5*H*-Isoindolo[1,2-*b*][3]benzazepines VI.* Synthesis of 7,8,13,14-tetrahydro-5*H*-isoindolo[1,2-*b*]-[3]benzazepin-5-one derivatives

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Dihydronarceine imide *N*-oxide reacted under various conditions with acetic anhydride to give derivatives of 1-benzylisoindolin-3-one, 7,8,13,14-tetrahydro- and 13,14-dihydro-5*H*-isoindolo[1,2-*b*][3]benzazepin-5-one. Some addition and substitution reactions of the above-mentioned compounds are also described.

Посредством реакции *N*-окиси дигидронарцеинимида с ацетангидридом в зависимости от условий реакции были получены производные 1-бензилизоиндолин-3-она, 7,8,13,14-тетрагидро- и 13,14-дигидро-5*H*-изоиндоло[1,2-*b*][3]бензазепин-5-она. В работе описываются некоторые реакции присоединения и замещения с участием приведенных соединений.

The secophthalideisoquinoline alkaloid narceine imide (*I*) is a well suited starting material for the synthesis of new heterocyclic compounds [1—4]. Thus *e.g.* derivatives of 7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]benzazepin-5-one substituted in position 7 by dimethylamino, hydroxyl or alkoxyl groups showed a remarkable *in vitro* activity on leukemia P-388 cells [5, 6]; nonetheless, this activity has not been pronounced in the *in vivo* tests. Aiming to obtain more perspective compounds of this series, derivatives of 7,8,13,14-tetrahydro-5*H*-isoindolo[1,2-*b*][3]benzazepin-5-one were synthesized from dihydronarceine imide.

Dihydronarceine imide (*II*) was obtained by hydrogenation of compound *I* on an Adams catalyst in acetic acid [7]. Oxidation of *II* with 3-chloroperbenzoic acid led to the formation of *N*-oxide *III*, which, on treatment with acetic anhydride, afforded various compounds in relation to experimental conditions: a 2 h reaction at −10 °C yielded compounds *IV* and *V*. Compound *IV*, $C_{24}H_{28}N_2O_7$ ($M_r = 456.5$), had in its IR spectrum absorption bands of two carbonyl groups ascribable to a 5-membered lactam ring (at $\tilde{\nu} = 1684\,cm^{-1}$) and

* For Part *V* see *Pharmazie 40*, 521 (1985).

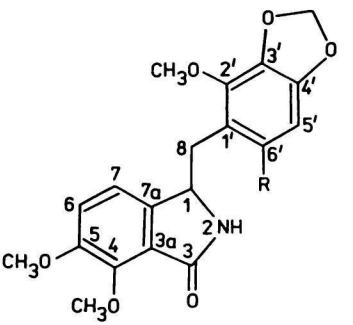
an *N*-methylacetamide grouping (at $\tilde{\nu} = 1636\,\mathrm{cm}^{-1}$). Its mass spectral fragmentation pattern with peaks at *m/z* 456 (M^+), 264 (100 %), 249, 221, 206, and 192 is analogous to that of *N*-methylacetamide *VI* [8]. These data allow to ascribe the structure of 1-{6′-[2″-(*N*-acetyl-*N*-methylamino)ethyl]-2′-methoxy--3′,4′-methylenedioxybenzyl}-4,5-dimethoxyisoindolin-3-one to compound *IV*. This assignment was also backed by the ^{1}H NMR spectrum, which reveals two forms of compound *IV* in solution. The existence of both forms was evidenced by an experiment showing a transfer of saturation [9] between the corresponding signals of *N*-acetyl or *N*-methyl groups of both forms. The most significant changes of chemical shifts were observed between the corresponding signals of NH protons ($\delta = 6.54$ ppm for form *A*, $\delta = 6.08$ ppm for form *B*), *N*-acetyl groups ($\delta = 2.92$ ppm for form *A*, $\delta = 2.81$ ppm for form *B*), and *N*-methyl groups ($\delta = 2.03$ ppm for form *A*, $\delta = 1.83$ ppm for form *B*). Both forms are present in a 2 : 1 volume ratio in chloroform solution. This phenomenon can be explained by the existence of two rotamers due to a hindered rotation of the side chain by the *N*-methylacetamide grouping in which a hydrogen bonding (form *A*) between the acetamide carbonyl and NH groups of the isoindoline moiety of the molecule is taking place. According to spectral evidence which follows, compound *V* is a dihydro derivative of isoindolo[1,2-*b*][3]benzazepine *VII* [4]. The IR spectrum of compound *V* discloses a broad band at $\tilde{\nu} = 3300\,\mathrm{cm}^{-1}$ associated with the vibration of a hydroxyl group linked through its hydrogen bonding with a carbonyl group; the band at $\tilde{\nu} = 1678\,\mathrm{cm}^{-1}$ indicates the presence of a five-membered lactam grouping. The presence of a hydroxyl group was manifested in the mass spectrum by a weak peak at $m/z = 399$ and an intense one at $m/z = 381$ ($M - 18$). Signals of the azepine ring protons in the ^{1}H NMR spectrum were found at δ/ppm: 5.91 (H-7), 3.49 (H-8 eq), 3.18 (H-8 ax), and 2.63 (H-13 ax), 3.64 (H-13 eq) and 4.93 (H-14). The alcohol *V* was methylated with BF_3/MeOH to the methoxy derivative *VIII*. Disappearance of the stabilizing hydrogen bonding between C-7—OH and C-5=O increased the flexibility of the 7-membered heterocycle so that the H-13 and H-14 proton signals in the ^{1}H NMR spectrum of *VIII* appeared as not resolvable multiplets only. Extention of the time of reaction of *III* with acetic anhydride at -10 °C for 24 h gave rise to *N*-methylacetamide *IV* and compound *IX*, $C_{21}H_{19}NO_6$ ($M_r = 381.3$), isomeric with isoindolo[1,2-*b*][3]benzazepine *X*, isolated from the mixture of minor opium alkaloids [10]. Compounds *IX* and *X* differ from each other in the positions of double bonds in the azepine ring between C-7—C-8 with compound *IX* and between C-13—C-14 with compound *X*. Signals of protons H-7 and H-8 of compound *IX* appeared in the ^{1}H NMR spectrum as a pair of doublets at $\delta = 7.08$ and 5.63 ppm, whereas compound *X* had only one proton at H-13 of the double bond resonating at $\delta = 6.34$ ppm. The mass spectrum of compound *IX* had the peak of molecular radical ion at $m/z = 381$

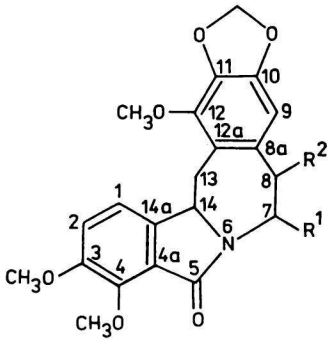
(base peak); further distinguished peaks were observed at *m/z*: 380 (*M* − 1), 366 (*M* − 15), 350, 338, 191, and 190. Compound *X* was found to react very slowly with halogens, therefore 13-chloro-3,4,12-trimethoxy-10,11-methylenedioxy--7,8-dihydro-5*H*-isoindolo[1,2-*b*][3]benzazepin-5-one had to be prepared with phosphorus pentachloride at elevated temperature [11]; on the other hand, compound *IX* reacted with bromine already at about 0 °C. The reaction mixture did not yield the anticipated vicinal dibromo derivative, but the dehydrobrominated product *XI*. Similarly, narceine imide (*I*) reacted with bromine under spontaneous dehydrobromination to furnish α-bromonarceine imide [12]. In contrast to compound *X*, substances *IX* and *XI* easily add alcohols; a short-term boiling of *IX* in methanol, or *XI* in ethanol afforded 7-alkoxyisoindolo[1,2--*b*][3]benzazepines *VIII* and *IX*, respectively.

Treatment of *N*-oxide *III* with acetic anhydride at 40 °C led to *N*-methylacetamide *IV* and dihydronarceonic imide *XIII*, which was also prepared by a Hofmann degradation of dihydronarceine imide methiodide [1].

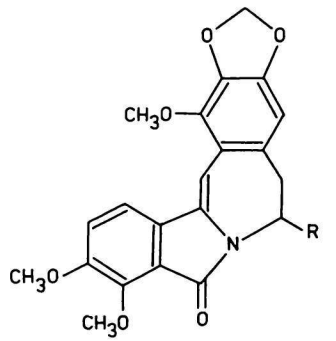
Reaction of narceine imide *N*-oxide with acetic anhydride yielded, in addition to *N*-methylacetamide *VI*, 7-dimethylaminoisoindolo[1,2-*b*][3]benzazepine *XIV* [3, 13]; the *N*-oxide *III* did not give the dihydro derivative of this compound, namely *XV* under the same conditions. This finding could be explained, considering the mechanism of Polonovsky reaction of *N*-oxides with anhydrides of organic acids [14, 15], by elimination of the dimethylamino group from the intermediate *XV* in the presence of acetic anhydride, which stemmed from the immonium salt *XVI* by an intramolecular nucleophilic reaction with the lactam hydrogen; therefore, only compound *IX* could be isolated. This hypothesis is backed by the fact that the alcohol *V* resisted acylation, and dehydrated to compound *IX* even at very mild conditions. Cyclization of the immonium salt *XVI* is a slow reaction, which was evidenced by the reaction of *N*-oxide *III* with acetic anhydride: if this reaction was stopped when all *N*-oxide was consumed at − 10 °C (after about 2 h), no compounds *IX* or *XV* were isolated, but only the alcohol *V*, which could be considered an acetal form of aldehyde *XVII* formed by hydrolysis of the immonium salt *XVI*.

I R = $CH_2CH_2N(CH_3)_2$; 1,8 — double bond
II R = $CH_2CH_2N(CH_3)_2$
III R = $CH_2CH_2NO(CH_3)_2$
IV R = $CH_2CH_2N(CH_3)COCH_3$
VI R = $CH_2CH_2N(CH_3)COCH_3$; 1,8 — double bond
XIII R = $CH{=}CH_2$
XVI R = $CH_2CH{=}N(CH_3)_2$
XVII R = CH_2CHO





V $R^1 = OH$; $R^2 = H$
VIII $R^1 = OCH_3$; $R^2 = H$
IX $R^1 = R^2 = H$; 7,8 — double bond
XI $R^1 = H$; $R^2 = Br$; 7,8 — double bond
XII $R^1 = OCH_2CH_3$; $R^2 = Br$
XV $R^1 = N(CH_3)_2$; $R^2 = H$



VII R = OH
X R = H
XIV $R = N(CH_3)_2$

Experimental

Melting points were determined on a Kofler micro hot-stage, the mass spectra were recorded with a JMS-100D apparatus at 70 eV ionizing energy, the IR spectra were taken with a Perkin—Elmer 983 spectrophotometer, and the ^1H NMR spectra were run with a Bruker, model AM-300 instrument operating at 300 MHz over a range of 4500 Hz corresponding to 32 K points. The digital resolution (after zero filling to 64 K) was 0.14 Hz. The 60 deg pulse was repeated every 5 s during the accumulation. The samples were measured in $CDCl_3$, tetramethylsilane being the internal reference. Silica gel (activity grade IV) was used for column chromatography, the eluates were checked by thin-layer chromatography on Silufol UV 254 sheets in the following solvent systems: chloroform—triethylamine—benzene, φ_r(volume ratio) = 8 : 2 : 2 (S_1); chloroform—methanol, $\varphi_r = 9 : 1$ (S_2).

1-{6'-[2''-(N-Acetyl-N-methylamino)ethyl]-2'-methoxy-3',4'-methylenedioxybenzyl}-4,5-dimethoxyisoindolin-3-one (IV)

Dihydronarceine imide (*II*) (2.0 g; 4.7 mmol) dissolved in chloroform (50 cm^3) was added to a solution of 3-chloroperbenzoic acid (1.008 g; 5.8 mmol) in chloroform

(30 cm^3) at an ambient temperature. The reaction course was monitored by thin-layer chromatography in S_1 (R_f = 0.76 (*II*), 0.15 (*III*)). After 40 min, the mixture was washed with aqueous $NaHCO_3$ (ϱ = 100 g dm^{-3}, 20 cm^3), the chloroform layer was dried with Na_2SO_4 and cooled to −10 °C. Acetic anhydride (1.5 cm^3; 15.9 mmol) was dropwise added to the cooled and stirred reaction mixture. Loss of the *N*-oxide *III* was monitored by thin-layer chromatography in S_1; the mixture was after 2 h washed with aqueous HCl (φ_r = 1 : 20, 10 cm^3) and water (10 cm^3), the organic layer was dried, the solvent was evaporated, the residue was crystallized from benzene, and recrystallized from benzene—heptane (φ_r = 2 : 1). Yield of *IV* 557 mg (25.5 %), m.p. = 189—192 °C. For $C_{24}H_{28}N_2O_7$ (M_r = 456.5) w_i(calc.): 63.15 % C, 6.18 % H, 6.14 % N; w_i(found): 63.21 % C, 6.19 % H, 5.93 % N. IR spectrum (KBr), $\tilde{\nu}$/cm^{-1}: 3436 (N—H), 1684 (C-3=O), 1636 (C=O) in $N(CH_3)COCH_3$. Mass spectrum, *m*/*z* (I_r/%): 456 (12), 441 (2), 382 (1), 381 (1), 265 (12), 264 (100), 249 (2), 222 (9), 221 (87), 206 (5), 192 (62), 191 (28). ^{1}H NMR spectrum ($CDCl_3$), δ/ppm: form *A* — 7.10, 7.06 (ABq, 2H, $J_{6,7}$ = 8.0 Hz, H-7, H-6), 6.54 (br s, 1H, NH), 6.39 (s, 1H, H-5'), 5.92 (s, 2H, OCH_2O), 4.70 (m, 1H, H-1), 4.08, 4.02, 3.89 (3 × s, 3 × OCH_3), 2.92 (s, 3H, N—CH_3), 2.03 (s, 3H, N—$COCH_3$); form *B* — 7.08 (d, 1H, $J_{6,7}$ = 8.0 Hz, H-7), 6.90 (d, 1H, H-6), 6.31 (s, 1H, H-5'), 6.08 (br s, 1H, NH), 5.94 (s, 2H, OCH_2O), 4.70 (m, 1H, H-1), 4.07, 4.06, 3.90 (3 × s, 3 × OCH_3), 2.81 (s, 3H, N—CH_3), 1.83 (s, 3H, N—$COCH_3$).

7-Hydroxy-3,4,12-trimethoxy-10,11-methylenedioxy-7,8,13,14--tetrahydro-5H-isoindolo[1,2-b][3]benzazepin-5-one (V)

The filtrate after separation of *IV* was concentrated and the residue was chromatographed over silica gel. The column was eluted with chloroform—methanol mixture (φ_r = 9 : 1) and the fractions were monitored by thin-layer chromatography in S_2. The fraction showing a spot of R_f = 0.52 was crystallized from acetone—chloroform (φ_r = 1 : 1). Yield of *V* 337 mg (18 %), m.p. = 185—189 °C. For $C_{21}H_{21}NO_7$ (M_r = 399.4) w_i(calc.): 63.15 % C, 5.30 % H, 3.51 %N; w_i(found): 63.09 % C, 5.18 % H, 3.41 % N. IR spectrum ($CHCl_3$), $\tilde{\nu}$/cm^{-1}: 3300 (O—H), 3001, 2940, 2840 (C—H), 1678 (C=O). Mass spectrum, *m*/*z* (I_r/%): 399 (6), 382 (19), 381 (78), 366 (5), 352 (2), 350 (5), 331 (1), 221 (2), 192 (6), 191 (7), 180 (10), 179 (100). ^{1}H NMR spectrum ($CDCl_3$), δ/ppm: 7.20 (d, 1H, $J_{1,2}$ = 8.0 Hz, H-1), 7.17 (d, 1H, H-2), 6.42 (s, 1H, H-9), 5.94 and 5.92 (ABq, 2H, *J* = 1.6 Hz, OCH_2O), 5.91 (d, 1H, $J_{7,8a}$ = 5.7 Hz, H-7), 4.93 (ddd, 1H, $J_{13a,14}$ = 11.8 Hz, $J_{13b,14}$ = 2.3 Hz, $J_{1,14}$ = 0.5 Hz, H-14), 4.10, 3.97, 3.89 (3 × s, 3 × OCH_3), 3.64 (dd, 1H, $J_{13a,13b}$ = 17.3 Hz, H-13b), 3.49 (d, 1H, $J_{8a,8b}$ = 16.2 Hz, H-8b), 3.18 (dd, 1H, H-8a), 2.63 (dd, 1H, H-13a).

3,4,7,12-Tetramethoxy-10,11-methylenedioxy-7,8,13,14-tetrahydro-5H--isoindolo[1,2-b][3]benzazepin-5-one (VIII)

A mixture of BF_3/MeOH (ϱ = 100 g dm^{-3}) was added to the solution of *V* (100 mg; 0.25 mmol) in methanol (10 cm^3). After a 24 h standing at room temperature aqueous NaOH (ϱ = 5 g dm^{-3}, 20 cm^3) was added, methanol was removed under diminished pressure, the residue was extracted with chloroform, the organic layer was dried and

concentrated to crystallization. The final product *VIII* was crystallized from diethyl ether—hexane ($\varphi_r = 2:1$); yield = 89 mg (86 %), m.p. = 198—199 °C. For $C_{22}H_{23}NO_7$ ($M_r = 413.4$) w_i(calc.): 63.92 % C, 5.61 % H, 3.39 % N; w_i(found): 64.16 % C, 5.60 % H, 3.19 % N. IR spectrum ($CHCl_3$), $\tilde{\nu}$/cm^{-1}: 2938, 2901, 2837 (C—H), 1690 (C=O), 1624, 1592, 1480 (aromatic rings). Mass spectrum, m/z (I_r/%): 413 (24), 398 (8), 381 (100), 336 (2), 223 (19), 191 (58). ^{1}H NMR spectrum ($CDCl_3$), δ/ppm: 7.25 (dd, 1H, $J_{1,2} = 8.2$ Hz, $J_{1,14} = 0.9$ Hz, H-1), 7.18 (d, 1H, H-2), 6.47 (s, 1H, H-9), 5.91, 5.90 (ABq, 2H, $J = 1.6$ Hz, OCH_2O), 5.77 (dd, 1H, $J_{7,8a} = 3.5$ Hz, $J_{7,8b} = 5.5$ Hz, H-7), 4.52 (m, 1H, H-13a), 4.07, 3.91, 3.86 (3 × s, 3 × OCH_3), 3.70 (m, 1H, H-13b), 3.24 (s, 3H, C-7—OCH_3), 3.11 (s, 2H, H-8a, H-8b).

3,4,12-Trimethoxy-10,11-methylenedioxy-13,14-dihydro-5H--isoindolo[1,2-b][3]benzazepin-5-one (IX)

Procedure specified for preparation of *IV* was applied with the exception that after addition of acetic anhydride at − 10 °C the mixture was left to stand for 24 h. Compound *IV* was crystallized from benzene to afford 581 mg (27.1 %). The mother liquors were concentrated and chromatographed on a silica gel-packed column with chloroform—methanol ($\varphi_r = 9:1$). The yellow fraction was evaporated and the residue was crystallized from acetone—hexane ($\varphi_r = 1:1$) to give 427 mg of *IX* (23.8 %), m.p. = = 221—222 °C, $R_f = 0.86$ (S_1). For $C_{21}H_{19}NO_6$ ($M_r = 381.4$) w_i(calc.): 66.14 % C, 5.02 % H, 3.67 % N; w_i(found): 66.10 % C, 4.96 % H, 3.62 % N. IR spectrum ($CHCl_3$), $\tilde{\nu}$/cm^{-1}: 3001, 2940, 2900, 2840 (C—H), 1695 (C=O), 1648 (C=C), 1610, 1494, 1475 (aromatic rings). Mass spectrum, m/z (I_r/%): 382 (24), 381 (100), 380 (7), 366 (8), 352 (4), 338 (2), 191 (8), 190 (29).^{1}H NMR spectrum ($CDCl_3$), δ/ppm: 7.22 and 7.16 (ABq, 2H, $J_{1,2} = 8.1$ Hz, H-1, H-2), 7.09 (d, 1H, $J_{7,8} = 10.5$ Hz, H-7), 6.46 (s, 1H, H-9), 5.95 and 5.94 (ABq, 2H, $J = 1.6$ Hz, OCH_2O), 5.63 (d, 1H, H-8), 4.58 (ddd, 1H, $J_{13a,14} = 9.4$ Hz, $J_{13b,14} = 1.0$ Hz, $J_{1,14} = 0.9$ Hz, H-14), 4.10 (dd, 1H, $J_{13a,13b} = 15.8$ Hz, H-13b), 4.10, 4.02, 3.92 (3 × s, 3 × OCH_3), 2.46 (dd, 1H, H-13a).

8-Bromo-3,4,12-trimethoxy-10,11-methylenedioxy-13,14-dihydro--5H-isoindolo[1,2-b][3]benzazepin-5-one (XI)

Bromine (64 mg; 0.394 mmol) in chloroform (10 cm^3) was dropwise added to a cooled and stirred solution of compound *IX* (150 mg; 0.39 mmol) in chloroform (20 cm^3). After 30 min of stirring the solvent was removed and the residue was crystallized from diethyl ether—hexane ($\varphi_r = 2:1$) to give *XI* (140 mg; 0.30 mmol), yield = 78 %, $R_f = 0.90$ (S_1), m.p. = 205 °C (decomp.); for $C_{21}H_{18}BrNO_6$ ($M_r = 460.3$) w_i(calc.): 54.80 % C, 3.94 % H, 3.04 % N; w_i(found): 54.72 % C, 3.89 % H, 3.06 % N. Mass spectrum, m/z (I_r/%): 461 (2), 459 (2), 380 (100), 367 (17), 366 (14), 349 (12), 190 (36), 189 (12). ^{1}H NMR spectrum ($CDCl_3$), δ/ppm: 7.85 (s, 1H, H-7), 7.19 (s, 2H, H-1, H-2), 7.17 (s, 1H, H-9), 5.99 and 5.98 (ABq, $J = 1.6$ Hz, OCH_2O), 4.49 (dd, 1H, $J_{13a,14} = 9.1$ Hz, $J_{13b,14} = 0.9$ Hz, H-14), 4.13 (dd, 1H, $J_{13a,13b} = 15.1$ Hz, H-13b), 4.10, 4.03, 3.91 (3 × s, 3 × OCH_3), 2.43 (dd, 1H, H-13a).

8-Bromo-7-ethoxy-3,4,12-trimethoxy-10,11-methylenedioxy-7,8,13,14-tetrahydro-5H-isoindolo[1,2-b][3]benzazepin-5-one (XII)

Compound *XI* (20 mg; 0.04 mmol) was heated in ethanol (5 cm^3) for 15 min, the solvent was evaporated and the residue was crystallized from ether—hexane ($\varphi_r = 2:1$) to yield *XII* (18 mg; 82 %), m.p. = 184 °C. For $C_{23}H_{24}BrNO_7$ ($M_r = 506.4$) w_i(calc.): 54.56 % C, 4.69 % H, 2.76 % N; w_i(found): 54.49 % C, 4.70 % H, 2.69 % N. ^{1}H NMR spectrum ($CDCl_3$), δ/ppm: 7.26 and 7.18 (ABq, 2H, $J_{1,2} = 8.2$ Hz, H-1, H-2), 6.57 (s, 1H, H-9), 5.98 and 5.96 (ABq, 2H, $J = 1.5$ Hz, OCH_2O), 5.94 (d, 1H, $J_{7,8} = 4.6$ Hz, H-7), 5.24 (d, 1H, H-8), 4.56 (m, 1H, H-14a), 4.11, 4.00, 3.92 (3 × s, 3 × OCH_3), 3.82 (m, 1H, H-13a), 3.50 (m, 2H, OCH_2CH_3), 2.90 (m, 1H, H-13b), 1.09 (t, 3H, $J = 7$ Hz, OCH_2CH_3).

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