The defatted seeds were extracted with 1% sulfuric acid by steeping. The extraction was carried out for 6 hr 7–8 times. The acid extract was filtered and passed through a column of KU-1 cation-exchanger.

Ammoniacal solutions of alcohols (methanol and ethanol) and also mixtures of organic solvents were used to elute the alkaloids. It was found that a 1.0–1.5% of ammonia in 80–86% ethanol or methanol is a good desorbing solvent. On the basis of the investigation performed, an industrial scheme for the production of perforine from the seeds of H. perforatum has been developed. The yield of combined alkaloids by this scheme is 0.15% and the yield of the perforine 0.07–0.08% of the weight of the dry seeds (from 300 kg of seeds).

We have studied the method of isolating foliosidine from Haplophyllum foliosum [4, 5]. The preparation has proved to be pharmacologically active [6]. At the present time it is at the stage of introduction into medical practice.

The total alkaloids obtained by an adsorption method from the epigeal parts of H. foliosum [5] were separated according to their basicities in a continuous apparatus for polybuffer distribution, but no satisfactory results were obtained. By using the different solubilities of the alkaloids in organic solvents (acetone and ether) we succeeded in isolating the foliosidine (yield 0.015–0.02% of the weight of raw material) and developing an industrial scheme for its production.

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22 May 1969

Institute of the Chemistry of Plant Substances AS UzSSR

UDC 547.944/1

THE STRUCTURE OF VINCANICINE

D. A. Rakhimov, V. M. Malikov, and S. Yu. Yunusov

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 5, pp. 461–462, 1969

By separating the combined ethereal alkaloids of Vinca erecta on a column of Al_2O_3, we have obtained vincanine, vinervidine, and a new base, which we have called vincanicine.

Vinervidine, $C_{19}H_{20}ON_2$, mp 190–191° C (acetone), R_f 0.23 [TLC, SiO_2; benzene–methanol (9 : 1)], $[\alpha]_D^{30}$ ±0° (c 0.6; methanol), has previously been isolated from the roots of this plant [1]. Its UV spectrum $\lambda_{max}^{C_2H_5OH}$ 244, 300, 362 mμ (lg ε 4.08; 3.67; 4.33)] and IR spectrum (743, 1575, 1665, 3220 cm^{-1}) are similar to those of vincanine [2].

The mass spectra of vinervidine and vincanine (table) proved to be identical. Consequently, vinervidine is dl-vincanine.

Substance	m/e (relative intensity, %)			
	M^+	M–15	M–29	C [5]
Vincanine	292 (83)	277 (9)	263 (20)	121 (100)
Vinervidine	292 (62)	277 (9)	263 (17)	121 (100)
Vincanicine	322 (60)	307 (7)	293 (15)	121 (100)

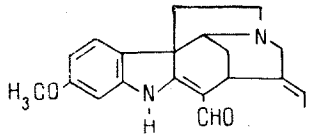
Vincanicine is an amorphous base with R_f 0.3 [TLC on SiO_2; benzene–methanol (9 : 1)], $[\alpha]_D^{24}$ −438.0° (c 0.6; chloroform). Its UV spectrum [$\lambda_{max}^{C_2H_5OH}$ 248, 293, 376 mμ lg ε 3.90; 3.29; 4.04] is similar to that of vincanidine [3].

The IR spectrum has absorption bands at 830–860 cm^{-1} (1,2,4-trisubstituted benzene ring), 1560, 1650 cm^{-1} (carbonyl group conjugated with a double bond), and 3400 cm^{-1} (NH).

On comparing the mass spectrum of vincanicine with the spectrum of vincanine (see table) it can be seen that the values of the peaks of the nonindole ions in them are similar while the indole peaks differ by thirty mass units, which corresponds to the presence of a methoxyl group in the benzene part of the molecule. The NMR spectrum (taken on a JNM-4H-100/100 MHz instrument in $CDCl_3$) of vincanicine differs from that of vincanine by a three-proton singlet (methoxyl group) at δ 3.80 ppm. The mass spectra were taken on a MKh-1303 instrument with an ionizing potential of 32 eV at 105, 95, and 100° C, respectively.

When an ethanolic solution of vincanicine was boiled with methyl iodide, vincanicine methiodide was obtained. It proved to be identical (R_f, mp, IR and UV spectra) with the methiodide of the O-methyl ether of vincanidine [4].

On the basis of what has been presented above, it has been established that vincanicine is the O-methyl ether of vincanidine and has the structural formula



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10 June 1969

Institute of the Chemistry of Plant Substances AS UzSSR

UDC 547.96

STRUCTURE OF THE PEPTIDES OF THE C-TERMINAL FRAGMENT OF PEPSIN

L. G. Muratova, E. D. Levin, and V. M. Stepanov

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 5, pp. 462–463, 1969

It has been shown previously that the specific cleavage of reduced carboxymethylated pepsin with cyanogen bromide forms peptide fragments one of which (B-1) occupies the C-terminal position in the molecule of pepsin and contains 47–49 amino acids [1].

The chymotrypsin hydrolysis of B-1 at pH 10 (4 hr; 37° C; enzyme–substrate ratio 1 : 30) and the fractionation of the hydrolysate on Dowex 1 × 2 enabled a number of peptides to be obtained and the amino acid sequence in some of them to be determined. The peptide "9-2" contains Ile_1, Leu_1, Gly_1, Asp_1, Val_1, and Phe_1. Carboxypeptidase A successively split off from this peptide phenylalanine, valine, and aspartic acid, and by a combination of Edman's method and dansylation the N-terminal sequence of "9-2" was established as Ile-Leu-Gly. Thus, the peptide "9-2" has the structure Ile-Leu-Gly-Asp-Val-Phe, which is identical with the structure of the hexapeptide occupying the N-terminal position in a pepsin fragment containing 27 amino acids and found at the carboxyl end of the molecule of the enzyme [2].

The "12-3" decapeptide was subjected to hydrolysis with oryzin (pH 5.6; 1 hr; 37° C; enzyme–substrate ratio 1 : 10) and paper chromatography yielded two tripeptides having the compositions Ser_2, Gly_1 and Ser_1, Gly_1, Glu_1. Both tripeptides had serine at the N-end and therefore in the "12-3" peptide they are present in the composition of the sequence Ser-Ser-Gly-Glu. Carboxypeptidase A splits out tryptophan and leucine from the "12-3" peptide. In view of the sequence established previously for the pentapeptide present at the N-terminal part of B-1, Asp-Val-Pro-Thr-Ser [1], the structure of the "12-3" peptide may be given in the following way: Asp-Val-Pro-Thr-Ser-Ser-Gly-Glu-Leu-Try.