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# ALKALOIDS OF *Hypecoum erectum*. THE STRUCTURE OF HYPERECTINE

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The structure of a new spirobenzylisoquinoline alkaloid from *Hypecoum erectum* L. has been established by spectral methods and also by the x-ray structural analysis of its methiodide. It has been shown that the alkaloid, which has been called hyperectine, has a pentacyclic structure in which the tetrahydroisoquinoline fragment has the sofa conformation and the indan fragment the envelope conformation. The natural alkaloid is a mixture of the enantiomeric C-8(S), C-16(R), and C-8(R), C-16(S) forms.

In preceeding communications [1, 2] we have described the isolation from the herb *Hypecoum erectum* L. of two new alkaloids hypecorine and hypecorinine, the determination of their structure, and some of their transformations. In a further study of the alkaloid composition of this plant by fractional separation according to basicities we have isolated another new compound which has been called hyperectine. Hyperectine (I) is a yellow base with the composition $C_{24}H_{21}N_3O_6$, mp 237-238°C (decomp., from a mixture of methanol and chloroform), optically inactive. The alkaloid undergoes hydrolysis in an alkaline medium but is stable to acid hydrolysis and to acetylation with acetic anhydride. Its reaction with diazomethane formed a N-methyl derivative, $C_{25}H_{23}N_3O_6$, mp 258-259°C (decomp.).

The PMR spectra of hyperectine (Fig. 1 and Table 1) contained the signals of four aromatic protons located in pairs in the ortho and para positions with respect to one another, of two aromatic methylenedioxy groups, of a N-methyl group, of a methylene group located between an aromatic nucleus and a saturated quaternary carbon atom, and of three mobile hydrogen atoms.

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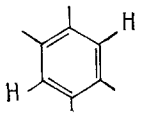
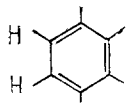
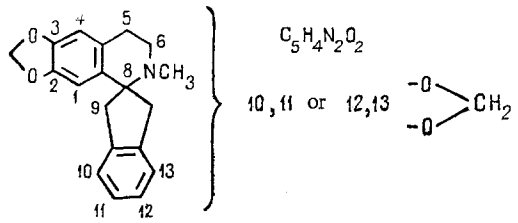
TABLE 1. Details of the PMR Spectra of Hyperectine and Its Methylation Product*

| Structural element | Hyperectine | | | N-Methylhyperectine |
|---|---|---|---|---|
| | $CDCl_3$, 0–TMS, 20°C | $CF_3COOH$, 0–HMDS, 20°C | pyridine-$d_5$, 0–HMDS, 20°C | $CDCl_3$, 0–TMS, 20°C |
|  | 6.45; s, 2H<br>or<br>6,77; s, 2H | 6.23; s, 2H<br>or<br>6,50; s, 2H | 6,87; s, 1H<br>6,41; s, 1H | 6.83; s, 2H<br>or<br>6.54; s, 2H |
|  | 6,77; s, 2H<br>or<br>6,45; s, 2H | 6,50; s, 2H<br>or<br>6.23; s, 2H | 6,63; d, 7 Hz 1H<br>6,68; d, 7 Hz 1H | 6,54; s, 2H<br>or<br>6,83; s, 2H |
| 2Ar—O—$CH_2$—O—Ar | 5,76 5.89; m 4H | 5,43; s, 2H<br>5,48; s, 1H<br>5,38; s, 1H | 5.74; br. s 2H<br>5.68; s, 1H<br>5,52; s, 1H | 5,83–5.96; m, 4H |
| N—$CH_3$ | 2,18; s, 3H | 2,65; d, 5Hz, 3H | 2,18; s, 3H | 2,38; s, 3H |
| N—$CH_3$ | — | — | — | 2,88; s, 3H |
| $C_{14}$—H | 4,64; s, 1H | 4,59; s, 1H | 5,03; s, 1H | 4,76; s, 1H |
| $NH_2$, NH | 3,96; br.s, 2H<br>3,45; br.s, 1H | 8,20; s, 1H<br>7.90; br.s, 1H | 4,86; br.s. 3H | 4,03; br.s, 2H |
| Ar—$CH_2$—C(—)— | 3.45; s, 2H | 3,31; s, 2H | 3.44; s, 2H | 3,52; s, 2H |

*s — singlet; d — doublet; br.s. — broadened signal in the form of a singlet.

The mass spectrum of hyperectine showed intense peaks of ions with m/z 190 and 322. The first of them is characteristic for the mass spectra of many types of tetrahydroisoquinoline alkaloids, and both these ions give strong peaks in the mass spectra of spirobenzylisoquinoline alkaloids with methylenedioxy groups in rings A and D and a methylene group in position 9 [3, 4]. Thus the PMR and mass spectrum of hyperectine permitted the assumption that it had a spirobenzylisoquinoline nucleus. In all known spirobenzylisoquinoline alkaloids, the substituents in ring A are present in positions 2 and 3 [5]. Consequently it is possible to put forward the following partial formula for hyperectine




Two nitrogen atoms and three mobile hydrogen atoms are present in primary and secondary amino groups: In the IR spectrum of hyperectine in chloroform (Fig. 2a) there were bands at 3500, 3395, and 1695 $cm^{-1}$ ($NH_2$) and at 3450 $cm^{-1}$ (NH) [6]. In the IR spectrum of the product of the diazomethane methylation of hyperectine in $CHCl_3$ (Fig. 2b) the band of the NH group at 3450 $cm^{-1}$ had disappeared while the bands of the $NH_2$ group at 3495, 3390, and 1665 $cm^{-1}$

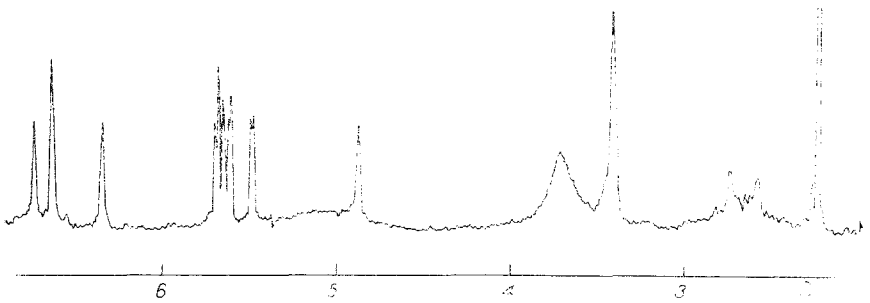



Fig. 1. PMR spectrum of hyperectine in pyridine (100°C).

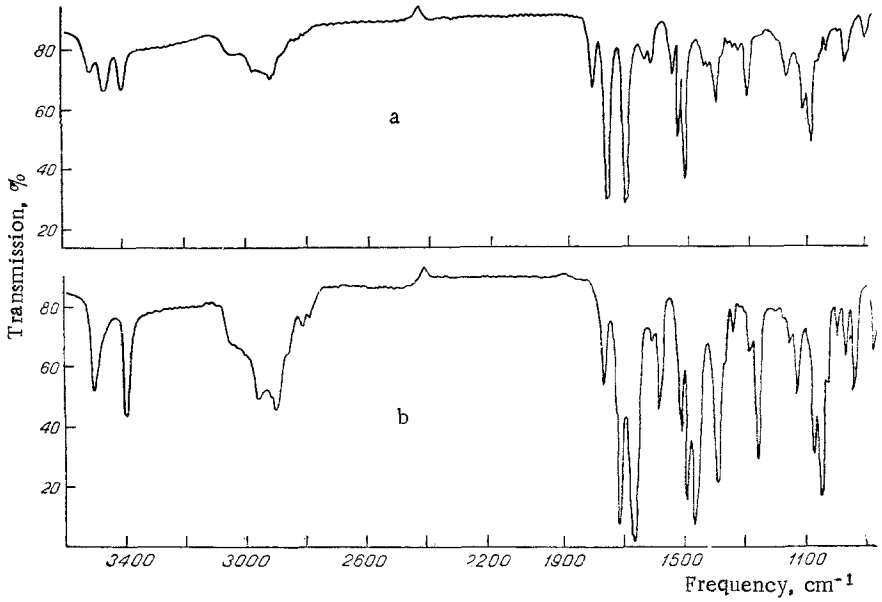
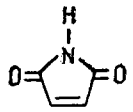


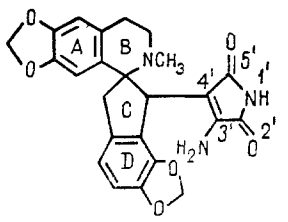

Fig. 2. IR spectra of hyperectine (a) and of N-methylhyperectine (b) in chloroform.

remained. In the PMR spectrum of the methylation product, a three-proton singlet appeared at 2.88 ppm (Table 1). In the IR spectrum of hyperectine in the region of the stretching vibrations of C=O groups there were two bands at 1730 and 1775 $cm^{-1}$ that are characteristic for substituted maleimimides [7, 8]. Thus, the structure of the $C_5H_4N_2O_2$ residue must contain the following fragments: $-NH_2$, CH, and




On the alkaline hydrolysis of hyperectine, the maleimide ring opened and formed a compound with the composition $C_{24}H_{20}N_2O_7 \cdot 2H_2O$, mp 232-233°C (decomp.). The most probable position of the methylenedioxy group in ring D (12–13) follows from biogenetic considerations. The possible biogenetic precursors of hyperectine are coptysine, which we have isolated from the same plant, and asparagine, which is found in many higher plants

Summarizing the results obtained, we arrive at the most probable structural formula (I) for hyperectine [9]



The position of the signal of the N—$CH_3$ group (2,88 ppm) formed on the diazomethanation of hyperectine agreed completely with the proposed structure. The long-wave band in the UV spectrum of hyperectine was shifted by 73 nm in the red direction as compared with the corresponding band of hypecorine [1], which also agreed with the presence in the structure of hyperectine of a maleimide residue with an $NH_2$ group in the 3' position.

For a definitive confirmation of the structure of the alkaloid and to establish its stereochemistry, we made an x-ray structural study of its methiodide, which crystallizes in the form of a solvate with one molecule of ethanol $C_{24}H_{21}N_3O_6 \cdot CH_3I \cdot C_2H_5OH$. The crystals are triclinic, $a$ = 14.918(3), b = 9.571(2), c = 10.193(1) Å, $\alpha$ = 99.38(1), $\beta$ = 95.08(1), $\gamma$ = 101.63(1)°, v = 1395.3(4) Å$^3$, z = 2, space group $P\bar{1}$.

The experimental material was obtained on a Syntex-$P\bar{1}$ diffractometer with $\lambda CuK_\alpha$ radiation, graphite monochromator, $\theta/2\theta$ scanning, $3 \leqslant 2\theta \leqslant 118°$, 2070 reflections with $F^2 \geqslant 2\sigma$. The structure was interpreted by the heavy-atom method and was refined by the method of least squares in the full-matrix anisotropic(1)-isotropic approximation to R = 0.090. The coordinates of the atoms and their temperature factors are given in Table 2.

The crystal was constructed of the organic cations $[C_{25}H_{24}N_3O_6]^+$ the anions $A^-$, and the solvate molecules $C_2H_5OH$* bound by intermolecular and Van der Waals interaction and by hydrogen bonds. The ethanol molecule is H-bound to the $I^-$ anion (I...H—O(7) (I...O 3.33(3) Å) and to the cation (O(7)...H—N(2) (1 − x, 1 − y, 1 − z) (O...N 2,78(4) Å).†

The H atom of the imide group participates in the formation of only one H bond. The I...N(3) distance is 3.73(3) Å. The other N(3)...N and N...O intermolecular distances are greater than 3.4 Å.

The main feature in the structure of the cation (Fig. 3) is the presence of the spiroatom C(8). The stereochemistry of the cation is extremely close to that found in the analogous spirobenzylisoquinoline alkaloid ochotensine, and the lengths of the bonds and the valence angles agree, within the limits of error, with the standard values [11]. In contrast to other spirostructures, the values of the valence angles at the C(8) atom differ little from the tetrahedral values (see Table 3).

The structure of the cation consists of two planar benzo-1,3-dioxolane fragments (the dihedral angle between them being 85°), one of which is condensed with a dihydropyridine ring (1,1-disubstituted 2-methyl-6,7-ethylenedioxy-1,2,3,4-tetrahydroisoquinoline), and the other with a cyclopentene ring forming three tricyclic systems with a common spiroatom C(8). The cyclopentene fragment C(8), C(13)—C(16) has the envelope conformation, and the dihydropyridine ring the sofa conformation and is characterized by the torsional angles given below (degrees):

| Angle | $\gamma$ |
|---|---|
| C(8) C(13) C(14) C(15) | —9 |
| C(13) C(14) C(15) C(16) | —8 |
| C(14) C(15) C(16) C(8) | 20 |
| C(15) C(16) C(8) C(13) | —24 |
| C(16) C(8) C(13) C(14) | 20 |
| C(5) C(6) C(8) N(1) | —29 |
| C(6) C(8) N(1) C(9) | 54 |
| C(8) N(1) C(9) C(10) | —56 |
| N(1) C(9) C(10) C(5) | 33 |
| C(9) C(10) C(5) C(6) | —8 |
| C(10) C(5) C(6) C(8) | —7 |

*The large temperature factors of the carbon atoms of the ethanol molecule (Table 2) show an unorderedness of the solvate molecules.

†Here and below the numbering of the atoms shown in Fig. 3 is used.

TABLE 2. Coordinates of the Atoms ($\times 10^3$, I $\times 10^4$) and Their Temperature Factors B†

| Atom* | X | Y | Z | B, Å² | Atom | X | Y | Z | B, Å² |
|---|---|---|---|---|---|---|---|---|---|
| O (1) | 978 (1) | 849 (2) | 375 (2) | 6,2 (5) | C (11) | 715 (2) | 187 (3) | −113 (3) | 5,0 (6) |
| O (2) | 1080 (1) | 720 (2) | 461 (2) | 6,2 (5) | C (12) | 878 (2) | 329 (3) | −101 (3) | 5,2 (6) |
| O (3) | 462 (1) | 657 (2) | 245 (2) | 5,9 (4) | C (13) | 756 (2) | 524 (3) | −16 (3) | 4,7 (6) |
| O (4) | 525 (1) | 465 (2) | 291 (2) | 5,1 (4) | C (14) | 676 (2) | 581 (2) | 43 (2) | 2,9 (4) |
| O (5) | 744 (1) | 363 (2) | 638 (2) | 4,5 (4) | C (15) | 647 (2) | 499 (2) | 141 (2) | 3,2 (5) |
| O (6) | 656 (1) | 86 (2) | 238 (2) | 4,9 (4) | C (16) | 690 (2) | 374 (2) | 149 (2) | 3,1 (4) |
| N (1) | 798 (1) | 280 (2) | −15 (2) | 3,6 (4) | C (17) | 570 (2) | 535 (2) | 197 (2) | 3,7 (5) |
| N (2) | 700 (1) | 206 (2) | 456 (2) | 3,8 (4) | C (18) | 537 (2) | 646 (3) | 172 (3) | 4,3 (5) |
| N (3) | 753 (1) | 582 (2) | 446 (2) | 4,0 (4) | C (19) | 566 (2) | 731 (3) | 82 (3) | 4,7 (6) |
| C (1) | 1057 (2) | 852 (3) | 469 (3) | 6,6 (8) | C (20) | 642 (2) | 695 (3) | 16 (2) | 3,8 (5) |
| C (2) | 953 (2) | 699 (3) | 314 (3) | 4,6 (6) | C (21) | 448 (2) | 531 (3) | 313 (3) | 4,8 (6) |
| C (3) | 1009 (2) | 627 (3) | 361 (3) | 4,7 (6) | C (22) | 708 (1) | 345 (2) | 288 (2) | 2,5 (4) |
| C (4) | 1000 (2) | 485 (3) | 321 (3) | 5,4 (6) | C (23) | 731 (1) | 437 (2) | 412 (2) | 2,3 (4) |
| C (5) | 925 (2) | 413 (3) | 216 (3) | 4,3 (5) | C (24) | 725 (2) | 341 (2) | 515 (2) | 3,6 (5) |
| C (6) | 866 (2) | 492 (3) | 168 (2) | 3,8 (5) | C (25) | 688 (2) | 199 (2) | 317 (2) | 3,3 (5) |
| C (7) | 879 (2) | 640 (3) | 214 (2) | 4,1 (5) | O (7)‡ | 310 (2) | 39 (2) | 428 (2) | 5,6 (5) |
| C (8) | 782 (2) | 421 (2) | 73 (2) | 3,3 (5) | C (26)‡ | 394 (4) | 139 (6) | 467 (6) | 13 (2) |
| C (9) | 830 (2) | 179 (3) | 72 (3) | 4,3 (5) | C (27)‡ | 438 (4) | 107 (6) | 346 (5) | 12 (2) |
| C (10) | 918 (2) | 252 (3) | 168 (3) | 5,7 (7) | I | 1615 (1) | 1566 (2) | 2323 (2) | 4,1 (1) |

*The numbering of the atoms corresponds to that adopted in Fig. 3.

†Anisotropic temperature factor of the I atom in the form

$$B_{eq} = \frac{1}{3}\sum_i\sum_j B_{ij} a_{ij}^* a_i^* a_j a.$$

‡Atoms of the solvate molecule $C_2H_5OH$.

TABLE 3. Valence Angles ω in the Cation, deg

| Angle | ω | Angle | ω | Angle | ω |
|---|---|---|---|---|---|
| C (1) O (1) C (2) | 101 (2) | C (5) C (6) C (7) | 121 (2) | O (4) C (17) C (15) | 125 (2) |
| C (1) O (2) C (3) | 103 (2) | C (5) O (6) C (8) | 122 (2) | O (4) C (17) C (18) | 112 (2) |
| C (18) O (3) C (21) | 106 (2) | C (7) C (6) C (8) | 117 (2) | C (15) C (17) C (18) | 123 (2) |
| C (17) O (4) C (21) | 106 (2) | C (2) C (7) C (6) | 115 (2) | O (3) C (18) C (17) | 110 (2) |
| C (8) N (1) C (9) | 111 (2) | N (1) C (8) C (6) | 110 (2) | O (3) C (18) C (19) | 125 (2) |
| C (8) N (1) C (11) | 117 (2) | N (1) C (8) C (13) | 110 (2) | C (17) C (18) C (19) | 125 (3) |
| C (8) N (1) C (12) | 107 (2) | N (1) C (8) C (16) | 108 (2) | C (18) C (19) C (20) | 115 (2) |
| C (9) N (1) C (11) | 106 (2) | C (6) C (8) C (13) | 111 (2) | C (14) C (20) C (19) | 120 (2) |
| C (9) N (1) C (12) | 108 (2) | C (6) C (8) C (16) | 113 (2) | O (3) C (21) O (4) | 105 (2) |
| C (11) N (1) C (12) | 108 (2) | C (13) C (8) C (16) | 105 (2) | C (16) C (22) C (23) | 131 (2) |
| C (24) N (2) C (25) | 109 (2) | N (1) C (9) C (10) | 113 (2) | C (16) C (22) C (25) | 122 (2) |
| O (1) C (1) O (2) | 113 (3) | C (5) C (10) C (9) | 113 (2) | C (23) C (22) C (25) | 106 (2) |
| O (1) C (2) C (3) | 111 (2) | C (8) C (13) C (14) | 106 (2) | N (3) C (23) C (22) | 132 (2) |
| O (1) C (2) C (7) | 124 (2) | C (13) C (14) C (15) | 108 (2) | N (3) C (23) C (24) | 122 (2) |
| C (3) C (2) C (7) | 125 (3) | C (13) C (14) C (20) | 128 (2) | C (22) C (23) C (24) | 106 (2) |
| O (2) C (3) C (2) | 111 (2) | C (15) C (14) C (20) | 124 (2) | O (5) C (24) N (2) | 129 (2) |
| O (2) C (3) C (4) | 126 (3) | C (14) C (15) C (16) | 115 (2) | O (5) C (24) C (23) | 121 (2) |
| C (2) C (3) C (4) | 122 (3) | C (14) C (15) C (17) | 113 (2) | N (2) C (24) C (23) | 110 (2) |
| C (3) C (4) C (5) | 117 (3) | C (16) C (15) C (17) | 130 (2) | O (6) C (25) N (2) | 122 (2) |
| C (4) C (5) C (6) | 119 (2) | C (8) C (16) C (15) | 100 (2) | O (6) C (25) C (22) | 128 (2) |
| C (4) C (5) C (10) | 116 (2) | C (8) C (6) C (22) | 116 (2) | N (2) C (25) C (22) | 109 (2) |
| C (6) C (5) C (10) | 125 (2) | C (15) C (16) C (22) | 114 (2) | | |

The maleimide ring attached to the C(16) atom forms angles of 51 an 115°, respectively, with the tricyclic fragments.

The cation contains two asymmetric atoms — C(8) and C(16) — in view of which the existence of four isomers is possible. In the crystal the two enantiomeric forms C(8)(S), C(16)(R), and C(8)(R), C(16)(S) are present, i.e., hyperectine is an example of the fairly rare case among alkaloids of the existence of racemic molecules in nature.

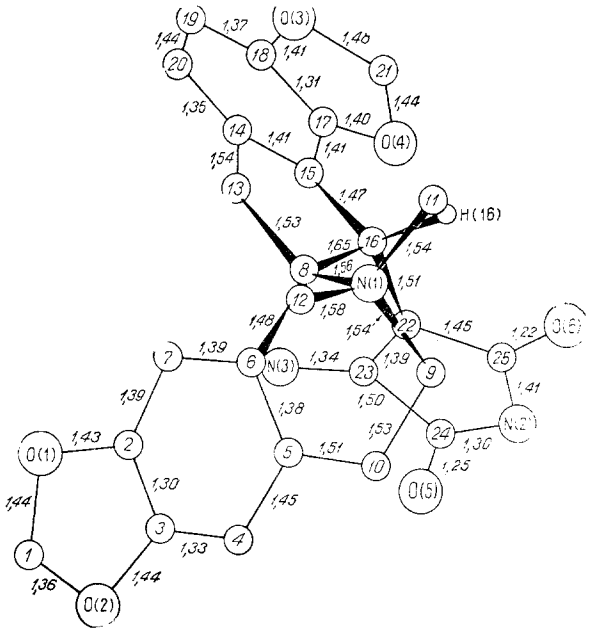



Fig. 3. Structure of the cation in hyperectine methiodide.

## EXPERIMENTAL

The PMR spectra were recorded on a HA-L00D spectrometer, the IR spectra on a UR-20 spectrophotometer, the mass spectra on a CH-8 spectrometer, and the UV spectra on a EPS-3T spectrophotometer. The analyses of all the compounds corresponded to the calculated figures.

Isolation of Hyperectine. The combined alkaloids were isolated from the herb *Hypecoum erectum* by the dichloroethane method and were subjected to separation as described previously [1]. After the separation of protopine, hypecorine, and hypecorinine, the aqueous mother solution was made alkaline to pH 8-9 and was extracted with chloroform. The chloroform extract was evaporated, the residue was dissolved in 5% sulfuric acid, and the alkaloids were subjected to the repeated fractional separation according to their basicities, with extraction by ether. At pH 5.5-6.0, a fraction was obtained from which a yellow crystalline substance was isolated. After recrystallization from a mixture of chloroform and methanol, the substance had the composition $C_{24}H_{21}N_3O_6$, mp 237-238°C. UV spectrum: $\lambda_{max}$ in ethanol (230 (in fraction), 292, 363 nm; log ε 4.29, 3.81, 3.35. Its hydrochloride had the composition $C_{24}H_{21}N_3O_6 \cdot HCl$, mp 264-265°C (decomp., from methanol).

Hyperectine was isolated both with the use of ammonium hydroxide to alkalinize the sulfuric acid solution of the combined alkaloids and when the whole process was carried out by extracting the combined alkaloids from the plant and separating them without the use of ammonia (using sodium carbonate as the alkalinizing agent). This shows that hyperectine is present in the plant and is not formed during the process of isolation.

Methylation of Hyperectine with Diazomethane. An ethereal solution of diazomethane was added to a solution of 0.5 g of hyperectine in 50 ml of methanol and the solution was left to stand for a day, after which the solvent was distilled off; the residue consisted of the methylated product contaminated with a small amount of initial hyperectine. It was purified on a column of alumina. Elution with a mixture of benzene and chloroform (35:15) gave a fraction from which, on concentration to small volume, a crystalline substance was obtained with mp 258-259°C (decomp.), composition $C_{25}H_{23}N_3O_6$ (0.38 g).

Hydrolysis of Hyperectine. A mixture of 0.8 g of hyperectine and 100 ml of 0.1 N caustic soda was heated in the boiling water bath for 6 h. Then the cooled reaction mixture was washed with chloroform to separate the initial alkaloid (the residue after the solvent had been distilled off amounted to 0.1 g). The aqueous alkaline solution was neutralized with 100 ml of 0.1 N hydrochloric acid, and the reaction product was extracted with chloroform. The residue after the elimination of the solvent was recrystallized from a mixture of equal

volumes of chloroform and methanol. This gave 0.5 g of the corresponding acid with the composition $C_{24}H_{20}N_2O_7 \cdot 2H_2O$, mp 232-233°C (decomp.).

Preparation of Hyperectine Methiodide. A solution of 0.1 g of heperectine in 10 ml of methanol was treated with 2 ml of methyl iodide, and the reaction mixture was heated at the boil for 8 h. On cooling, yellow crystalline hyperectine methiodide deposited from the solution (0.07 g). After recrystallization of the substance from methanol it had mp 240-243°C (decomp.). For the x-ray structural studies, crystals of the methiodide were obtained from ethanol.

## CONCLUSIONS

The structure of a new spirobenzylisoquinoline alkaloid from *Hypecoum erectum* L. has been established by spectral methods and also by the x-ray structural analysis of its methiodide. It has been shown that the alkaloid, which has been called hyperectine, has a pentacyclic system in which the tetrahydroisoquinoline fragment has the sofa conformation and the dihydroindane fragment the envelope conformation. The natural alkaloid consists of a mixture of the enantiomeric C(8)(S), C(16)(R) and C(8)(R), C(16)(S) forms.

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