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ALKALOIDS OF *Peganum harmala*

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The plant *Peganum harmala* L. (harmel peganum) is widespread on the territory of our country, including Central Asia. More than 20 alkaloids have been isolated from *P. harmala* from various growth sites [1], but the alkaloid composition of this plant from the territory of Tadzhikistan has not yet been investigated.

We have studied the alkaloid composition of *P. harmala* growing in the regions of Lyaura, Leninskii region (900 m above sea level) and in the basin of the R. Yagnob, Anzobskii region (1500 m above sea level) of the Tadshik SSR.

From 18 kg of air-dry epigeal part of *P. harmala* chloroform extraction yielded 139.82 g (0.77%) of technical alkaloids. A solution of the total alkaloids was separated on a column of alumina (Brockman activity grade II). Elution was performed in the following solvent systems: benzene-ethyl acetate (2:1); ethyl acetate; and chloroform-ethanol (7:3). The fractions obtained were separated and analyzed by TLC (alumina fixed with gypsum) in the systems mentioned.

The alkaloids were repurified on a column of alumina and were identified by thin-layer chromatography with markers, and also on the basis of the UV, IR, and NMR spectra, which agreed with those given in the literature [1]. The amounts of the various alkaloids are given in Table 1.

As can be seen from Table 1, the largest amount of alkaloids was present in the ripe fruit and the smallest amount in the stems and leaves. The percentages of the individual alkaloids in various organs of *P. harmala* were determined by gas chromatography on a Chron-5 chromatograph. Flame ionization detector, 1.20 m × 3 mm column, 5% of SE-30 on Chromaton N, temperature of the evaporator 310°C, temperature of the detector 280°C.

The areas of the peaks were determined with the aid of an IT-2 electronic integrator. The combined error of chromatograph and integrator in the determination of the areas was 2%. Analysis was performed under the regime of the programming of the thermostat temperature. After injection, the temperature was kept at 120°C for 4 min, and then it was raised in the course of 4 min to 260°C.

TABLE 1. Amounts of Alkaloids in *Peganum harmala* in the Flowering-Fruit-Bearing Phases

Plant organ	Sum of the alkaloids, % on the air-dry weight		Amounts of alkaloids in sample 2, % on the total				
	sample 1	sample 2	harmine	harmaline	deoxy-vascicine	peganine	total identified
Leaves	0,3	0,4					
Stems	0,9	0,6	10	5	70	5	10
Flowers	2,6	1,3	—	—	—	—	100
Fruit							
green	4,6	5,7	75	5	5	5	10
ripe	5,4	6,3	15	10	15	40	20
Roots	2,5	2,3	60	5	5	20	10

Note. Sample 1 was collected in the Lyaura region in June, 1981; sample 2 was collected in the R. Yagnob basin in July, 1982.

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It was shown by special experiments that there was no appreciable amount of substances in the samples that appeared at temperatures above 260°C. The amounts of the individual alkaloids calculated as percentages of the total are given in Table 1.

The main alkaloids of *Peganum harmala* L. growing in the basin of the R. Yagnob were harmine and deoxyvascisinone. The amount of harmine was highest in the green fruit and roots, and deoxyvascisinone was the main alkaloid of the epigeal part. The amount of peganine was higher in the ripe fruit. The alkaloid harmaline was not predominating in any part of the plant.

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ALKALOIDS OF THE SEEDS OF *Dipthychocarpus strictus*

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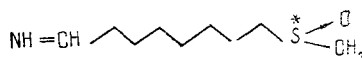
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In an investigation of the seeds of *Dipthychocarpus strictus* (Fisch) Trautv. collected in the environs of the village of Dzhilga (Chimkentskii region, Kaz. SSR) we have found 0.13% of combined alkaloids. From the ethereal fraction of the total, by treatment with acetone we have isolated diptocarpine sulfate [1], and from the mother liquor diptocarpilidine [2]. Treatment of the chloroform fraction with petroleum ether led to diptocarpilidine and treatment with acetone to a very small amount of a crystalline substance with mp 91-92°C which we have called diptocarpinine. The combined mother liquors from the ethereal and chloroform fractions were chromatographed on a column of silica gel (1:30). The alkaloids were eluted successively with benzene, chloroform, and chloroform-methanol (9:1, 8:2, and 1:1). From the benzene eluate we isolated diptocarpilidine and from the other fractions, successively, deoxydiptocarpine [3], diptocarpiline [4], and deoxydiptocarpidine [5].

Diptocarpinine (I) is an optically active white crystalline substance with the composition $C_9H_{19}NOS$, M^+ 189, mp 91-92°C (chloroform). Its IR spectrum showed the absorption bands of $=NH$ (3400 cm^{-1}), $-N=CH$ (1680 cm^{-1}), and $S \rightarrow O$ (1030 cm^{-1}) bonds. The PMR spectrum contained the signals of the protons of the following groups: $CH_3-S \rightarrow O$ (2.53 ppm, 3 H, s); $O \leftarrow S-CH_2$ (2.65 ppm, 2 H, q, $J = 6\text{ Hz}$); $-N=CH-CH_2-$ (2.16 ppm, 2 H); $-N=CH-$ (6.26 ppm, 1 H); and $-CH=NH$ (6.51 ppm, 1 H). The signals of the protons from five methylene groups appeared in the 1.15-2.00 region (10 H).

The presence of the signal of a $=NH$ proton and one olefinic proton in the PMR spectrum and also the absorption of a double bond ($HC=NH$) in the IR spectrum showed that (I) contained a terminal $-HC=NH$ group. In the mass spectrum of diptocarpinine there were intense peaks of ions with m/z 189 [$M(6\%)$] $^+$, 174 (5), 172 (22), 174 (5), 126 (61), 112 (14), 106 (52), 84 (19), 70 (12), 59 (22), 43 (100).

A comparative study of the mass spectra of diptocarpilidine [2] and (I) showed that their molecular weights differed by 16 m/z . The absence of any oxygen-containing group in addition to $S \rightarrow O$, and also the results of IR and PMR spectroscopy, permit the proposal for diptocarpinine of the following most probable structure:



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