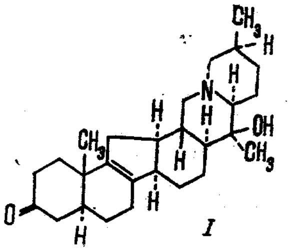
On the basis of the facts given, edpetisine has the most probable structure and configuration of 20β-hydroxycevan-8-en-3-one (I):



I

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POLYBUFFER SEPARATION OF THE COMBINED ALKALOIDS OF Peganum harmala

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UDC 547.944/945

The mother solution from the combined alkaloids of Peganum harmala (the plant was collected in May, 1973 in the building stage), after the isolation of the peganine and deoxypeganine [1, 2] was separated according to basicities on an apparatus for automatic liquid extraction [3] between buffer solutions of various pH values and chloroform.

The chloroform mother solution (4 liters) contained 400 g of combined alkaloids. The solution of the combined alkaloids, and also the washing chloroform, was passed through the column of the apparatus at the rate of 1000-1200 ml/h, and then the buffer solutions were washed with pure chloroform. Each buffer solution was decanted off separately and made alkaline with concentrated ammonia to pH 8-9, and the alkaloids were extracted with chloroform. Then the chloroform was evaporated to dryness. The fractions of alkaloids obtained in this way were analyzed to TLC [nonfixed alumina; chloroform–methanol–benzene (5 : 1 : 4)], and the fractions with identical chromatograms were combined. The alkaloids were isolated according to their solubilities, by recrystallization, by the preparation of salts, by precipitation, etc. [2]:

Thus, the total alkaloids of Peganum harmala contain strongly basic (pH of the extracting buffer 6.5-7.0), moderately basic (pH 3-6.5), and weakly basic (pH 2 and 10% H_2SO_4) alkaloids, additional amounts of which can be obtained by using polybuffer separation.

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 3, pp. 404-405, May-June, 1976. Original article submitted December 4, 1975.

Buffer solution, pH	Weight of the fractions, g	Isolated, g
7.0	37.38	7.8 peganine
6.8	52.80	3.9 deoxypeganine perchlorate
6.6	31.51	14.33 deoxypeganine hydrochloride
6.4	7.73	8.80 deoxypeganine perchlorate 0.86 peganine 1.2 deoxypeganine tetrate
6.2	4.28	0.1 peganine
5.8	3.98	
5.6	2.97	
5.4	3.11	
5.2	2.53	5 harmine
5.0	3.10	
4.0	8.10	
3.0	5.50	0.46 vasicinone
2.0	3.00	4.14 vasicinone 1.3 deoxyvasicone hydrochloride
10% H_2SO_4	20.7	
Washing chloroform	210	Mixture of vasicinone and deoxyvasicinone (on TLC)

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CHARACTERISTIC INFLUENCE OF TRIFLUOROACETIC ACID ON THE CHEMICAL SHIFT ON THE SIGNAL OF A PROTON GEMINAL TO AN OH GROUP

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UDC 577.94:543.42:542.61

To detect and to determine the multiplicity of the masked signal of $\underline{H}-C-OH$ in the NMR spectra of complex organic compounds, recourse is usually had to esterification (acetylation and benzoylation) of the secondary OH group, in consequence of which the signal of the proton under consideration shifts downfield by 0.85-1.2 ppm [1-3]. However, the masked signal of the $\underline{H}-C-OH$ proton can be revealed without having recourse to such reactions. In particular, for this purpose it is possible to use paramagnetic shift reagents or various nonpolar solvents.

In spite of the large number of publications in which the instruments of solvents in NMR spectroscopy for resolving structural and chemical problems have been considered [4-6], trifluoroacetic acid (TFA) has not been the object of a systematic study, particularly in the case of natural compounds.

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prorodnykh Soedinenii, No. 3, pp. 405-406, May-June, 1976. Original article submitted December 23, 1975.