] from *Vouacapoua macropetala* and by Grassmann *et al.*[3,4] from spruce bark. The structure of this compound was established by Cunningham *et al.*[5]

Experimental

The heartwood collected from a mature tree on the grounds of the Central Leather Research Institute, Madras, was used in the experiments.

Preparation of Acetone Extract

Powdered heartwood (2 kg) was extracted with acetone (4 l.) for 48 hr at room temperature. Then the acetone solution was decanted off and a fresh lot of acetone was added and the process continued till the extraction was complete. The combined acetone extract was then filtered and distilled in vacuum (40°; nitrogen atmosphere) to dryness. The solid brown residue (75 g) obtained was phenolic but not hygroscopic.

Ether Solubles of Acetone Extract

Acetone extract (20 g) was repeatedly extracted with ether. The ether solution on evaporation left a solid (2 g) which was subjected to cellulose column chromatography for the separation of 3,4,3′,5′-tetrahydroxystilbene as described below.

Preparation of Cellulose Column

A glass column (90 by 3 cm) was filled with an aqueous suspension of Whatman ashless cellulose powder up to a length of 65 cm.

The ether solubles (1 g) in methanol (5 ml) were placed on top of the column. After the initial adsorption of the methanol phase, the column was successively eluted with 6% acetic acid (250 ml) to give fraction I and with ethyl alcohol (200 ml) to obtain fraction II.

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† Central Leather Research Institute, Madras 20, India.

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3,4,3′,5′-Tetrahydroxystilbene from Fraction II

Fraction II on evaporation in vacuum (nitrogen atmosphere) gave a residue (0·5 g) which on crystallization from water gave 3,4,3′,5′-tetrahydroxystilbene (piceatannol), m.p. and mixed m.p. 229° (dec.), R_F 0·03 (6% acetic acid), R_F 0·80 (butan-2-ol–acetic acid–water (14 : 1 : 5 v/v) (Found: C, 68·85; H, 5·1. Calc. for $C_{14}H_{12}O_4$: C, 68·8; H, 4·95%).

3,4,3′-5′-Tetrabenzoyloxystilbene

The tetrabenzoate was prepared with benzoyl chloride and pyridine and crystallized from acetone–methanol as needles, m.p. 154° (lit.[5] 153–154°) (Found: C, 75·9; H, 4·5. Calc. for $C_{42}H_{28}O_8$: C, 76·3; H, 4·3%).

3,4,3′,5′-Tetramethoxystilbene

The tetramethyl ether was prepared using excess of ethereal diazomethane and crystallized from aqueous methanol, m.p. 66° (lit.[5] 64–66°) (Found: C, 71·8; H, 6·5. Calc. for $C_{18}H_{20}O_4$: C, 72·0; H, 6·7%).

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