5,6-Dihydro-8*H*-isoquinolo[2,3-*a*]phthalazin-5-ones and 8*H*-isoquinolo[2,3-*c*][2,3] benzoxazin-5-ones

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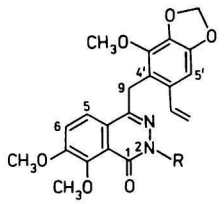
Condensation of narceonic acid (*I*), obtained from narceine (*II*), with derivatives of hydrazine or with hydroxylamine afforded derivatives of 4-benzyl-1,2-dihydrophthalazin-1-one or 4-benzyl-1*H*-[2,3]benzoxazin-1-one; both cyclize in acid medium to give 5,6-dihydro-8*H*-isoquinolo[2,3-*a*]phthalazin-5-ones and 8*H*-isoquinolo[2,3-*c*][2,3]benzoxazin-5-ones, respectively.

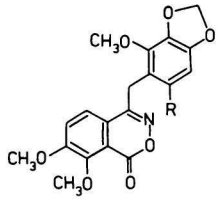
Из нарцеоновой кислоты (*I*), которая была приготовлена из нарцеина (*II*) конденсацией с производными гидразина или с гидроксиламином, были приготовлены производные 4-бензил-1,2-дигидрофталазин-1-она или 4-бензил-1*H*-[2,3]бензоксазин-1-она, которые в кислой среде замыкают цикл с образованием 5,6-дигидро-8*H*-изохиноло[2,3-*a*]фталазин--5-онов или 8*H*-изохиноло[2,3-*c*][2,3]бензоксазин-5-онов.

Narceonic acid — 3-(6′-ethenyl-2′-methoxy-3′,4′-methylenedioxy)benzylidene-6,7-dimethoxyphthalide (*I*) — which can be prepared from narceine (*II*) by methylation with methyl iodide and degradation of the obtained quaternary salt of narceine methyl ester with potassium hydroxide, is a convenient starting material for preparation of 5,6-dihydro-8*H*-isoquinolo[2,3-*a*]phthalazin-5-ones and 8*H*-isoquinolo[2,3-*c*][2,3]benzoxazin-5-ones. A mixture of *Z* and *E* isomers was synthesized according to a modified Freund's procedure [1] analogously as with synthesis of 3-benzylidenephthalide [2, 3]; no separation of the mixture was needed for further synthesis. Condensation of *I* with derivatives of hydrazine or with hydroxylamine yields the respective derivatives of 4-benzyl-1,2-dihydrophthalazin-1-one (*III*, *IV*) or 4-benzyl-1*H*-[2,3]benzoxazin-1-one (*V*).

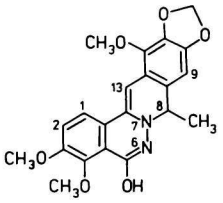
Structure of the synthesized compounds was proved by characteristic signals of protons at C-9 (compounds *III*—*VI*) in the ^{1}H-n.m.r. spectrum (δ scale, p.p.m.) and those of an ABX multiplet of the vinyl group at C-6′ (compounds *III*—*V*) or possibly, by the absence of the proton signal at C-9 in Ar—CH= grouping in the starting material *I* (6.40 p.p.m.). Thus *e.g.* the ^{1}H-n.m.r. spectrum of *IV* revealed



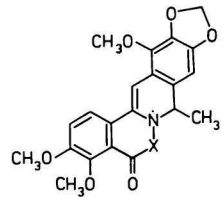
III R = H
IV R = C_6H_5



V R = $CH{=}CH_2$
VI R = $CH_2CH_2N(CH_3)_2$



XII



XIII X = $N{-}C_6H_5$
XIV X = O

signals of C-9 protons at 4.33 and vinyl protons signals at 7.00 (H_A), 5.47 (H_C), 5.15 (H_B), J_{AB} 11 Hz, J_{AC} 17 Hz, J_{BC} 1 Hz. The i.r. absorption bands of the 6-membered lactams $\nu(C{=}O)$ were reported to be in the 1660 cm^{-1} region [4, 5] (compounds *III*, *IV*), those of lactones at about 1730 cm^{-1} (compounds *V*, *VI*).

Phthalic anhydride and hydrazine reacted to give mainly 1,2,3,4-tetrahydrophthalazine-1,4-dione (*VII*) and a small amount of 2-aminophthalimide (*VIII*) [6—9]; heating of 2-phenyl-1,2,3,4-tetrahydrophthalazin-1,4-dione (*IX*) in sodium methoxide led to isomerization of *IX* to 2-anilinophthalimide (*X*) [10]. An analogous preparation of compounds *III*—*VI* gave us a single product and heating of *IV*, which is structurally close to *IX*, in sodium methoxide at a reflux temperature for 10 h was not associated with any isomerization. Compound *VII* can exist in two tautomeric forms, the enol form being favourized [11] and therefore, acetylation in pyridine gave *O*-acetyl derivative [12, 13]. Compound *III*, as it follows from spectral data, exists prevalently in a keto form; the acetyl derivative *XI* revealed a u.v. spectrum identical with that of the starting material *III*, nevertheless its i.r. spectrum lacked the band ascribable to a 6-membered lactam at 1660 cm^{-1}, and new bands at 1752 ($\nu(C{=}O)$) and 1677 cm^{-1} ($\nu(C{=}N)$) appeared; consequently, compound *XI* is the *O*-acetyl derivative of *III*.

The final step of the synthesis of tetracyclic substances from narceine is the intramolecular cyclization; such an acid-catalyzed intramolecular cyclization is

quite frequently applied for addition to double bond, *e.g.* in preparation of pseudocryptopine [14], structure elucidation of mammeisine [15], or synthesis of yohimbine derivatives [16]. Cyclization of *III*—*V* proceeds smoothly in alcoholic hydrochloric acid affording substances *XII*—*XIV* in the u.v. spectra of which a bathochromic shift of absorption bands was observed, the greatest being in the couple of substances *IV*, *XIII* (124 nm). This shift may be caused by the extension of conjugation of multiple bonds due to a C-13—C-13a new double bond in a planar arrangement; this arrangement is backed by appearance of a $\nu(C=C)$ band in the i.r. spectrum at 1640 cm^{-1}. The last absorption bands in the u.v. spectra of compounds *XII*—*XIV* can be ascribed to an $n \rightarrow \pi^*$ transition associated with the presence of a lone electron pair of nitrogen in position 7. The positions of the above-mentioned bands shifted bathochromically with lowering of pH. Such a shift was not observed in spectra of uncyclized compounds *III*—*VI*. The ^{1}H-n.m.r. spectra of *XII*—*XIV* are characteristic of a doublet of a methyl group bound to C-8 (*e.g.* in *XIII* at 1.20), a quadruplet of a C-8 proton (at 5.44), and a singlet of C-13 (at 6.01). All those signals as well as the absence of proton signals of vinyl and C-9 Ar—CH_2— groups provide an evidence of cyclization of *III*—*V*. The i.r. spectrum of *XII* does not contain vibration of a carbonyl group at 1660 cm^{-1}, but a more intense absorption band of an aromatic ring. The mass spectrum displayed a peak at *m/z* $M - 17$ which, together with the i.r. band at 3420 cm^{-1} indicates the presence of an OH group. Compound *XII* in which an enol-keto tautomerism is possible exists, therefore, in an enol form.

Experimental

Melting points were measured on a Kofler micro hot-stage; the respective spectra were recorded with following apparatuses: mass spectra with a JMS-100 D at an ionizing energy 70 eV, u.v. spectra with a Specord UV VIS (Zeiss, Jena), i.r. spectra with a Perkin—Elmer, model 457, and ^{1}H-n.m.r. spectra with a Tesla BS 487 B (δ scale, p.p.m., internal reference tetramethylsilane). Solvent systems for analytical thin-layer chromatography on Silufol UV-254 plates (Kavalier, Votice) and preparative thin-layer chromatography on Kieselgel GF-254 (Merck) were as follows: S_1 (benzene—methanol 9:1), S_2 (chloroform—methanol 9:1), S_3 (ethyl acetate), S_4 (acetone), S_5 (methanol—ammonium hydroxide 99:1), S_6 (chloroform—n-hexane—2-butylamine 5:4:1).

3-(6′-Ethenyl-2′-methoxy-3′,4′-methylenedioxy)benzylidene-6,7--dimethoxyphthalide (I)

A mixture consisting of narceine (3.0 g), KOH (2.8 g), and methyl iodide (7 g) in methanol (30 ml) was refluxed on a steam bath for 3 h. Methanol and methyl iodide were

distilled off, 30% KOH (20 ml) was added to the dry residue and the mixture heated on a steam bath for 30 min. The content of the flask was cooled, diluted to 50 ml and poured into 10% HCl (100 ml): yield 2.15 g (73.1%) of a white *I*, R_f 0.13 (S_1), 0.52 (S_2), 0.80 (S_4); m.p. 217°C (ethanol). For $C_{21}H_{18}O_7$ (382.3) calculated: 65.96% C, 4.74% H; found: 65.88% C, 4.65% H. UV spectrum λ_{max}^{MeOH}, nm (log ε): 222 (4.60), 247 (4.00), 270 (4.32). IR spectrum ($CHCl_3$): 3020, 2940, 2900, 2840, 1760, 1707, 1680, 1600, 1570, 1498, 1478, 1455, 1425, 1395, 1360, 1320, 1280, 1270, 1250, 1135, 1080, 1050, 1000, 938 cm^{-1}. Mass spectrum *m/z:* 382 (M^+, 11%), 367 (4%), 356 (16%), 341 (5%), 338 (5%), 209 (26%), 192 (74%), 178 (6%), 165 (100%), 149 (7%), 133 (12%).

4-(6'-Ethenyl-2'-methoxy-3',4'-methylenedioxy)benzyl-1,2-dihydro--7,8-dimethoxyphthalazin-1-one (III)

Compound *I* (1.0 g) and hydrazine hydrate (4 ml) dissolved in ethanol (30 ml) were refluxed for 4 h. Crystals, which separated after cooling, were recrystallized from ethanol. Yield 0.32 g (31.1%), R_f 0.72 (S_1), 0.61 (S_3); m.p. 276—278°C. For $C_{21}H_{20}N_2O_6$ (396.3) calculated: 63.63% C, 5.08% H, 7.07% N; found: 63.55% C, 5.01% H, 7.05% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 227 (4.73), 283 (4.10). IR spectrum (KBr): 3280, 3160, 3080, 3000; 2940, 2880, 2840, 1650, 1600, 1550, 1492, 1480, 1450, 1410, 1363, 1328, 1288, 1225, 1200, 1168, 1120, 1086, 1064, 1043, 1010, 1008, 992, 945, 920, 860, 820, 785 cm^{-1}. Mass spectrum *m/z:* 396 (M^+, 70%), 381 (100%), 366 (36%), 365 (80%), 351 (12%), 321 (9%), 192 (8%), 179 (27%).

4-(6'-Ethenyl-2'-methoxy-3',4'-methylenedioxy)benzyl-2-phenyl--1,2-dihydro-7,8-dimethoxyphthalazin-1-one (IV)

Narceonic acid (0.50 g) and phenylhydrazine (0.50 g) in methanol (30 ml) were refluxed for 4 h. Crystals (0.36 g, 60.4%), which separated after cooling had R_f 0.40 (S_1), 0.80 (S_2), 0.74 (S_3), 0.87 (S_4); m.p. 185—186°C (ethanol). For $C_{27}H_{24}N_2O_6$ (472.4) calculated: 68.63% C, 5.12% H, 5.93% N; found: 68.59% C, 5.02% H, 5.90% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 231 (4.71), 270 (4.19), 298 (4.10). IR spectrum (KBr): 3000, 2980, 2930, 2840, 1660, 1600, 1580, 1495, 1478, 1450, 1425, 1410, 1370, 1310, 1280, 1147, 1125, 1089, 1057, 1047, 1010, 985, 945, 930, 890, 788, 775 cm^{-1}. Mass spectrum *m/z:* 472 (M^+, 38%), 458 (26%), 457 (100%), 441 (6%), 426 (6%), 381 (10%), 366 (29%). ^{1}H-N.m.r. spectrum ($CDCl_3$): 7.90—7.12 (m, 7H) aromat. H; 6.75 (s, 1H) $H_{5'}$; 5.90 (s, 2H) OCH_2O; 7.00 (dd, 1H) H_A, 5.47 (dd, 1H) H_C, 5.15 (dd, 1H) H_B, J_{AB} 11 Hz, J_{AC} 17 Hz, J_{BC} 1 Hz Ar—CH_A=CH_BH_C; 4.33 (s, 2H) H_9; 3.94 (s, 3H), 3.98 (s, 6H) 3 × OCH_3.

4-(6'-Ethenyl-3',4'-methylenedioxy-2'-methoxy)benzyl-7,8-dimethoxy--1H-[2,3]benzoxazin-1-one (V)

Narceonic acid (0.3 g) and hydroxylamine hydrochloride (0.5 g) were refluxed in ethanol (20 ml) and pyridine (5 ml) for 20 h. The solution was cooled and the separated crystals

were suction-filtered. Yield 0.21 g (67.9%), R_f 0.48 (S_1), 0.92 (S_2), 0.79 (S_3), 0.77 (S_4), m.p. 196—197°C (ethanol). For $C_{21}H_{19}NO_7$ (397.3) calculated: 63.47% C, 4.82% H, 3.52% N; found: 63.38% C, 4.73% H, 3.50% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 226 (4.68), 271 (4.22), 316 (3.76). IR spectrum ($CHCl_3$): 3080, 3000, 2950, 2890, 2840, 1728, 1603, 1590, 1560, 1497, 1477, 1455, 1420, 1395, 1360, 1318, 1283, 1250, 1175, 1128, 1086, 1048, 1038, 1010, 985, 940, 918, 848, 820 cm^{-1}. Mass spectrum *m/z:* 397 (M^+, 27%), 382 (7%), 366 (15%), 353 (24%), 352 (100%), 339 (27%), 338 (53%), 322 (14%), 307 (19%). ^{1}H-N.m.r. spectrum (pyridine-d_5 and DMSO-d_6): 7.90 (d), 7.68 (d), ABq, H_4H_5, $J_{4,5}$ 10 Hz; 7.01 (dd, 1H) H_A, 5.60 (dd, 1H) H_C, 5.24 (dd, 1H) H_B, J_{AB} 18 Hz, J_{AC} 12 Hz, J_{BC} 1.5 Hz, Ar—CH_A=CH_BH_C; 6.93 (s, 1H) $H_{5'}$; 6.00 (s, 2H) OCH_2O; 3.98 (s, 3H), 3.94 (s, 6H) 3 × OCH_3; 4.41 (s, 2H) H_9.

4-[6'-(2"-Dimethylamino)ethyl-2'-methoxy-3',4'-methylenedioxy]benzyl--7,8-dimethoxy-1H-[2,3]benzoxazin-1-one (VI)

A mixture of narceine (0.3 g) and hydroxylamine hydrochloride (0.3 g) in water (15 ml) was heated for 6 h. The title product separated after cooling upon addition of 5% NH_4OH. Yield 0.22 g (76.2%), R_f 0.10 (S_1), 0.16 (S_2), 0.09 (S_4); m.p. 175.5—176.5°C (ethanol). For $C_{23}H_{26}N_2O_7$ (442.5) calculated: 62.54% C, 5.93% H, 6.34% N; found: 62.48% C, 5.88% H, 6.30% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 214 (4.68), 278 (4.04), 326 (3.74). IR spectrum ($CHCl_3$): 3080, 3020, 3010, 2940, 2890, 2860, 1730, 1620, 1590, 1498, 1478, 1458, 1320, 1283, 1130, 1090, 1057, 1038, 1011, 983, 940, 918, 877, 838, 820 cm^{-1}. Mass spectrum *m/z:* 397 (4%), 383 (13%), 366 (3%), 352 (6%), 235 (8%), 234 (10%), 221 (38%), 206 (23%), 205 (10%), 191 (22%), 190 (82%), 188 (17%), 58 (100%). ^{1}H-N.m.r. spectrum ($CDCl_3$): 7.68 (d, 1H), 7.42 (d, 1H), ABq, H_5H_6, $J_{5,6}$ 8 Hz; 6.46 (s, 1H) $H_{5'}$; 5.90 (s, 2H) OCH_2O; 4.22 (s, 2H) H_9; 3.96 (s, 3H), 4.00 (s, 6H) 3 × OCH_3; 2.90—2.40 (m, 4H) Ar—CH_2CH_2N; 2.26 (s, 6H) $N(CH_3)_2$.

1-Acetoxy-4-(6'-ethenyl-3',4'-methylenedioxy-2'-methoxy)benzyl--7,8-dimethoxyphthalazine (XI)

Compound *III* (0.15 g) in pyridine (5 ml) and acetic anhydride (2 ml) was acetylated by heating for 1 h. The mixture was cooled, poured into cold water, the separated substance was filtered off and crystallized from ethyl acetate. Yield 75 mg (45%), R_f 0.20 (S_1), 0.83 (S_2), 0.71 (S_3), 0.81 (S_4); m.p. 128—130°C. For $C_{23}H_{22}N_2O_7$ (438.4) calculated: 63.07% C, 5.06% H, 6.39% N; found: 62.59% C, 4.92% H, 6.33% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 226 (4.68), 282 (4.11), IR spectrum ($CHCl_3$): 3010, 2940, 2900, 2850, 1752, 1677, 1605, 1498, 1480, 1455, 1425, 1285, 1265, 1088, 1070, 1050, 1010, 991, 956, 950, 928, 828 cm^{-1}. Mass spectrum *m/z:* 438 (M^+, 8%), 396 (45%), 395 (19%), 382 (33%), 381 (90%), 366 (12%), 365 (43%), 361 (3%), 351 (10%), 321 (11%), 293 (5%), 191 (15%), 179 (100%), 178 (12%), 133 (17%), 43 (55%).

5-Hydroxy-3,4,12-trimethoxy-8-methyl-10,11-methylenedioxy-8H--isoquinolo[2,3-a]phthalazine (XII)

Methanolic HCl (50 ml) and *III* (0.20 g) were refluxed for 1 h, concentrated to 10 ml, poured into cold water, and pH of this solution was adjusted to 6.5 by addition of 1% NH_4OH. The solution was extracted with chloroform, the extract dried and after evaporation of chloroform the residue was purified by preparative thin-layer chromatography in S_2. Yield 80 mg, R_f 0.42 (S_2), 0.60 (S_5), 0.29 (S_6); m.p. 179—181°C (methanol). For $C_{21}H_{20}N_2O_6$ (396.4) calculated: 63.63% C, 5.08% H, 7.07% N; found: 63.55% C, 4.91% H, 7.03% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 214 (4.66), 255 (4.40), 340 (4.12); $\lambda_{max}^{0.1\ M\text{-}HCl}$, nm (log ε): 213 (4.67), 254 (4.61), 333 (4.16). IR spectrum (KBr): 3420, 3060, 3020, 2990, 2930, 2890, 1640, 1620, 1590, 1570, 1535, 1480, 1470, 1450, 1380, 1360, 1190, 1055, 1035, 1019, 995, 980, 935 cm^{-1}. Mass spectrum *m/z:* 396 (M^+, 18%), 395 (22%), 381 (53%), 379 (45%), 366 (100%), 351 (30%), 193 (21%), 192 (82%), 191 (20%), 179 (27%), 177 (25%). ^{1}H-N.m.r. spectrum ($CDCl_3$): 7.43 (d, 1H), 7.85 (d, 1H), ABq, H_1H_2, $J_{1,2}$ 10 Hz; 6.42 (s, 1H) H_9; 6.08 (s, 1H) H_{13}; 5.95 (s, 2H) OCH_2O; 5.46 (q, 1H) J 7.0 Hz, $CH_3CH=$; 4.66 (s, 1H) OH; 4.02 (s, 3H), 4.04 (s, 3H), 4.17 (s, 3H) $3\times OCH_3$; 1.73 (d, 3H) J 7.0 Hz, $CH_3CH=$.

6-Phenyl-5,6-dihydro-3,4,12-trimethoxy-8-methyl-10,11-methylene-dioxy-8H-isoquinolo[2,3-a]phthalazin-5-one (XIII)

Compound *IV* (0.50g) was refluxed in 5 % methanolic HCl (25 ml) for 30 min; the solution was concentrated to 10 ml and poured into 5% NH_4OH (50 ml). The separated orange coloured precipitate was filtered off an dried. Yield 0.44 g of crude *XIII*. A part of this substance (200 mg) was purified by preparative thin-layer chromatography in S_1; crystallization from methanol gave the pure *XIII* (120 mg), R_f 0.52 (S_1), 0.88 (S_2), 0.80 (S_3), 0.92 (S_4); m.p. 185.5°C. For $C_{27}H_{24}N_2O_6$ (472.4) calculated: 68.63% C, 5.12% H, 5.93% N; found: 68.55% C, 5.01% H, 5.91% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 209 (4.53), 234 (4.51), 268 (4.10), 319 (4.23), 394 (4.00); $\lambda_{max}^{0.1\ M\text{-}HCl}$, nm (log ε): 216 (4.53), 229 (4.40), 256 (4.15), 351 (4.16). IR spectrum (KBr): 3040, 2970, 2930, 2900, 1648, 1616, 1583, 1490, 1475, 1445, 1428, 1402, 1375, 1360, 1330, 1285, 1250, 1095, 1065, 1050, 1025, 987, 948, 938, 848, 790 cm^{-1}. Mass spectrum *m/z:* 472 (M^+, 77%), 457 (49%), 443 (37%), 429 (25%), 413 (9%), 381 (53%), 380 (100%), 366 (33%), 236 (15%), 190 (20%), 93 (44%), 66 (16%). ^{1}H-N.m.r. spectrum ($CDCl_3$): 7.06—7.73 (m, 7H) aromat. H; 6.42 (s, 1H) H_9; 6.01 (s, 1H) H_{13}; 5.85 (s, 2H) OCH_2O; 5.44 (q, 1H) $CH_3CH=$; 3.92 (s, 3H), 3.98 (s, 3H), 4.08 (s, 3H) $3\times OCH_3$; 1.20 (d, 3H), J 6 Hz, $CH_3CH=$.

3,4,12-Trimethoxy-8-methyl-10,11-methylenedioxy-8H-isoquinolo--[2,3-c][2,3]benzoxazin-5-one (XIV)

Compound *V* (0.18 g) in 5% methanolic HCl (70 ml) was refluxed for 1 h; the solution was concentrated to 15 ml and poured into 10% NH_4OH (50 ml). Yield 95 mg (52.2%), R_f

0.45 (S_1), 0.89 (S_2), 0.76 (S_3), 0.87 (S_4); m.p. 196—198°C (ethanol). For $C_{21}H_{19}NO_7$ (397.3) calculated: 63.47% C, 4.83% H, 3.52 N; found: 63.38% C, 4.73% H, 3.49% N. UV spectrum λ_{max}^{MeOH}, nm (log ε): 227 (4.77), 274 (4.29), 319 (3.86), 333 (3.80). IR spectrum (KBr): 3080, 2940, 2890, 2840, 1725, 1648, 1600, 1560, 1495, 1470, 1450, 1418, 1370, 1320, 1280, 1250, 1223, 1214, 1090, 1047, 1032, 1008, 985, 945, 915, 850, 808, 730 cm^{-1}. Mass spectrum *m/z:* 397 (M^+, 30%), 382 (15%), 381 (20%), 366 (20%), 353 (62%), 352 (100%), 338 (50%), 307 (22%), 208 (44%), 207 (40%), 192 (60%), 191 (27%), 178 (18%), 174 (20%), 162 (17%). ^{1}H-N.m.r. spectrum (pyridine-d_5): 7.73 (d, 1H), 7.58 (d, 1H), ABq, H_1H_2, $J_{1,2}$ 9 Hz; 6.44 (s, 1H) H_9; 6.12 (s, 1H) H_{13}; 5.98 (s, 2H) OCH_2O; 5.47 (q, 1H), *J* 8 Hz, $CH_3\underline{CH}=$; 4.00 (s, 6H), 4.12 (s, 3H) $3\times OCH_3$; 1.70 (d, 3H) $\underline{CH_3}CH$, *J* 8 Hz.

All spectra were measured and substances analyzed in the Department of Analytical Chemistry (Head Dr. C. Peciar), Institute of Chemistry, Slovak Academy of Sciences, Bratislava.

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