5-Hydroxy-8-methoxy-3:7:4'-tribenzyloxy-flavone (IV)

The 5:8-dihydroxy combound (III) ($0.8 \, \mathrm{g}$.) was dissolved in dry acetone (25 c.c.) and anhydrous potassium carbonate ($5.0 \, \mathrm{g}$.) and dimethyl sulphate ($0.14 \, \mathrm{c.c.}$, 1 mol.) were added. The mixture was refluxed for 6 hours. The potassium salts were filtered off and washed with hot acetone. The filtrate was concentrated over a water-bath to remove acetone. The solid that remained behind was stirred up with water and filtered. It crystallised from a mixture of alcohol and acetone as pale yellow prismatic needles melting at 113–15° (Found: C, 75.5; H, 5.0; $C_{37}H_{30}O_7$ requires C, 75.8; H, 5.1%). It gave a green colour with alcoholic ferric chloride and was sparingly soluble in aqueous alkali.

8-Methoxy-5:7:3:4'-tetrahydroxy-flavone (V) (Tambuletin)

The above 5-hydroxy compound (0.5 g.) was treated with a mixture of glacial acetic acid (5.0 c.c.) and concentrated hydrochloric acid (2.5 c.c.) and the mixture was heated at 100° over a water-bath for one hour. The solution was cooled and diluted with water. A bright yellow solid separated out. It was filtered and crystallised from glacial acetic acid whereby it was obtained as bright yellow short needles melting at 269-70°. It gave a dull green colour with alcoholic ferric chloride and was readily soluble in aqueous alkali to give a bright yellow solution. It gave an orange-red precipitate with neutral lead acetate in alcohol. The mixed melting point with a sample of tambuletin isolated from the seeds of Zanthoxylum acanthopodium was not depressed (Found: C, 60.3; H, 4.0; $C_{16}H_{12}O_7$ requires C, 60.7; H, 3.8%).

SUMMARY

The synthesis of tambuletin has been effected. Kæmpferol is first benzylated to the tribenzyl-ether which is subjected to oxidation with alkaline persulphate, partial methylation and debenzylation in succession.

REFERENCES

1. Balakrishna and Seshadri .. Proc. Ind. Acad. Sci., A, 1947, 25, 449.

Ibid., 1947, 26, 72.
Rao, Rao and Seshadri .. Ibid., 1947, 26, 13.

ERRATUM

In Part III, A, 1947, 26, 216 read (VIII), for (XIII) at the bottom of the page.