

by dissolving it in diluted HBr and standing in a refrigerator, the latter was yellow needles having mp 157° (decomp.) and its IR spectrum also had the band of 1650 cm⁻¹. Elementary analysis of the hydrobromide gave the values of C, 36.79; H, 4.46; N, 9.73.

The final determination of the structure was carried out by X-ray crystallography using the hydrobromide. The determined structure of the hydrobromide is shown as (I) and the phosphate is presumed to have the structure of (II).

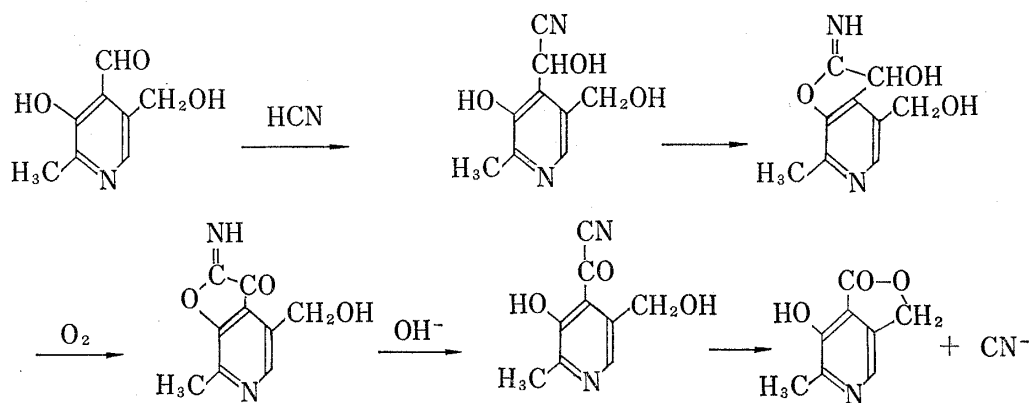


Chart 2

The calculated percentages for the elements of the hydrobromide are C, 36.87; H, 4.47; N, 9.59, and those of the phosphate are C, 40.00; H, 5.32; N, 10.37 and its molecular weight is 270.21. These values are in good agreement with those found in the experiments. The 1650 cm⁻¹ band of the IR spectrum was thought to be due to the imino group. The mechanism of the reaction of PAL with cyanide is presumed as shown in Chart 2.

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Isolation of Insect Moulting Substances from *Pleopeltis thunbergiana*, *Neocheiropteris ensata*, and *Lemmaphyllum microphyllum*

Recent survey of insect moulting substances in the plant kingdom has revealed the wide distribution of a variety of steroids possessing the moulting hormone activity.¹⁾ As part of our study on insect moulting substances from plant sources, we have screened a number of vegetable materials by means of bioassay and found that the methanol extracts of the whole plants of *Pleopeltis thunbergiana* KAULF., *Neocheiropteris ensata* CHING, and *Lemmaphyllum microphyllum* PRESL²⁾ (Polypodiaceae) grown in Japan have proved positive. Then the polar fraction of each extract was submitted to column chromatography leading to the isolation of the following substances: ecdysterone, mp 243—245°, from *P. thunbergiana*, ecdysterone, mp 246—250°, from *N. ensata*, and ecdysterone, mp 245—249°, and a novel steroid

1) cf. T. Takemoto, Y. Hikino, T. Arai, M. Kawahara, C. Konno, S. Arihara, and H. Hikino, *Chem. Pharm. Bull.* (Tokyo), **15**, 1815 (1967).

2) Japanese names: nokishinobu, kuriharana, and mamezuta, respectively.

with moulting hormone activity, mp 258—259°, which is now designated as lemmasterone, from *L. microphyllum*. Although the ecdysterones isolated from *N. ensata* and *L. microphyllum* are identical with authentic ecdysterone in behavior on thin-layer chromatography (showing a single spot) and in their infrared and nuclear magnetic resonance spectra, their melting points are rather higher and of wider range than authentic ecdysterone. Acetylation of each ecdysterone produced, along with ecdysterone triacetate, a small amount of a less-polar acetylated impurity, existence of a similar substance in each plant being indicated.

In the insect (*Sarcophaga*) test, lemmasterone exhibits the high moulting hormone activity. Lemmasterone, as with the other insect-moulting substances (*i.e.*, ponasterone A, pterosterone, ecdysterone, inokosterone, and cyasterone),³⁾ shows also the high accelerating effect on the protein anabolism in mouse liver.

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A Mangiferin Monomethyl Ether from *Mangifera indica* L.

Mangiferin, a xanthone C-glycoside first isolated from *Mangifera indica* L. and later found in several different families of plants,¹⁻³⁾ has been studied extensively by many research groups,^{1,4-8)} and the structure has quite recently been established as 2-C- β -D-glucopyranosyl-1,3,6,7-tetrahydroxyxanthone. However, so far none of the corresponding methyl ethers in nature has been reported.⁹⁾

A new compound which was isolated from a sample of mangiferas has now been found to be one of them, named homomangiferin (I), and characterized as mangiferin 3-methyl ether.

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- 9) P.E. Nott and J.C. Roberts¹⁾ have reported that mangiferin, mp 270° (decomp.), obtained from mango bark (*Mangifera indica*) showed two spots (*Rf* 0.4 (major), and 0.6) on thin-layer of "Kieselgel H nach Stahl" (Merck) but that attempted separation of the components failed even by column chromatography on "Polyamidpulver nach Stahl" (Merck).