STUDIES ON PLANTS

XI. ALKALOIDS OF ASPIDOSPERMA SPEGAZZINII[1,2]

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ABSTRACT

Extraction of the trunk bark of *Aspidosperma spegazzinii* Molf. ex Meyer yielded ajmaline and two quaternary alkaloids. One of the latter, characterized as the tetraphenylboride salt, was found to be identical with the N_b-metho derivative of akuammidine. Methylation of the phenolic group of the second quaternary alkaloid (named spegatrine) followed by thermal degradation led to the corresponding nor-base, which was identified as lochnerine; therefore, the structure and absolute stereochemistry of spegatrine are given by formula I. A confirmation was provided by N_b-methylation of sarpagine to spegatrine.

Five species of the genus *Aspidosperma* (Apocynaceae) may currently be recognized in the Argentinian flora, namely, *A. quebracho-blanco*, *A. chakensis*, *A. australe*, *A. polyneuron*, and the more recently described *A. spegazzinii* Molf. ex Meyer (1). Unlike the other species, no alkaloids have yet been isolated from *A. spegazzinii*, a tree 10–15 m high, indigenous to Argentina and Paraguay, and referred to by the vernacular names "quina" or "paratudo". The plant material examined in the present work (the trunk bark of old trees) was collected in Río Iguazú, Parque Nacional Iguazú (Misiones, Argentina).

The alcoholic extract of the bark was separated into tertiary bases and quaternary chlorides. The tertiary bases consisted of a mixture of several alkaloids, from which ajmaline was isolated and identified as the ethanol solvate (about 0.3% yield based on dried bark).

The new alkaloid spegatrine (I), the major component (0.25% yield based on dried bark) of the quaternary alkaloidal fraction, was characterized as the crystalline chloride and perchlorate salts. Analytical data give the empirical formula $C_{20}H_{25}N_2O_2$ for the cation and indicate the presence of one N-methyl group, three active hydrogens, and no methoxyl groups. The nuclear magnetic resonance spectrum of the chloride of I, with a doublet centered at δ 1.80 p.p.m. (3H) and an unresolved multiplet at δ 5.95 p.p.m. (1H), suggests the existence of one ethylidene grouping. The quaternary nature of I and the number of active hydrogens require the presence of two OH groups and one NH group.

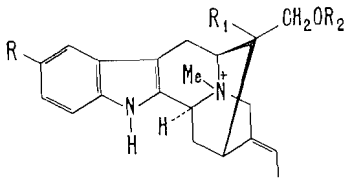
One of the oxygen atoms of I belongs to a phenolic function, as established by the following data. The chloride of I gives positive color reactions with aqueous ferric chloride and diazotized sulfanilic acid, and its ultraviolet spectrum suffers a bathochromic shift on addition of base; in addition, it may be methylated to compound II, which analyzes for one methoxyl group and shows identical ultraviolet absorption in neutral and basic media.

Acetylation of I gives compound III, which may easily be reconverted into the starting material. The acetylated product gave inconsistent results when subjected to repeated acetyl group analysis; however, the presence of infrared bands at 3 390 (NH) and 1 754 cm^{-1} (broad, ester carbonyls) coupled with the absence of amide absorption indicates that it is the O,O-diacetyl derivative of I rather than the O,N-diacetyl or O,O,N-triacetyl

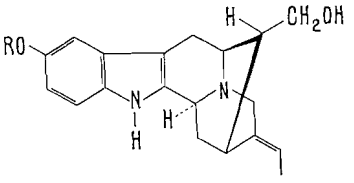
[1]*Part of the doctoral thesis (1965) of M. E. S. is included in this paper.*
[2]*For part X in this series, see R. A. Corral and O. O. Orazi, Tetrahedron, in press.*

derivatives. Furthermore, when going from compound I to III, the nuclear magnetic resonance absorption in the range δ 2.10–2.70 p.p.m. shows the appearance of two sharp peaks (δ 2.25 and 2.50 p.p.m.) and an area increase corresponding to approximately six protons.

Thermal decomposition of the chloride of II gives, besides methyl chloride, its genuine nor-base as proved by a methylation experiment. Analytical and mass spectrometric data on the nor-base establish the molecular formula $C_{20}H_{24}N_2O_2$ and the presence of two active hydrogens and one methoxyl group; the Herzig–Meyer analysis also indicated one N-methyl group, but, as observed in several other instances (2), this is a fictitious result.



	R	R_1	R_2
I	OH	H	H
II	OCH_3	H	H
III	AcO	H	Ac
VI	H	CO_2Me	H



IV R = H
V R = CH_3

The ultraviolet absorption of the nor-base is typical of an indole chromophore, and its mass spectrum shows intense M-1 and M-31 peaks; these peaks suggest a tetrahydro-β-carboline structure and the presence of a primary alcoholic function, respectively (3). Moreover, all the strong peaks (Fig. 1) are displaced by 14 mass units from those of sarpagine (IV) (4).

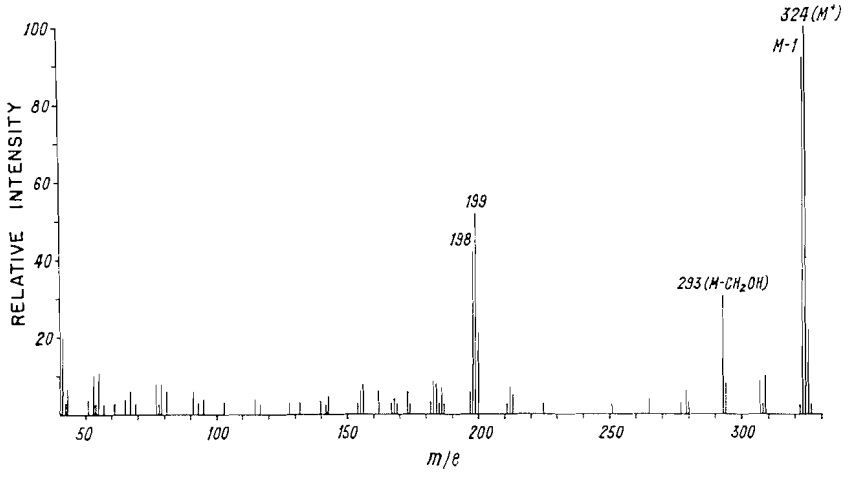


FIG. 1. The mass spectrum of the nor-base of II, inserted in the ion source; only peaks of intensity higher than 2% are plotted.

The nor-base was identical in all respects with the alkaloid lochnerine (10-O-methyl-sarpagine), the structure and absolute stereochemistry of which are shown in V (5); therefore, formula I is a complete description for the spegatrine ion. This result was confirmed by N_b-methylation of sarpagine (IV), the quaternary chloride that was obtained being identical with spegatrine chloride.

The foregoing structural elucidation also demonstrates that the ion II, characterized as the perchlorate salt, must be identical with the quaternary alkaloid lochneram, which was obtained as the iodide and tetraphenylboride salts (6).

The other quaternary alkaloid isolated from *A. spegazzinii*, characterized as the crystalline tetraphenylboride salt and then converted into the amorphous chloride salt, was available in much lesser amounts (0.01% yield based on dried bark). However, structure VI was brought into consideration by its co-occurrence with I and by the following data: ultraviolet absorption almost coincident with that of macusine-A (7); the tetraphenylboride salt shows infrared bands at 3 532, 3 357, and 1 728 cm^{-1} ascribable to alcoholic OH (ultraviolet measurements exclude the phenol group), indole NH, and CO_2Me (one methoxyl by analysis) groups, respectively; the presence of an ethylidene group according to the nuclear magnetic resonance spectrum (multiplets for one methyl group and one hydrogen at δ 1.78 and 5.9 p.p.m., respectively); the optical rotation of the chloride salt ($[\alpha]_D$ +12° (in water)) in comparison with those of macusine-A ($[\alpha]_D$ −58° (in water)) and the nonnatural akuammidine methoiodide ($[\alpha]_D$ +16° (in methanol)) (7, 8).

Methylation of akuammidine under the conditions used for the nor-base of II and sarpagine (IV) led to a quaternary tetraphenylboride salt identical (mixture melting points and superimposable infrared curves) with the sample obtained from the plant; the corresponding amorphous chlorides showed concordant chromatographic mobility, ultraviolet absorption, and optical rotation. The structure and absolute stereochemistry shown in VI are firmly established for the N_b-methylakuammidine cation (9) prepared by methylation of the natural tertiary base (8).

The complete formula VI has recently been assigned to the crystalline alkaloid macusine-C chloride, isolated from *Strychnos toxifera* bark (5); however, the sign and magnitude of the optical rotation ($[\alpha]_D$ −61° (in water)) indicate that it is not identical with the akuammidine methochloride here obtained.

The quaternary alkaloids found in *A. spegazzinii* belong to the sarpagine structural type; two tertiary bases of the same type, polyneuridine and normacusine-B, have recently been isolated from another *Aspidosperma* species (2*b*). On the other hand, the occurrence in this genus of alkaloids with the ajmaline skeleton is unprecedented; besides the biogenetic significance, this may be interesting from the chemical taxonomy viewpoint because of the peculiar anatomy of the *A. spegazzinii* bark (1*c*).

EXPERIMENTAL

Melting points were determined in sealed capillaries and are not corrected; all of the evaporations were carried out under reduced pressure. The ultraviolet spectra in neutral, acidic, or basic medium were recorded with 50% alcohol as the solvent; unless otherwise noted, the infrared spectra were taken in Nujol mulls. The nuclear magnetic resonance spectra were measured in trifluoroacetic acid at 60 Mc/s, the δ values (parts per million) being expressed downfield from internal $SiMe_4$ ($\delta = 0$). The detection of spots in paper chromatograms and electrophoreses was made by fluorescence under ultraviolet light and by iodoplatinate reagent. The microanalyses were performed by Dr. A. Bernhardt (Mülheim, Germany) on samples dried, unless otherwise stated, to a constant weight (100° and a high vacuum) immediately before analysis.

Extraction of Alkaloids

The dried trunk bark (1 kg) of *Aspidosperma spegazzinii* Molf. ex Meyer was ground and extracted during 4 h with boiling 96% alcohol (4 l), with mechanical stirring; the liquid was separated by filtration and the process repeated three times. The combined alcoholic extracts, after concentration to a volume of 300 ml, were diluted at 0° with acetic acid (100 ml) in water (600 ml). The mixture was left in the refrigerator and then filtered; after addition of chloroform (250 ml), the aqueous layer was adjusted (ice bath) to pH 11 with aqueous sodium hydroxide.

The chloroform was decanted, and repeated extractions were made (15 × 100 ml) until the tertiary bases were completely separated. The total chloroform extracts, washed with water and dried over magnesium sulfate, gave, upon evaporation to dryness, the fraction of crude *tertiary alkaloids* (23 g).

The aqueous layer was acidified with 6 *N* hydrochloric acid to pH 2 and the quaternary alkaloids were precipitated by adding an excess of concentrated Mayer's reagent (10). The precipitate (22 g) was suspended in methanol (400 ml) and a current of hydrogen sulfide passed through until the Mayer complex had completely decomposed. The filtered methanolic solution was concentrated and passed successively through Dowex-3 (base form, 100 g) and Dowex-2 chloride (80 g); the eluate was adjusted to pH 6.5 by addition of 0.1 *N* hydrochloric acid, and subsequent removal of the solvent gave the fraction of crude *quaternary chlorides* (13 g).

Isolation of Ajmaline

A chloroform solution of the crude tertiary bases (1 g) was filtered through a column of neutral alumina (activity V, 10 g). Evaporation of the eluate left a solid (0.9 g), which was dissolved in hot absolute ethanol (2.5 ml); to obtain a very crystalline material the solution was allowed to stand undisturbed for several days and gradually cooled down to −10°. Thus was separated 125 mg of ajmaline ethanol solvate, m.p. 194–196° with apparent melting at 125° (gas evolution). A sample that was dried at 130° and 10^{-3} Torr melted at 194–196° without modification at lower temperatures.

The earlier procedure followed for the ajmaline isolation was based on adsorption chromatography. The crude tertiary bases (2 g) were fractionated over neutral alumina (activity II, 200 g); elution was carried out with benzene, benzene–chloroform (9:1 to 1:4), chloroform, and chloroform – absolute ethanol (24:1 to 1:4), and 200 samples (20 ml each) were collected. Eluates 124–167 from chloroform – absolute ethanol (15:1 to 5:1) contained essentially a single alkaloid, as indicated by paper chromatography; upon crystallization from absolute ethanol, 200 mg of ajmaline ethanol solvate, m.p. 191–193° (gas evolution at 125°), was obtained.

An analytical sample was prepared by repeated recrystallizations from the same solvent and dried at 76° and 10^{-3} Torr; $[\alpha]_D^{25}$ +130° (*c*, 1.11 in chloroform); λ_{max} 248 (log ϵ 3.96) and 291 mμ (log ϵ 3.50), λ_{min} 226 (log ϵ 3.58) and 271 mμ (log ϵ 3.20).

Anal. Calcd. for $C_{20}H_{26}N_2O_2 \cdot C_2H_5OH$: C, 70.93; H, 8.66; N, 7.52; O, 12.89; one OC_2H_5, 12.09; one $(N)CH_3$, 4.05; three active H, 0.81. Found: C, 71.23; H, 8.60; N, 7.67; O, 12.86; OC_2H_5, 12.02; $(N)CH_3$, 3.83; active H, 0.81; mol. wt. (by mass spectrum) 326 (calcd. for $C_{20}H_{26}N_2O_2$, 326.42).

An authentic specimen of ajmaline was recrystallized several times from absolute ethanol; it melted, showing the same behavior as the above samples. The identity was ascertained by chromatographic mobility, mixture melting point, and coincidence of the infrared absorptions.

Isolation of Quaternary Alkaloids

The first successful isolation of the alkaloids described below was attained by subjecting the crude quaternary chlorides to preparative paper chromatography; water-saturated methyl ethyl ketone plus 5% methanol was used as the developing solvent, and ultraviolet light and iodoplatinate reagent were used for detection of the bands. Fractionation attempts on powdered cellulose columns gave poorer results.

The work-up of subsequent batches was as follows. The mixture of crude chlorides (5 g) was chromatographed through a column of neutral alumina (activity III, 500 g); the elution was made with chloroform–methanol mixtures, methanol, 90% ethanol, and finally 80% ethanol, and 180 samples (20 ml each) were collected.

The weight distribution and paper chromatography (with the above-mentioned solvent system) showed the presence of several alkaloids; fractions 19–48 (800 mg) and the last 10 fractions (containing only a few milligrams) gave weak spots of mixtures of alkaloids. The most important fractions were fractions 62–101 (from chloroform–methanol (6:1 to 4:1), 250 mg) and fractions 102–170 (chloroform–methanol (4:1 to 90% ethanol), 1.9 g); each group of fractions gave principally a single alkaloid spot, the R_f values of which were 0.45 and 0.32, respectively.

N_b-Methylakuammidine (VI) Tetraphenylboride

(*a*) The combined fractions 62–101 were rechromatographed over neutral alumina (activity III, 25 g) with chloroform–methanol mixtures (19:1 to 1:1). The alkaloidal material of R_f 0.45 (130 mg) eluted with chloroform–methanol (7:1) was dissolved in water and mixed with an aqueous solution of sodium tetraphenylboride.

The precipitated salt (165 mg) was purified on an adsorption column of neutral alumina (activity II, 8 g); the fractions eluted with chloroform–acetone (19:1) were pure when examined by paper chromatography (150 mg). It was further purified by crystallization from alcohol to a constant melting point of 203–205° (decomp.); ν_{max} 3 532, 3 357, and 1 728 cm^{-1}.

Anal. Calcd. for $C_{22}H_{27}N_2O_3 \cdot (C_6H_5)_4B$: C, 80.45; H, 6.90; N, 4.08; one OCH_3, 4.52. Found: C, 80.63; H, 6.75; N, 3.99; OCH_3, 3.90.

This tetraphenylboride was identical with that obtained in experiment *b*, as shown by mixture melting point and complete coincidence of their infrared spectra (KBr).

The pure salt was converted into the corresponding chloride by means of Dowex-2 chloride with methanol as solvent. The resulting solid showed $[\alpha]_D^{20}$ +12° (*c*, 2.05 in water); in neutral or 0.01 *N* NaOH λ_{max} 219 (log ϵ 4.70), 271 (log ϵ 3.87), 277 (inflection) (log ϵ 3.86), and 288 mμ (log ϵ 3.75), λ_{min} 241 (log ϵ 3.55) and 285 mμ (log ϵ 3.72); ν_{max} 1 732 cm^{-1} (ester carbonyl).

(*b*) *From akuammidine.*—A solution of this base (25 mg) in a mixture of methanol (1.5 ml) and methyl iodide (1 ml) was heated in a sealed tube during 10 h at 100° in the dark (cf. ref. 8). After evaporation to dryness, the residue was dissolved in methanol and passed through Dowex-2 chloride. The crude chloride, after being washed with chloroform (2 × 1 ml) to remove unreacted akuammidine (2 mg, identified by paper chromatography), was purified by alumina chromatography as in experiment *a* and then transformed into the tetraphenylboride salt (39 mg); the latter was recrystallized from alcohol to a constant melting point of 203–205° (decomp.).

The amorphous chloride, prepared from the purified tetraphenylboride salt by ionic exchange, exhibited the same chromatographic mobility (R_f 0.45) as the sample obtained in experiment *a* when run on the same paper (water-saturated methyl ethyl ketone plus 5% methanol); $[\alpha]_D^{19}$ +13° (*c*, 1.01 in water); λ_{max} 219 (log ϵ 4.64), 270 (log ϵ 3.88), 276 (inflection) (log ϵ 3.86), and 288 mμ (log ϵ 3.74), λ_{min} 245 (log ϵ 3.68) and 285 mμ (log ϵ 3.70).

Spegatrine (I) Salts

The material of fractions 102–170, obtained from the alumina chromatogram of the crude quaternary alkaloids, was crystallized twice from absolute ethanol to give 1.1 g of spegatrine chloride, m.p. 283° (decomp.), increased to 294° (decomp.) by repeated recrystallizations. Its homogeneity was verified by paper chromatography with several solvent systems and by paper electrophoreses with buffers of pH 3 and pH 8.

It showed $[\alpha]_D^{17}$ +38° (*c*, 1.00 in methanol); in neutral or 0.3 *N* HCl λ_{max} 272 (log ϵ 3.87) and 296 mμ (inflection) (log ϵ 3.63), λ_{min} 245 mμ (log ϵ 3.43); in 0.01 *N* NaOH λ_{max} 270 (log ϵ 3.81), 309 (log ϵ 3.43), and 320 mμ (inflection) (log ϵ 3.34), λ_{min} 253 (log ϵ 3.66) and 293 mμ (log ϵ 3.34).

Anal. Calcd. for $C_{20}H_{25}N_2O_2Cl$: C, 66.56; H, 6.98; N, 7.76; O, 8.87; Cl, 9.83; one OCH_3, 8.60; one $(N)CH_3$, 4.17; three active H, 0.84. Found: C, 65.78; H, 7.08; N, 7.70; O, 8.52; Cl^-, 10.35; OCH_3, 0.61; $(N)CH_3$, 5.13; active H, 0.78.

Hot aqueous solutions of spegatrine chloride and magnesium perchlorate were mixed, thereby precipitating spegatrine perchlorate, constant m.p. 271° (decomp.) after several recrystallizations from butanol.

Anal. Calcd. for $C_{20}H_{25}N_2O_2 \cdot ClO_4$: C, 56.53; H, 5.93; N, 6.59; O, 22.60; Cl, 8.35. Found: C, 56.66; H, 6.02; N, 6.55; O, 22.55; Cl, 8.25.

10-O-Methylspegatrine (II) Perchlorate

A solution of spegatrine (I) chloride (100 mg) in methanol (2 ml) was mixed with anhydrous potassium carbonate (80 mg), and to the stirred solution was added dimethyl sulfate (125 mg) in portions.

After 12 h, the methanolic phase was decanted and evaporated to dryness. The residue was dissolved in water and a solution of ammonium reineckate (in water – acetic acid (2:1)) added; the precipitated reineckate salt was then converted into the corresponding chloride by ionic exchange. Purification by alumina chromatography (neutral, activity IV, elution with chloroform–methanol mixtures) provided the chromatographically pure quaternary chloride (100 mg), which was transformed into the perchlorate by adding an excess of magnesium perchlorate in water.

There was obtained 105 mg that, after repeated recrystallizations from butanol, melted at 147–150° (decomp.); the homogeneity was assessed by paper chromatograms and electrophoreses as indicated for the chloride of I. The perchlorate of II had $[\alpha]_D^{18}$ +45° (*c*, 1.03 in methanol); in neutral, acidic, or basic medium λ_{max} 272 (log ϵ 3.86), 291 (inflection) (log ϵ 3.63), and 303 mμ (inflection) (log ϵ 3.51), λ_{min} 245 mμ (log ϵ 3.44).

Anal. Calcd. for $C_{21}H_{27}N_2O_2 \cdot ClO_4$: C, 57.47; H, 6.20; N, 6.38; O, 21.87; Cl, 8.08; one OCH_3, 7.07; one $(N)CH_3$, 3.43. Found: C, 57.60; H, 6.33; N, 6.38; O, 21.32; Cl, 7.58; OCH_3, 6.72; $(N)CH_3$, 2.96.

10,17-O,O-Diacetylspegatrine (III) Perchlorate

A mixture of spegatrine (I) chloride (100 mg) and acetic anhydride (4 ml) was magnetically stirred at 90° for 10 h; the resulting solution was evaporated, and the solid residue was dissolved in water and mixed with a hot aqueous solution of magnesium perchlorate. The crude quaternary perchlorate (130 mg, m.p. 152–156°) was purified from alcohol to a constant melting point of 155–159°. It was homogeneous according to paper chromatograms and electrophoreses; $[\alpha]_D^{15}$ +34° (*c*, 1.89 in methanol); λ_{max} 223 (log ϵ 4.38), 268 (log ϵ 3.91), and 291 mμ (inflection) (log ϵ 3.73), λ_{min} 258 mμ (log ϵ 3.88); ν_{max} 3 390 and 1 754 (broad) cm^{-1}.

Anal. Calcd. for $C_{24}H_{29}N_2O_4 \cdot ClO_4$: C, 56.64; H, 5.74; N, 5.50; O, 25.15; Cl, 6.97. Found: C, 56.89; H, 5.78; N, 5.46; O, 25.14; Cl, 7.06.

A solution of the perchlorate of III (10 mg) in 2 *N* hydrochloric acid (1 ml) was maintained for 1 h at 90°; evaporation to dryness yielded a material which, when dissolved in methanol, was passed through Dowex-2 chloride. The eluted material was identified as spegatrine chloride by its chromatographic mobility and infrared absorption.

Degradation of 10-O-Methylspegatrine (II) Chloride

This salt, obtained above as an intermediate in the preparation of the corresponding perchlorate, was subjected to thermal degradation according to a literature technique (11).

It was heated (10 batches; in total, 330 mg) in a vacuum sublimation apparatus introduced into a bath at 250° and 10^{-4} Torr; the temperature was then increased to 280° in 1 h and the bath was maintained at this temperature for another hour.

The evolved gaseous product was condensed in an air–liquid trap and identified as practically pure methyl chloride by gas chromatographic and infrared absorption comparisons with an authentic specimen.

The combined sublimed material was extracted with several portions of chloroform; the extract was washed with water and dried, and the solvent was removed, leaving a residue (275 mg) which was purified either by column or thick-layer chromatography with thin-layer chromatograms (alumina Fluka D5F, chloroform–cyclohexane (1:1) plus 1% absolute ethanol) for analytical purposes.

In the former procedure the material (100 mg) was passed over neutral alumina (activity II, 10 g), elution being carried out with chloroform-cyclohexane (1:1) followed by the same system enriched with absolute ethanol. The main fraction (from eluant containing 0.5% ethanol, R_f 0.60) was homogeneous, and upon crystallization from methanol – ethyl ether gave 35 mg, m.p. 192–194°, increased to 204–205° by further recrystallizations. Similar results were obtained by the thick-layer procedure with chloroform–cyclohexane (1:1) plus 1% absolute ethanol as the solvent system.

The nor-base had $[\alpha]_D^{20}$ +70° (*c*, 0.62 in alcohol); λ_{max} 227 (log ϵ 4.50) and 278 mμ (log ϵ 3.94), λ_{min} 215 (log ϵ 4.47) and 250 mμ (log ϵ 3.48).

Anal. Calcd. for $C_{20}H_{24}N_2O_2$: C, 74.04; H, 7.46; N, 8.64; O, 9.86; two active H, 0.62; mol. wt. 324.41. Found: C, 73.53; H, 7.47; N, 8.94; O, 10.42; active H, 0.64; mol. wt. (by mass spectrum) 324.

A sample was dried *in vacuo* at 56°, but not quantitatively.

Anal. Calcd. for $C_{20}H_{24}N_2O_2 \cdot H_2O$: C, 70.15; H, 7.65; N, 8.18; O, 14.02; one OCH_3, 9.06. Found: C, 70.20; H, 7.42; N, 8.11; O, 14.07; OCH_3, 8.88.

The nor-base was identical with an authentic sample of the alkaloid lochnerine, as shown by mixture melting point and infrared spectra (KBr); furthermore, the values of the ultraviolet absorption and optical rotation (12) are virtually coincident.

The nor-base (10 mg) and methyl iodide (0.3 ml) in methanol (0.15 ml) were heated in a sealed tube at 100° for 10 h in the dark. Evaporation of the solution was followed by passage of the residue in methanol through Dowex-2 chloride; the material recovered from the eluate (11 mg) did not contain starting compound, as shown by a paper chromatogram.

The chloride salt was transformed into the perchlorate by magnesium perchlorate in water. The crude salt (13 mg) was crystallized from butanol, m.p. 148–150°, undepressed when admixed with a sample of the above 10-O-methylspegatrine (II) perchlorate. This identity was confirmed by coincidence of the chromatographic mobilities in several systems and of the infrared curves.

Spegatrine (I) Chloride from Sarpagine (IV)

Sarpagine was methylated as described above for the nor-base. The crude quaternary chloride was purified on neutral alumina (100 times by weight, activity III), chloroform–methanol mixtures (19:1 to 2:1) being used as eluants. The major fraction, coming from chloroform–methanol (4:1), gave a single spot on a paper chromatogram; crystallization from absolute ethanol gave N_b-methosarpagine chloride (68% yield) melting at 293° (decomp.); $[\alpha]_D^{20}$ +37° (*c*, 0.96 in methanol).

Anal. Calcd. for $C_{20}H_{25}N_2O_2Cl$: N, 7.76. Found: N, 7.86.

The quaternary salt thus obtained was identical with spegatrine chloride, as demonstrated by paper chromatographic behavior, mixture melting point, and superimposable ultraviolet and infrared spectra.

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