ALKALOIDS OF *Delphinium dictyocarpum*

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Continuing a study of the alkaloids of the epigeal part of *Delphinium dictyocarpum* DC., collected in the environs of the village of Topolevka in the flowering phase [1, 2], we have isolated a new base (I) with mp 116-118°C (acetone).

The IR spectra of (I) has absorption bands at (cm^{-1}) 3460 (hydroxy groups), 1595 (aromatic ring), 1705 (carbonyl group), 1690 (ester grouping), and 1090 (ether C–O bonds). The NMR spectrum shows signals from the methyl part of an ethyl group (three-proton triplet at 1.03 ppm), an acetyl group (three-proton singlet at 2.20 ppm), from three methoxy groups (three-proton singlets at 3.21, 3.31, and 3.33 ppm), and from four aromatic protons (one-proton multiplets at 7.07, 7.53, 7.93, and 8.66 ppm), and a one-proton broadened singlet at 10.93 ppm. The mass spectrum contained, in addition to the peak of the molecular ion, peaks of the ions M–15, M–17, M–18, M–31 (100%), and M–33.

The features of the IR, NMR, and mass spectrum make it possible to assign (I) to the diterpene alkaloids with a lycoctonine skeleton.

The saponificiation of (I) gave an amino alcohol $C_{24}H_{39}O_7N$ (II) with mp 167-169°C (hexane–acetone) and anthranilic acid. A direct comparison of (II) with the amino alcohol delectine [2] showed that they were identical.

A comparison of the NMR spectrum of lappaconitine [3], in which the amino alcohol is acylated with acetylanthranilic acid, with that of (I) showed their complete identity in the region of aromatic protons. In both compounds there are signals at 10.96 and 10.93 ppm from the N proton of a $-\underline{NH}COCH_3$ group. These facts show that in both cases the ester grouping includes an acetylanthranilic acid residue which in the saponification of (I) is decomposed to anthranilic acid. The presence of an acetylanthranilic fragment in (I) is confirmed by the presence in its NMR spectrum of the signal of an acetyl group, and the presence in the mass spectrum of the peak of the molecular ion M^+ 614 and a strong peak of an ion with m/e 162 due to the acyl residue of the acetylanthranilic acid.

The absence of a characteristic signal from C_{10}–H in the weak-field region of the NMR spectrum of (I) [4] and its mass spectrum, which is characteristic for lycoctonine alkaloids with an acyl residue at C_{19} [2, 5], shows that the new base that we have isolated is N-acetyldelectine (I).

Separation of the combined alkaloids of the roots of *Delphinium dictyocarpum* collected on the R. Kuyandysai in the budding period yielded methyllycaconitine, anthranoyllycoctonine, and a base $C_{24}H_{39}O_7N$ (III) with mp 98-100°C (hexane–acetone).

The IR spectrum of (III) shows absorption bands at 3455 cm^{-1} (hydroxy groups) and 1104 cm^{-1} (ether C–O bonds). The NMR spectrum has the signals due to a tertiary methyl group (three-proton singlet at 0.91 ppm), the methyl part of an ethyl group (three-proton triplet at 0.98 ppm), and of three methoxy groups (three-proton singlets at 3.17, 3.25, and 3.35 ppm). In the mass spectrum of (III), the maximum peak is that of the ion M–31.

The characteristics of the IR, NMR, and mass spectrum of (III) permit it to be assigned to the diterpene alkaloids with a lycoctonine skeleton and are practically identical with those of demethyleneeldelidine [6]. A direct comparison of the two compounds showed their identity.

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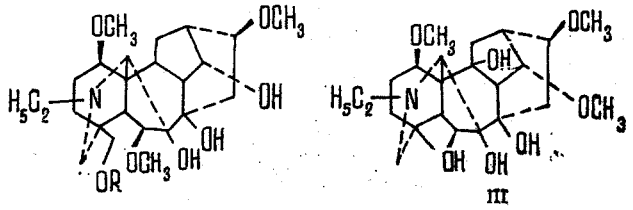
This is the first time that demethyleneeldelidine has been isolated from plants.



I. R=N-acetylanthranoyl
II. R=H
Delectine, R= anthranoyl

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METHOD OF DETERMINING PHYTIN IN RICE FLOUR

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Phytin — a mixture of the calcium and magnesium salts of inositol hexaphosphate — is the main phosphorus-containing reserve compound of higher plants [1]. For the sorting standardization of rice flour for its phytin content, a method for its quantitative determination [2] is used that is based on the precipitation of the phytin from an extract with lead nitrate, which does not always give satisfactory results. We have developed a simple method of quantitative determination of phytin in brans.

Phytin is obtained by extracting rice flour with 1% nitric acid and formalin is added to the acid with the aim of preventing fermentation. Under laboratory conditions, we isolated phytin from rice flour and, in parallel, from cottonseed meal with 1% nitric acid without the addition of formalin. No decrease in the amount of phytin and, consequently, no fermentation process was observed even after 4-5 days in the case of the rice flour, but in the cottonseed meal (after the extraction of the neutral lipids and phospholipids) fermentation took place (2-3 days) and the yield of phytin fell sharply.

Having shown the high solubility of phytin in 1% nitric acid containing 20% of ethanol, we extracted the phytin with this mixture, which ensured the rapid filtration of the meal. To clarify the phytin extract it was passed through a filter mass, and the filtrate was precipitated by adding concentrated ammonia solution to pH 7.0-7.5. At pH 8 and above, the phytin obtained is dark-colored. The precipitate of phytin was purified by reprecipitation and was dried in a drying chest at 80-100°C. The main index of the quality of phytin is its phosphorus content, which, calculated as phosphorus pentoxide, must not be less than 39%.

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