

ISOLATION OF SIMPLE ACID AMIDES FROM *ALLOPHYLUS COBBE*
(SAPINDACEAE), *HOMALIUM FOETIDUM* (FLACOURTIACEAE),
AND FROM AN *AGLAIA* SPECIES (MELIACEAE)

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Simple acid amides have been isolated from three unrelated species which have been examined in the course of a survey of the New Guinea flora for the presence of alkaloids. The method of extraction was aimed primarily at the isolation of alkaloids, and consequently accurate yields of the amides cannot be given as it may be assumed that at least some of the amides present were discarded with other non-basic constituents.

Several samples of leaves and bark of *Allophylus cobbe* (L.) Raeusch. gave negative tests for alkaloids when tested in the field, but laboratory assay of the leaves indicated the presence of traces of alkaloids. As no alkaloids have been previously isolated from plants of the family Sapindaceae, a large-scale extraction of the leaves was carried out, but the crude "alkaloid" fraction was found to be an extremely complex mixture consisting mainly of non-alkaloidal material. Extensive chromatography on alumina led to the isolation of only one crystalline constituent; this has been identified as phenylacetamide.

The leaves of *Homalium foetidum* (Roxb.) Benth. (family Flacourtiaceae) gave a positive field test for alkaloids, but extraction gave a crude "alkaloid" fraction from which only benzamide was isolated by chromatography on alumina. Further investigation of this species may be warranted because of the positive field test for alkaloids and because of the isolation of an unusual alkaloid homaline from the African species *Homalium alnifolium* reported recently.¹ Benzamide has been previously reported to occur as a plant constituent in *Lophira lanceolata* (family Ochnaceae).²

Because of taxonomic relationships to the families Rutaceae and Simaroubaceae, of which the former has been widely studied for alkaloids, any isolation of alkaloids from a plant of the family Meliaceae would be of interest. The leaves of an *Aglaia* species, which gave a positive field test for alkaloids, were therefore extracted. No alkaloids were isolated from the crude "alkaloid" fraction, but tiglamide was identified as a major constituent.

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¹ Pais, M., Rattle, G., Sarfati, R., and Jarreau, F.-X., *C. r. hebd. Séanc. Acad. Sci., Paris, (C)*, 1968, **266**, 37.

² Persinos, G. J., Quimby, M. W., Mott, A. R., Farnsworth, N. R., Abraham, D. J., Fong, H. H. S., and Blomster, R. N., *Planta med.*, 1967, **363**.

Experimental

Milled, dried plant material was extracted by continuous percolation with ethanol at 40°, and a crude "alkaloid" fraction isolated from the concentrate by the method previously described.³

(a) *Allophylus cobbe* (L.) *Raesch.*

Leaves of *A. cobbe* were collected from a tree (40 ft high, 1 ft diameter) on the border of rain forest on the Huon Gulf (Voucher specimen TGH 12,182). The crude fraction, which was obtained by re-extraction from chloroform solution into 2N dilute sulphuric acid and should have contained alkaloids, amounted to 0.03% of the leaves. Chromatography of this material on alumina gave a series of fractions, most of which were shown to be very complex mixtures by thin-layer chromatography. A few fractions, eluted from the column by mixtures of benzene and chloroform, became partly crystalline on standing, and crystallization from benzene afforded phenylacetamide, m.p. 157–158°, which was identified by its spectroscopic properties and by comparison with authentic phenylacetamide.

(b) *Homalium foetidum* (Roxb.) *Benth.*

Leaves were collected from a tree (approximately 100 ft high by 3 ft diameter) growing in rain forest near Lae, New Guinea (Voucher specimen TGH 10,122). Extraction of the leaves gave an alkaloid fraction in 0.06% yield, but titration indicated the presence of only 0.008% of basic material, on the assumption of a mean molecular weight of 250. Chromatography on alumina gave a series of crystalline fractions from which benzamide, m.p. 128–129°, was obtained on recrystallization from benzene.

(c) *Aglaia* *Species*

Leaves of the *Aglaia* species were collected from a tree (20 ft high by 3 in. diameter) growing in rain forest near Lae, New Guinea. Only the generic name is available for this species, which was given the reference number TGH 10,315 by the collector Dr T. G. Hartley, and a specimen has been retained at the herbarium at Lae, New Guinea. Extraction of the dried leaves on a 20-kg scale gave a 0.10% yield of crude alkaloid, but titration indicated the presence of about 0.03% of basic material (with an assumed molecular weight of 250). Chromatography on alumina gave a series of crystalline fractions (50% of the total crude base fraction) from which tiglamide was readily isolated. After purification by sublimation, tiglamide was obtained as colourless crystals, m.p. 73–74°, with a pervasive unpleasant odour. Tiglamide was characterized by its i.r. spectrum (ν_{\max} 1640, 1680, 3200 cm^{-1} in CCl_4), n.m.r. spectrum, mass spectrum (molecular ion at m/e 99), and by the formation of tiglic acid on mild alkaline hydrolysis.

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³ Johns, S. R., Lamberton, J. A., and Sioumis, A. A., *Aust. J. Chem.*, 1966, **19**, 2331.