

ISOLATION OF 6,7-DEHYDRO-8-OXOKOPSININE,
VINCANINE N-OXIDE, AND VENALSTONINE
FROM *Vinca erecta*

M. Sharipov, V. M. Malikov,
and S. Yu. Yunusov

UDC 547.944/1

From *V. erecta*, in addition to alkaloids isolated previously [1, 2], we have obtained three more individual bases. Etheral eluates of the combined alkaloids of the roots of *V. erecta* gave a crystalline base (I) with the composition $C_{21}H_{22}N_2O_3$, mp 231-232°C (methanol), $[\alpha]_D^{20} -93^\circ$ (c 0.3; chloroform). UV spectrum: $\lambda_{\text{max}}^{C_2H_5OH}$ 244, 292 nm (log ϵ 3.93; 3.42). IR spectrum (in KBr), cm^{-1} : 3365, 1720, 1660, 1600, 815, 760. Mass spectrum, m/e: 350 (M^+), 227, 215, 214, 195, 168, and 154. NMR spectrum: (δ scale, ppm, $CDCl_3$): $COOCH_3$ (3.69, singlet); four aromatic protons (6.55-7.2) and two olefinic protons (5.20-6.10, $J = 10$ Hz). The Adams hydrogenation of (I) gave a product with mp 210°C, mol. wt. 352 (mass spectrometrically); IR spectrum, cm^{-1} : 3360, 1730, 1650, 760. In respect of the properties given, base (I) is identical with 6,7-dehydro-8-oxokopsinine [3].

From the chloroform eluates we obtained a hydrobromide with mp 203-205°C (methanol), from which we isolated an amorphous base (II), $C_{19}H_{20}N_2O_2$, mol. wt. 308 (mass spectrometrically), R_f 0.2 [TLC on SiO_2 ; chloroform-methanol (9:1)]. The UV spectrum - $\lambda_{\text{max}}^{C_2H_5OH}$ 244, 300, 365 nm (log ϵ 3.50; 3.84; 4.25) - is similar to that of vincanine. In the IR spectrum there are absorption bands of an NH group (3340 cm^{-1}), of an aldehyde group conjugated with a double bond (1560 and 1652 cm^{-1}), and of a disubstituted benzene ring (770 cm^{-1}). Mass spectrum, m/e (%): 308 (M^+)-6, 292 ($M - 16$)-12, 290 ($M - 18$)-4, 121-13, 107-21, 92-100. The similarity of the IR and UV spectra of (II) and vincanine and the difference of 16 m/e of the peaks of the molecular ions in the mass spectrum, and also the high solubility of (II) in water permitted the assumption that the base isolated is vincanine N-oxide. The reduction of (II) with Zn/H_2SO_4 gave a base with mp 192-194°C having the UV spectrum $\lambda_{\text{max}}^{C_2H_5OH}$ 248, 300 nm (log ϵ 3.84; 3.50), which proved to be identical with the deoxydihydrovincanine obtained by the reduction of vincanine under similar conditions [4].

The third alkaloid was isolated from etheral eluates of the combined alkaloids of the epigeal part of *V. erecta*. Its composition was $C_{21}H_{24}N_2O_2$, mol. wt. 336 (mass spectrometrically), mp 139-140°C (methanol), R_f 0.47 [TLC on SiO_2 ; benzene-ether (3:2)], $[\alpha]_D^{20} -85^\circ$ (c 0.3; chloroform). UV spectrum: $\lambda_{\text{max}}^{C_2H_5OH}$ 243, 293 nm (log ϵ 3.82; 3.27).

IR spectrum (in KBr, cm^{-1}): 3370 (NH), 1720 ($COOCH_3$), 760 (disubstituted benzene ring). The mass spectrum showed peaks of ions with m/e 336 (M^+), 216, 156, 149, 135, and 107. The NMR spectrum of the base (δ scale, ppm, $CDCl_3$) contained the signals of protons from a $COOCH_3$ group (3.67, singlet) of four aromatic protons (6.50-7.10 ppm), and of two olefinic protons (5.30-5.70 ppm).

The above-given constants of the base coincide with the properties of venalstonine L [3].

LITERATURE CITED

1. D. A. Rakhimov, M. R. Sharipov, Kh. I. Aripov, V. M. Malikov, T. T. Shakirov, and S. Yu. Yunusov, *Khim. Prirodn. Soedin.*, 713 (1970).
2. D. A. Rakhimov, M. R. Sharipov, V. M. Malikov, and S. Yu. Yunusov, *Khim. Prirodn. Soedin.*, 677 (1971).
3. H. H. A. Linde, *Helv. Chim. Acta*, 48, 1822 (1965).
4. P. Kh. Yuldashev and S. Yu. Yunusov, *Uzb. Khim. Zh.*, No. 1, 44 (1963).

Order of the Red Banner of Labor Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR. Translated from *Khimiya Prirodnikh Soedinenii*, No. 2, pp. 263-264, March-April, 1974. Original article submitted October 29, 1973.

© 1975 Plenum Publishing Corporation, 227 West 17th Street, New York, N.Y. 10011. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, microfilming, recording or otherwise, without written permission of the publisher. A copy of this article is available from the publisher for \$15.00.