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Isolation and identification of the basic components of Aristolochia macedonica

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Magnoflorine was isolated and identified as the styphnate (m.p. 235°) and the iodide (m.p..245°), and by thin-layer chromatography.

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Magnoflorine, a quarternary base with aporphinic structure, occurs in many plants. It was isolated for the first time from the bark of *Magnolia grandiflora* in 1954 by Nakano (1), who also determined its structure (2). Later it was isolated from many plant species, in some of them as the primary basic component and in others as the secondary one.

It was the aim of this work to isolate the quarternary bases from the roots of *Aristolochia macedonica.*

In several *Aristolochia* species the basic components have been investigated by Pilarczyk (3), Pailer and Pruckmayer (4), Coutts *et al.* (5),Priestap *et al.* (6) and others. They found that magnoflorine was the main quarternary base.

For the isolation of magnoflorine a number of methods have been described. We tried the method by Pailer and Pruckmayer, using an acidic ion-exchange column to separate the basic components, as well as two precipitation methods — one by Coutts *et al.* (5) using reineckate, the other by Priestap *et al.* (6) using styphnic acid as precipitans. However, none of these methods gave satisfactory results. We therefore used a modified procedure based on the last two methods.

EXPERIMENTAL

Melting points are uncorrected.

A. macedonica was collected on Mt. Kitka in the flowering season.

The dried roots, 500 g, previously defatted with light petroleum (b. p. 40—70°) were extracted with methanol 70 h in a Soxhlet apparatus. The solvent was evaporated under reduced pressure leaving a dark brown syrup. The syrup was suspended in water and ammonia (pH = 9.7) and the tertiary bases were extracted with ether. To the aqueous layer HCl was added and the acids were extracted with ether. The aqueous layer was concentrated and ammonium reineckate was added; the precipitate was col-

lected on a filter and dried. A 3-g portion was extracted with acetone; 1 g of the solid remained undissolved. The reineckate was decomposed by addition of Ag_2SO_4 and $BaCl_2$ to the solution. The silver reineckate and $BaSO_4$ were removed by filtration, and styphnic acid was added to the filtrate. The precipitated styphnate (200 mg) was collected and recrystalized several times from acetone, giving a product with m. p. 235°.

Anal. $C_{26}H_{26}N_4O_{12}$ (586.60): Calcd.: C, 53.15; H, 4.63; N, 9.54;
Found: C, 53.50; H, 5.00; N, 9.36.

Magnoflorine styphnate (100 mg) was dissolved in 10 ml acetonewater (1 : 1) and passed through an Amberlitte-400 (HCO_3^-) column. The column was eluted using the same solvent, and the combined fractions containing the eluate were evaporated. Sodium iodide in ethanol was added, whereupon 30 mg magnoflorine iodide were obtained. This product was recrystalized from methanol to give crystals of m. p. 245°.

Thin-layer chromatography of the iodide on silica gel GF_{254} plates using EtOH—H_2O—NH_4OH (15 : 9 : 1), when compared with an authentic sample of magnoflorine iodide gave the same R_F value = 0.37.

The advantage of the described method over those previously mentioned (5, 6) is that magnoflorine was isolated in better yield as well as in a purer form.

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ИЗВОД

Изолација и идентификација на базите составни делови од *Aristolochia macedonica*

Бојан ПОДОЛЕШОВ и Зоран ЗДРАВКОВСКИ

Испитувана е изолацијата на базните компоненти од *A. macedonica.* Успешно е изолиран магнофлоринот кој е идентификуван како магнофлорин стифнат (т. т. 235°) и магнофлорин јодид (т. т. 245°). За неговата идентификација користена е и танкослојна хроматографија.

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