THE SYNTHESIS OF 2,3,7,8-BISMETHYLENEDIOXY-11-METHOXY--5-METHYLBENZO[*c*]PHENANTHRIDINIUM CHLORIDE*

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Acylation of 3-methoxy-6,7-methylenedioxy-1-methylaminonaphthalene (*VI*) with 6-bromo--2,3-methylenedioxybenzoyl chloride gave N-(3-methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 6-bromo-2,3-methylenedioxybenzoic acid (*IIIa*) the irradiation of which with ultraviolet light in acetonitrile and triethylamine gave N-methyl-2,3-methylenedioxy-1-(3′-methoxy-6′,7′-methylenedioxynaphth-1-yl)benzamide (*Xa*) in low yield in addition to 2,3,7,8-bismethylenedioxy-11-methoxy-5-methyl-6-oxobenzo(*c*)phenanthridine (*Va*). The last compound was converted to dihydro derivative *VIa* from which 2,3,7,8-bismethylenedioxy-11-methoxy-5-methylbenzo[*c*]-phenanthridinium chloride (*Ia*) was prepared. Chloride *Ia* differs from the alkaloid chelirubine (*IIa*) to which the structure *Ia* was assigned originally. Under analogous conditions irradiation of N-(3-methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 6-bromo--2,3-dimethoxybenzoic acid (*IIIb*) leads to debrominated starting amide *XIIIb* on the one hand and to N-methyl-2,3-dimethoxy-1-(3′-methoxy-6′,7′-methylenedioxynaphth-1-yl)benzamide (*Xb*) on the other. The expected phenanthridone derivative *Vb* could not be detected in the reaction mixture.

In our previous paper[1] we described unsuccessful attempts at the synthesis of so far unknown 11-methoxy-substituted benzo[*c*]phenanthridines. In this paper we describe a photosynthetic approach which permitted us to prepare 2,3,7,8-bismethylenedioxy-11-methoxy-5-methylbenzo[*c*]phenanthridinium chloride (*Ia*) and some of its derivatives. Using analogous photosynthetic reactions several benzo[*c*]phenanthridines substituted in a different way were prepared earlier, for example avicine[2], nitidine[2,3], chelilutine[4], sanguilutine[4] and chelirubine[5,6] (*IIa*).

The starting compound for photocyclization, N-(3-methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 6-bromo-2,3-methylenedioxybenzoic acid (*IIIa*), was obtained by acylation of 3-methoxy-6,7-methylenedioxy-1-methylaminonaphthalene[1] (*IV*) with 6-bromo-2,3-methylenedioxybenzoyl chloride[7]. From the solution of amide *IIIa* in a mixture of acetonitrile and triethylamine, irradiated for 12 h with a medium-pressure 125 W mercury discharge lamp, we isolated two crystalline compounds in low yield (about 5%).

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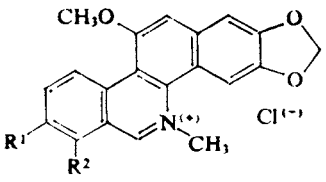

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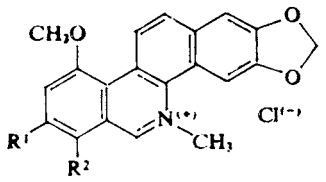

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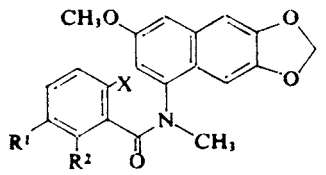

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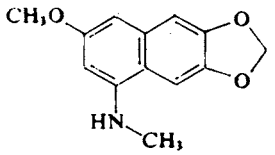
XIII, X = H



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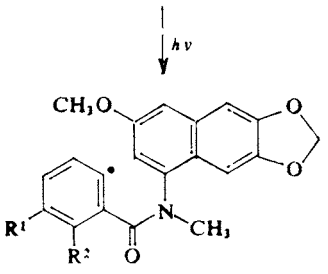
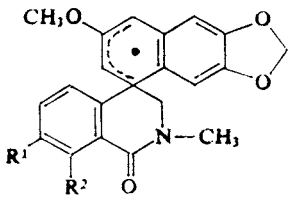
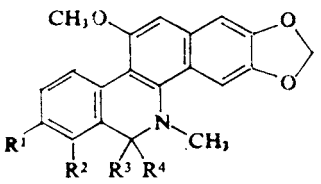


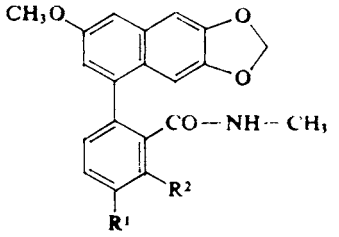
XI



XII



V, $R^3R^4 = O$
VI, $R^3 = R^4 = H$
VII, $R^3 = OH$, $R^4 = H$
VIII, $R^3 = OCH_3$, $R^4 = H$
IX, $R^3 = CN$, $R^4 = H$



X

In formulae: *a*, $R^1R^2 = O{-}CH_2{-}O$; *b*, $R^1 = R^2 = OCH_3$.

The first (m.p. 319–320°C) could be identified on the basis of analytical data and spectral properties (UV, IR and mass spectra) as the required 2,3,7,8-bismethylenedioxy-11-methoxy-5-methyl-6-oxobenzo[*c*]phenanthridine (*Va*). In agreement with this, phenanthridone *Va* is converted on reduction with lithium aluminum hydride to corresponding 5,6-dihydrophenanthridine *VIa* the structure of which we confirmed by the analysis of its ^{1}H NMR spectrum. Under the effect of 2,5-dichloro-3,6-dicyanobenzoquinone in an aqueous sodium hydroxide solution dihydrophenanthridine *VIa* is converted to pseudobase *VIIa* which was converted with methanol to *VIIIa* and with hydrochloric acid to 2,3,7,8-bismethylenedioxy-11-methoxy-5-methylbenzo[*c*]phenanthridinium chloride (*Ia*) which reacted with potassium cyanide to *IXa*. All these transformations represent general reactions of benzo[*c*]-phenanthridine and they further demonstrate the correctness of the structures assigned. Both the orange quaternary chloride *Ia* and its derivatives *VIa* and *IXa* differ considerably from the purple-red chelirubine chloride[8,9] (*IIa*) and its derivatives to which originally the structures *Ia*, *VIa* and *IXa* were assigned[10].

The second substance (m.p. 179–180°C) we assigned the structure of N-methyl-2,3-methylenedioxy-1-(3′-methoxy-6′,7′-methylenedioxynaphth-1-yl)benzamide (*Xa*) on the basis of spectral data (UV, IR, ^{1}H NMR and mass).

The course of the photoreaction leading to the formation of *Va* and *Xa* is very probably analogous to that of the anilides of 2-iodobenzoic acid[11]. The radical *XIa* is its key intermediate which is formed by homolytic cleavage of the C–Br bond by irradiation of the starting amide *IIIa*. This radical is either stabilized by loss of hydrogen, affording the phenanthridine derivative *Va*, or it is converted to the spiro radical *XIIa* which gives rise to the substituted benzamide *Xa* by addition of hydrogen.

The irradiation of N-(3-methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 6-bromo-2,3-dimethoxybenzoic acid (*IIIb*) under analogous conditions had a somewhat different course. We isolated the analogue of amide *Xa* from the reaction mixture, *i.e.* N-methyl-2,3-dimethoxy-1-(3′-methoxy-6′,7′-methylenedioxynaphth-1-yl)benzamide (*Xb*), it is true, but we could not detect the phenanthridine derivative *Vb* in the mixture. Instead, we found debrominated starting amide, N-(3-methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 2,3-dimethoxybenzoic acid (*XIIIb*), the structure of which we confirmed by synthesis from 2,3-dimethoxybenzoic acid and substituted naphthylamine[1] *IV*. It is interesting that the irradiation of amide *IIIa* does not give the debrominated product *XIIIa*. We consider that the mechanism of the photoreaction of amide *IIIb* is the same in principle as of amide *IIIa* and that the observed differences in the composition of the products are caused by solubility factors. The formation of phenanthridone derivative *Va* is in the case of the photoreaction of amide *IIIa* made possible by the insolubility of compound *Va* in the reaction medium. An analogous phenomenon has also been described by Dyke and Sainsbury[12].

EXPERIMENTAL

The melting points were determined on a Boetius microblock and they are not corrected. Analytical samples were dried in a vacuum at 27 Pa over phosphorus pentoxide at room temperature or at 80°C. The purity of the compounds was checked chromatographically on silica gel thin layer (Silufol UV_{254}, Kavalier, Czechoslovakia), in chloroform–benzene–ethanol (1 : 1 : 0·1), using UV light of 366 and 254 nm wavelength for detection. The ultraviolet spectra (λ_{max}, nm (log ε)) were measured on a Unicam SP 8000 spectrophotometer (Great Britain). The infrared spectra (cm^{-1}) were recorded on a Unicam SP 2000 G spectrophotometer (Great Britain). The ^1H NMR spectra (δ, ppm) were taken on a Tesla BS 487 C instrument (Czechoslovakia). The mass spectra (m/z, %) were measured on an AEI MS 902 spectrometer (70 eV, direct inlet).

N-(3-Methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 6-Bromo-2,3-methylenedioxybenzoic Acid (*IIIa*)

A solution of 6-bromo-2,3-methylenedioxybenzoic acid[7] (m.p. 92–93°C) (2·45 g, 10·0 mmol), chloroform (10 ml) and thionyl chloride (3 ml) was refluxed for 1 h and evaporated in a vacuum. The acyl chloride obtained was dissolved without further purification in 20 ml of chloroform and added dropwise to a stirred solution of 3-methoxy-6,7-methylenedioxy-1-methylaminonaphthalene[1] (*IV*) (2·31 g, 10·0 mmol) in chloroform (20 ml) and triethylamine (5·5 ml) at 0°C. After 3 h stirring at 20°C the mixture was washed with 6% hydrochloric acid (100 ml) and 3% sodium hydrogen carbonate (100 ml), dried over anhydrous sodium sulfate and evaporated. The residue was dissolved in benzene (180 ml), filtered, concentrated to 80 ml and allowed to crystallize. Amide *IIIa* (3·8 g, 82%), m.p. 206–209°C. For $C_{21}H_{16}BrNO_6$ (458·3) calculated: 55·01% C, 3·52% H, 17·44% Br, 3·06% N; found: 55·34% C, 3·54% H, 17·89% Br, 2·82% N; UV spectrum (CH_3OH): 233 (4·72), 280 (3·76), 291 (3·80), 327 (3·57), 340 (3·70). IR spectrum ($CHCl_3$): 1 610, 1 640 ($CONCH_3$).

Photocyclization of N-(3-Methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 6-Bromo-2,3-methylenedioxybenzoic Acid (*IIIa*)

A solution of amide *IIIa* (3·67 g, 8·0 mmol) in acetonitrile (400 ml) and triethylamine (4·04 g, 4·04 g, 40·0 mmol) in a quartz apparatus was irradiated with a 125 W medium pressure mercury lamp for 12 h. The separated yellowish needles of 2,3,7,8-bismethylenedioxy-11-methoxy-5-methyl-6-oxobenzo[*c*]phenanthridine (*Va*) were filtered off under suction and crystallized from chloroform–ethanol; 160 mg (5·3%), m.p. 319–320°C. For $C_{21}H_{15}NO_6$ (377·3) calculated: 66·84% C, 4·01% H, 3·71% N; found: 66·63% C, 4·00% H, 3·81% N. UV spectrum (CH_3OH): 239 (4·26), 383 (4·19), 356 (3·96). IR spectrum (nujol): 1 650 ($CONCH_3$). Mass spectrum: 377(100; M^+), 376 (40; M^+-H), 348 (12; M^+-H-CO), 334 (23; $M^+-H-CO-CH_2$), 319 (8; M^+-H $CO-CH_2-CH_3$).

The filtrate after the suction off of compound *Va* was evaporated in a vacuum and the residue was crystallized from methanol; 165·0 mg (5·5%) of needles of N-methyl-2,3-methylenedioxy-1-(3′-methoxy-6′,7′-methylenedioxynaphth-1-yl)benzamide (*Xa*), m.p. 179–180°C. For $C_{21}H_{17}NO_6$ (379·4) calculated: 66·48% C, 4·51% H, 3·69% N; found: 66·17% C, 4·93% H, 3·64% N. UV spectrum (CH_3OH): 235 (4·74), 294 (3·94), 326 (3·77), 339 (3·80). IR spectrum ($CHCl_3$): 1 604, 1 657 ($CONHCH_3$), 3 452 (NH). ^1H NMR spectrum ($C^2H_3SOC^2H_3$): 8·04 (1 H, bq, J = 5·0 Hz; $CONHCH_3$); 7·20 (1 H, s; $C_{(5')}$— or $C_{(8')}$—H); 7·14 (1 H, d, J = 2·5 Hz; $C_{(2')}$— or $C_{(4')}$—H; 7·00 (1 H, d, J = 8·0 Hz; $C_{(5)}$—H); 6·82 (1 H, d, J = 2·5 Hz; $C_{(4')}$— or $C_{(2')}$—H; 6·80 (1 H, s; $C_{(8')}$— or $C_{(5')}$—H); 6·99 (1 H, d, J = 8·0 Hz; $C_{(4)}$—H); 6·11 (2 H, s; OCH_2O); 6·02 (2 H, m; OCH_2O); 3·82 (3 H, s; OCH_3); 2·48 (3 H, d, J = 5·0 Hz; $CONHCH_3$). Mass spectrum: 379 (100; M^+), 349 (16; M^+-NHCH_3).

2,3,7,8-Bismethylenedioxy-11-methoxy-5-methyl-5,6-dihydrobenzo[*c*]phenanthridine (*VIa*)

Lithium aluminum hydride (190 mg, 5·0 mmol) was added to a stirred solution of phenanthridone *Va* (188 mg, 0·5 mmol) in tetrahydrofuran (50 ml) and the mixture was refluxed under stirring for 3 h. Additional lithium aluminum hydride (190 mg, 5·0 mmol) was then added and the mixture refluxed for another 3 h. After cooling it was decomposed with 2M-HCl. The reaction mixture should remain weakly alkaline. The precipitate formed was filtered off, washed with tetrahydrofuran and the filtrate was dried over anhydrous magnesium sulfate. After filtration and evaporation the residue was crystallized from a chloroform–ethanol mixture; the needles of dihydrophenanthridine *VIa* (164 mg, 90%) melted at 203–205°C. For $C_{21}H_{17}NO_5$ (363·4) calculated: 69·40% C, 4·71% H, 3·86% N; found: 69·16% C, 4·82% H, 3·57% N. UV spectrum (CH_3OH): 229 (4·52), 275 (4·52), 329 (4·21), 362 (3·87). ^{1}H NMR spectrum (C^2HCl_3): 7·95 (1 H, d, J = 8·0 Hz; $C_{(10)}$—H); 7·60 (1 H, s; $C_{(4)}$—H); 7·00 (1 H, s; $C_{(1)}$—H); 6·90 (1 H, s; $C_{(12)}$—H); 6·80 (1 H, d, J = 8·0 Hz; $C_{(9)}$—H); 5·97 (4 H, s; 2 OCH_2O); 4·10 (2 H, s; Ar—CH_2—N); 3·91 (3 H, s; OCH_3), 2·53 (3 H, s; NCH_3). Mass spectrum: 363·1107 (M^+, 100; for $C_{21}H_{17}NO_5$ calculated: 363·1120), 362 (67; M^+—H), 348 (32; M^+—CH_3).

2,3,7,8-Bismethylenedioxy-11-methoxy-5-methylbenzo[*c*]phenanthridinium Chloride (*Ia*)

A solution of dihydrophenanthridine *VIa* (54·4 mg, 0·15 mmol) in benzene (2·5 ml) and a solution of 2,5-dichloro-3,6-dicyanobenzoquinone (68·0 mg, 0·30 mmol) in benzene (2·5 ml) were added to a stirred 5% aqueous sodium hydroxide solution (1·5 ml). After 2 h stirring the mixture was poured into water (20 ml) and extracted with ethyl acetate (80 ml). The extract was dried over anhydrous magnesium sulfate and evaporated. The residue was dissolved in 7 ml of a chloroform–ethanol mixture (5 : 2) and a mixture of concentrated hydrochloric acid (1 ml) and ethanol (1 ml) was added to it. The light yellow solution turned orange and orange needles of chloride *Ia* separated from it (42·0 mg, 70%) m.p. 281–285°C. For $C_{21}H_{16}ClNO_5.H_2O$ (415·8) calculated: 60·66% C, 4·36% H, 8·52% Cl, 3·37% N; found: 60·72% C, 4·19% H, 8·72% Cl, 3·77% N. UV spectrum (CH_3OH): 229 (4·54), 275 (4·54), 331 (3·23).

2,3,7,8-Bismethylenedioxy-6,11-dimethoxy-5-methyl-5,6-dihydrobenzo[*c*]phenanthridine (*VIIIa*)

A solution of dihydrophenanthridine *VIa* (18·2 mg, 0·05 mmol) in benzene (0·8 ml) and a solution of dichlorodicyanobenzoquinone (23·0 mg, 0·10 mmol) in benzene (0·8 ml) were added to a 5% aqueous solution of sodium hydroxide (0·5 ml) under stirring, which was continued for 2 h. Then the mixture was poured into water (7 ml) and the product was extracted with ethyl acetate (25 ml). The extract was dried over anhydrous magnesium sulfate, filtered and evaporated. The residue was crystallized from methanol. Yield, 16·0 mg (82%), m.p. 208–210°C. ^{1}H NMR spectrum (C^2HCl_3): 8·05 (1 H, d, J = 9·5 Hz; $C_{(10)}$—H); 7·55 (1 H, s; $C_{(4)}$—H); 6·95 (1 H, s; $C_{(1)}$—H); 6·85 (1 H, s; $C_{(12)}$—H); 6·80 (1 H, d, J = 9·5 Hz; $C_{(9)}$—H); 5·92 (4 H, s; 2 OCH_2O); 5·22 (1 H, s; $C_{(6)}$—H); 3·89 (3 H, s; $C_{(11)}$—OCH_3; 3·31 (3 H, s; $C_{(6)}$—OCH_3); 2·61 (3 H, s; N - CH_3).

2,3,7,8-Bismethylenedioxy-6-cyano-11-methoxy-5-methyl-5,6-dihydrobenzo[*c*]phenanthridine (*IXa*)

Potassium cyanide (13·0 mg, 0·2 mmol) was added to a solution of chloride *Ia* (38·0 mg, 0·09 mmol) in water (20 ml) and the mixture was stirred for 0·5 h. The precipitate formed was extracted with chloroform, the extract was dried over anhydrous magnesium sulfate and evaporated and the residue was crystallized from a mixture of chloroform and ethanol. Yield, 21·1 mg (59%), colour-

less needles, m.p. 264–266°C. Mass spectrum: 388·1048 (M^+, 100; for $C_{22}H_{26}N_2O_5$ calculated: 388·1059); 362 (67; M^+—CN). UV spectrum (CH_3OH): 230 (4·24), 274 (4·22), 333 (3·93).

N-(3-Methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 2,3-Methylenedioxybenzoic Acid (*XIIIa*)

A mixture of 2,3-methylenedioxybenzoic acid (332 mg, 2·0 mmol) in chloroform (2 ml) and thionyl chloride (0·6 ml) was refluxed for 1 h and evaporated in a vacuum. The acyl chloride obtained was dissolved in 6 ml of chloroform and the solution was added dropwise at 0°C to a stirred solution of 3-methoxy-6,7-methylenedioxy-1-methylaminonaphthalene[1] (462 mg, 2·0 mmol) in chloroform (6 ml) and triethylamine (1 ml). After 3 h stirring at 20°C the mixture was shaken with 6% hydrochloric acid (20 ml) and 3% sodium hydrogen carbonate (20 ml). After drying with anhydrous sodium sulfate the extract was evaporated. After crystallization of the residue from benzene (8 ml) and ethanol (10 ml) the amide *XIIIa* was obtained in the form of needles (470 mg, 62%), m.p. 158–160°C. For $C_{21}H_{17}NO_6$ (379·4) calculated: 66·48% C, 4·52% H, 3·69% N; found: 66·67% C, 4·59% H, 3·34% N. UV spectrum (CH_3OH): 234 (4·75), 284 (3·80), 327 (3·59), 342 (3·69). IR spectrum ($CHCl_3$): 1 630 ($CONCH_3$). ^1H NMR spectrum (C^2HCl_3): 7·12 (1 H, s; $C_{(8)}$—H); 6·98 (1 H, s; $C_{(5)}$—H); 6·85 (2 H, bm; $C_{(2)}$—H, $C_{(4)}$—H); 6·50 (3 H, m; $C_{(4')}$—H, $C_{(5')}$—H, $C_{(6')}$—H); 6·00 (2 H, s; $C_{(6)}$—OCH_2O—$C_{(7)}$); 5·82, 5·69 (2 × 1 H, 2 d, J = 1·5 Hz; $C_{(2')}$—OCH_2O—$C_{(3')}$); 3·78 (3 H, s; OCH_3); 3·47 (3 H, s; NCH_3).

N-(3-Methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 6-Bromo-2,3-dimethoxybenzoic Acid (*IIIb*)

A solution of 6-bromo-2,3-dimethoxybenzoic acid[7] (3·4 g, 13·3 mmol) and thionyl chloride (3·6 ml) in chloroform (13 ml) was refluxed for 1 h and evaporated in a vacuum. The acyl chloride obtained was dissolved directly in 26 ml of chloroform and added dropwise at 0°C to a stirred solution of 3-methoxy-6,7-methylenedioxy-1-methylaminonaphthalene[1] (3·01 g, 13·0 mmol) in chloroform (26 ml) and triethylamine (7·2 ml). After 3 h stirring at 20°C mixture was washed by shaking with 6% hydrochloric acid (150 ml) and 3% sodium hydrogen carbonate solution (150 ml). The organic layer was dried over anhydrous sodium sulfate and evaporated under reduced pressure. Yield, 4·5 g (73%), m.p. 183–185°C (benzene). For $C_{22}H_{20}BrNO_6$ (474·3) calculated: 55·71% C, 4·25% H, 16·85% Br, 2·95% N; found: 56·05% C, 4·30% H, 17·21% Br, 3·23% N. UV spectrum (CH_3OH): 235 (4·77), 281 (3·65), 290 (3·64), 328 (3·43), 341 (3·56). IR spectrum ($CHCl_3$); 1 617, 1 645 ($CONCH_3$).

Attempt at Photocyclization of Amide *IIIb*

A solution of amide *IIIb* (1·19 g, 2·5 mmol) in acetonitrile (250 ml) and triethylamine (1·0 g, 10·0 mmol) was irradiated with a 125 W medium pressure mercury lamp in a quartz apparatus. After 6 h no starting amide *IIIb* could be detected in the system by TLC. The solution was evaporated and the residue containing, according to TLC, two predominant components was chromatographed on 50 g of silica gel. Using a mixture of benzene–chloroform (8 : 2) N-(3-methoxy-6,7-methylenedioxynaphth-1-yl)-N-methylamide of 2,3-dimethoxybenzoic acid (*XIIIb*) (132 mg, 13%), m.p. 198–200°C (ethanol) was eluted. The mixture melting point with an authentic synthetic preparation was undepressed. For $C_{22}H_{21}NO_6$ (395·4) calculated: 66·82% C, 5·53% H, 3·54% N; found: 66·72% C, 5·29% H, 3·23% N. UV spectrum (CH_3OH): 234 (4·74), 281 (3·84), 327 (3·68), 341 (3·78). IR spectrum ($CHCl_3$): 1 612, 1 634 ($CONCH_3$). ^1H NMR spectrum (C^2HCl_3): 6·46–7·31 (7 H, m; 7 arom. H); 5·97 (2 H, s; OCH_2O); 3·15–4·02 (12 H, m; 3 OCH_3, NCH_3). Mass spectrum: 395 (24; M^+), 165 (100; M^+—$C_{13}H_{12}NO_3$). The product was identical with a synthetic amide *XIIIb* in all respects.

Using a benzene–chloroform mixture (6 : 4) N-methyl-2,3-dimethoxy-1-(3′-methoxy-6′,7′-methylenedioxynaphth-1-yl)-benzamide (*Xb*) (129 mg, 13%) was eluted, with m.p. 207—208°C (ethanol). For $C_{22}H_{21}NO_6$ (395·4) calculated: 66·82% C, 5·35% H, 3·54% N; found: 66·67% C, 5·48% H, 3·28% N. UV spectrum (CH_3OH): 237 (4·73), 285 (3·94), 327 (3·90), 340 (3·70). IR spectrum ($CHCl_3$); 1 605, 1 654 ($CONHCH_3$), 3 451 (NH). ^{1}H NMR spectrum (C^2HCl_3): 6·70—7·10 (6 H, m; 6 arom. H); 5·90 (2 H, s; OCH_2O); 5·50 (1 H, bq; $NHCH_3$); 3·90 (6 H, s; 2 OCH_3); 3·82 (3 H, s; OCH_3); 2·50 (3 H, d, J = 5·0 Hz; $NHCH_3$). Mass spectrum: 395 (100; M^+), 365 (15; M^+—$NHCH_3$).

N-(3-methoxy-6,7-Methylenedioxynaphth-1-yl)-N-methylamide of 2,3-Dimethoxybenzoic Acid (*XIIIb*)

Analogously as in the case of amide *XIIIa*, amide *XIIIb* was prepared from 2,3-dimethoxybenzoic acid (m.p. 120—122°C) and 3-methoxy-6,7-methylenedioxy-1-methylaminonaphthalene[1] (*IV*) in a 66% yield; m.p. 198—200°C (ethanol). For $C_{22}H_{21}NO_6$ (395·4) calculated: 66·82% C, 5·35% H, 3·54% N; found: 66·40% C, 5·47% H, 3·31% N. The IR and ^{1}H NMR spectra were identical with the spectra of the isolated amide *XIIIb*.

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