

The method of isolating the glycoside was as follows: from the ground *Convallaria* herb the cardenolides were extracted with 96% ethanol, the extract was evaporated to eliminate the solvent, and the residue was treated with boiling water and the mixture was filtered. The filtrate was purified with a mixture of chloroform and benzene (3:1), after which the glycosides were extracted with chloroform containing 5% of ethanol. Partition chromatography on silica gel (stationary phase water, mobile phase a mixture of benzene and ethyl methyl ketone in various proportions) led to the isolation of the glycoside described, desglucocheirotxin, and a small amount of convallatoxin.

Periplogenin 3-(O- α -L-rhamnopyranoside) has the composition $C_{29}H_{49}O_9$, mp 170–174° C/219–226° C, $[\alpha]_D^{20}$ – 20° (c 0.8; methanol). It crystallized from methanol–ether. With 84% sulfuric acid it gives a coloration changing with time: 0 min–red-orange; 1 min–red-orange with a blue border; 5–15 min–light blue; 20 min–gray-green; 30–60 min–gray.

By paper chromatography of the products of acid hydrolysis according to Mannich and Siewert [4], periplogenin and L-rhamnose were detected. The configuration of the glucosidic linkage was determined by Klyne's method [5].

From its physicochemical properties, its coloration with 84% sulfuric acid, its R_f values in various systems, and a mixed melting point, the substance investigated was identical with a synthetic sample [6] of periplogenin 3-O- α -L-rhamnopyranoside.

This glycoside has previously been isolated from *Antiaris toxicaria* Lesch. [7]. It has also been detected by paper chromatography in *C. majalis* L. and *C. transcaucasica* Utkin.

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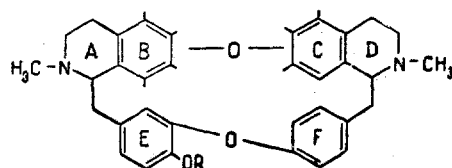
MASS SPECTROMETRY OF THE BISBENZYLISOQUINOLINE ALKALOIDS OF THALICTRUM

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We have studied the mass spectra of the alkaloids hernandezine (I), thalsimine (II), O-methylthalicberine (III), thalmine (IV), and thalfoetidine (V) [1–4]. The spectra were recorded by B. V. Rozynov on a Hitachi RMU-6DS mass spectrometer with the direct introduction of the sample into the ion source (70 eV, 30 μ A, 200° C).

All the bases mentioned belong to the group of bisbenzylisoquinoline alkaloids with two ether bridges [(I)–(IV), R = CH₃, (V), R = H]. The maximum intensity of the peak of the molecular ion in substance (II) is due to the presence of a 1,2-dehydroisoquinoline ring. The high intensity of the peak of the molecular ion in (V) remains unexplained.



The decomposition of the molecular ions forms the ions $(M - 1)^+$, $(M - 15)^+$, $(M - 31)^+$, a , a' , b , c , d , and e . The doubly-charged fragment a is formed by β -cleavage with the loss of rings E and F. Dimethyl ether splits off from

fragment a, giving rise to an ion with an additional ether bridge. The loss of rings C and D leads to fragment b with the appearance of a 13- or 14-membered ring.

Data on the Mass Spectra of Substances (I)-(V)

Fragment	(I)	(II)	(III)	(IV)	(V)
	m/e and relative intensity, %				
M ⁺	652 (62)	636 (100)	622 (52)	608 (40)	638 (100)
(M-1) ⁺	651 (31)	635 (64)	621 (26)	607 (23)	637 (46)
(M-15) ⁺	637 (12)	621 (51)	607 (6)	593 (5)	623 (9)
(M-31) ⁺	621 (5)	605 (17)	591 (2)	577 (5)	607 (6)
a'	426 (17)	—	396 (100)	382 (70)	—
a	213 (100)	205 (13)	198 (24)	191 (100)	213 (67)
(a-15) ⁺	206 (3)	—	381 (18)	183 (10)	206 (18)
(a-46) ⁺	190 (13)	—	175 (5)	168 (31)	190 (69)
b	461 (16)	—	—	—	417 (63)
(b-15) ⁺	446 (2)	—	—	—	402 (57)
c	515 (3)	499 (11)	—	—	515 (2)
d	175 (8)	175 (5)	175 (5)	175 (15)	175 (7)
(d-1) ⁺	174 (27)	174 (7)	174 (10)	174 (40)	174 (5)
e	90 (3)	90 (15)	90 (2)	90 (10)	90 (4)
(e-1) ⁺	89 (6)	89 (6)	89 (2)	89 (5)	89 (2)

As was to be expected, this fragment is absent from the spectrum of (II). The loss of ring E forms a new 8-membered ring, giving fragment c. The singly-charged ion with m/e 175 (fragment d) is formed either from one of the isoquinoline rings or, as in the case of (V), it is doubly charged and arises from the (a - 46)⁺⁺ ion through the loss of OCH₃ and the migration of hydrogen.

The ions m/e 90 and 89 [fragments e and (e - 1)⁺] are characteristic for bisbenzylisoquinoline alkaloids with an unsubstituted benzene ring F.

The formation of these ions is confirmed by the presence in the spectra of substances (I)-(V) of metastable and isotopic peaks (table).

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ALKALOIDS OF *SENECIO JACOBEEA*, *HELIOTROPIMUM ACUTIFLORUM*, AND *H. TRANSOXANUM*

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From 500 g of the epigeal part of *S. jacobea* L., collected on 8 May 1967 in the budding period in the Oshskaya Oblast of KirgSSR (gorge of the R. Akbur) we have isolated by chloroform extraction 2.1 g of ethereal and 0.9 of chloroformic total alkaloids (0.6% of the total dry plant).

Treatment of the total ethereal alkaloids with methanol yielded 1.2 g of crystals with mp 232-235° C (methanol),