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ARUNDINE — A NEW DIMERIC ALKALOID FROM THE ROOTS OF *Arundo donax*

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The alkaloids of *Arundo donax* growing in Central Asia were studied by A. P. Orekhov and S. S. Norkina and then by K. Ubaidullaev [1-3].

We have studied the alkaloid composition of *A. donax* cultivated in the Tashkent Botanical Garden, Academy of Sciences of the Republic of Uzbekistan.

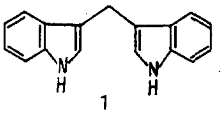
Chloroform extraction of the root of this plant showed that it contained 0.4% of alkaloids. Chromatography on a column of alumina of the ether fraction of the total bases led to the isolation of the alkaloid donaxine, of known structure, and a new base with mp 164-165°C, composition $C_{17}H_{14}N_2$, which we have called arundine.

Arundine (1) is a white crystalline substance moderately soluble in organic solvents. Its IR spectrum contained an absorption band of active hydrogen (3400 cm^{-1}) and bands of the stretching and deformation vibrations of a substituted aromatic ring at 1600, 1490, 800, and 750 cm^{-1}.

The mass spectrum of (1) revealed the 100% peak of the molecular ion with *m/z* 246 and peaks of lower intensity with *m/z* 130, 96, 75, and 51 formed through the fragmentation of an indole aromatic ring. The ion with *m/z* 130 had the elemental composition C_9H_8N, determined by high-resolution mass spectrometry, and corresponded to structure (2).

The PMR spectrum of the alkaloid contained a broadened singlet with its center at 3.85 ppm and an intensity of two proton units from a CH_2 group linked to an aromatic ring. The signals from ten aromatic protons were detected in the 6.85-7.65 ppm region.

The facts given above, together with spectral characteristics, showed that arundine has the structure (1):



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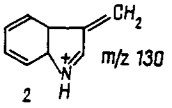

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The structure proposed for arundine was confirmed by a published synthesis [4] from donaxine N-oxide: heating the later in boiling water led to 3,3′-diindolylmethane (1). The arundine synthesized was, in terms of R_f in a thin layer, mixed melting point, and spectral characteristics, identical with the natural product.

IR spectra were taken on a UR-20 spectrometer (tablets with KBr), mass spectra on a MKh-1310 spectrometer with a system for direct injection into the ion source, and PMR spectra on a Tesla BS 567A spectrometer in deuteromethanol. The chloroform—methanol (9:1) system was used for TLC.

Isolation and Separation of the Total Alkaloids from the Roots of *Arundo donax*. The comminuted roots (1.5 kg), previously moistened with 5% ammonia solution, were extracted exhaustively with chloroform. The working up of the combined and concentrated chloroform extract gave 3.64 g of an ether fraction and 1.66 g of a chloroform fraction of total alkaloids. The ether fraction of bases was chromatographed on a column of alumina (1:30). The alkaloids were eluted with chloroform and with chloroform—methanol in various proportions. The chloroform eluates yield 16 mg of a base with mp 164-165°C, R_f 0.15 (chloroform—methanol (9:1)) (arundine).

IR spectrum (cm^{-1}): 3400, 1600, 1490, 1350, 1095, 1050, 800, 750.

Mass spectrum, *m/z* (%): 246 (M^+, 100), 130 (30), 96, 75, 51.

PMR spectrum (δ, ppm): 3.85 (2H, s, CH_2); 6.85-7.65 (10H, m, H-Ar).

The chloroform (9:1) fraction yielded 0.9 g of donaxine.

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Synthesis of Arundine. A suspension of 1.74 g of donaxine in 4 ml of ethanol was treated with 2.82 ml of perhydrol, and the mixture was left in the dark at room temperature. After a day, 1.85 g of donaxine N-oxide with mp 132-135°C, R_f 0.15 (chloroform—methanol), was isolated from the reaction mixture. The crystals obtained (1.85 g) were heated in boiling water for 2 h. The reaction mixture yielded 0.18 g of crystals with mp 164-165°C (3,3′-diindolylmethane (1)).

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