ALKALOIDS OF *Arundo donax*. XVIII. NITROGENOUS BASES IN FLOWERS OF CULTIVARS

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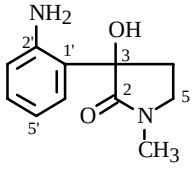
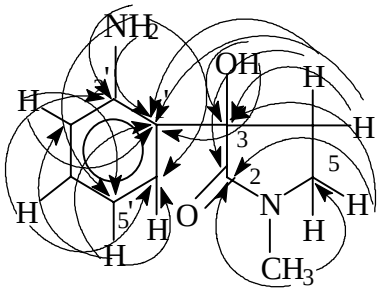
We are studying alkaloids in flowers of *Arundo donax* cultivated in the Tashkent Botanical Garden. Total alkaloids were obtained by usual $CHCl_3$ extraction of dried and ground plant material moistened with aqueous ammonia (5%). Five decantations were performed. The combined $CHCl_3$ extracts were condensed and treated with H_2SO_4 solution (5%). Alkaloids were extracted by $CHCl_3$ from the acidic solutions after they were made basic with conc. aqueous ammonia. The extracts were dried and condensed to afford a mixture of bases (0.042%). Chromatography (TLC) of the total alkaloids identified three alkaloids with R_f values 0.1, 0.6, and 0.7 (TLC, Al_2O_3, C_6H_6:CH_3OH, 9:1) and 0.75, 0.80, and 0.85 ($CHCl_3$:CH_3OH, 9:1). Treatment of the total alkaloids with acetone separated crystals with mp 134-135°C, which were identified by R_f and mixed-melting point as the known alkaloid donaxine [1, 2].

The mother liquors from donaxine were chromatographed over a column of Al_2O_3. Elution with $CHCl_3$ separated crystals with mp 178-180°C (acetone) that were identified as the alkaloid donaxaridine [1, 3] by mixed-melting point and comparison of spectra.

Signals for C atoms in the ^{13}C NMR spectrum of donaxaridine were previously incorrectly assigned [3]. Therefore, we studied its PMR and ^{13}C NMR spectra. The magnitude of the chemical shifts and the nature of the substitution enabled the signals to be grouped into aromatic and aliphatic parts. Table 1 gives chemical shifts of atoms in the PMR and ^{13}C NMR and their assignments.

The HMBC spectrum of donaxaridine exhibits the following important correlation peaks: CH_3/C-5; H-4a,5a,CH_3/C-2; H-4a,b,NH_2,OH,H-3′,5′/C-1′; H-4a,b,5a,NH_2′,OH/C-3; H-4′,5′,NH_2/C-6′; NH_2-2′/C-4′; H-4′/C-2′; H-5′/C-3′; NH_2,H-3′/C-5′.





Continued elution of the column with $CHCl_3$ gave fractions that afforded a base with mp 148-149°C that was identified as arundinine [4].

Thus, flowers of *Arundo donax* contain three pure bases that are described in the literature: donaxine, donaxaridine, and arundinine. These alkaloids were isolated from the flowers of this plant for the first time.

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TABLE 1. Chemical Shifts of ^{1}H and ^{13}C in Donaxaridine

C atom	δ, ppm	Proton	δ, ppm (J/Hz)
C-2	174.0		
C-3	78.7	OH-3	6.03
C-4	33.4	H-4a, H-4b	2.44, 2.17 ($J_{4.4}$ = 12.6; $J_{4a,5a}$ = 2; $J_{4b,5a}$ = 8.6)
C-5	44.5	H-5a, H-5b	3.29, 3.00 ($J_{5.5}$ = 12.6; $J_{5b,4a}$ = 6; $J_{5b,4b}$ = 8.6)
CH_3-1	29.45	CH_3-1	2.85
C-1′	124.4		
C-2′	125.8	H-3′	6.675 ($J_{3',4'}$ = 7.5)
C-3′	115.35	H-4′	6.46 ($J_{4',5'}$ = 7.5)
C-4′	128.0	H-5′	6.97 ($J_{5',6'}$ = 7.5)
C-5′	116.1	H-6′	6.665
C-6′	147.0	NH_2-2′	5.21

NMR spectra were recorded on a Bruker AM-500 spectrometer at working frequency 500 MHz for ^{1}H and 125.8 MHz for ^{13}C in $CDCl_3$.

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