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REACTIONS OF 6,7-DIMETHOXY-3,4-DIHYDROISOQUINOLINE WITH *o*-QUINONE METHIDES

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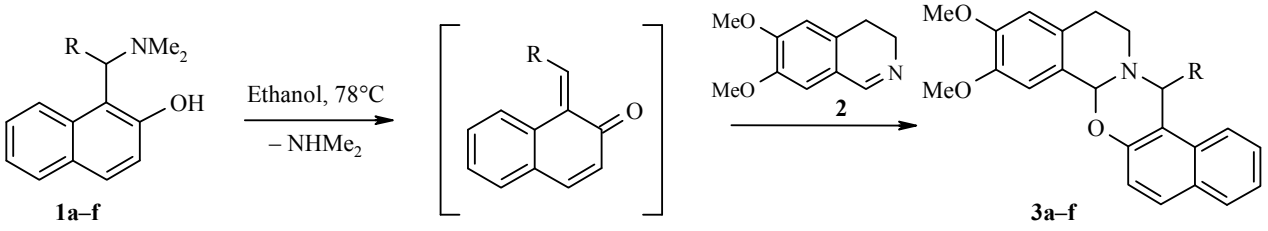
Products of heterocyclization, 9,10-dimethoxy-12,13-dihydro-7aH,15H-naphtho[1',2':5,6][1,3]oxazino-[2,3-a]isoquinolines, were isolated on condensing 6,7-dimethoxy-3,4-dihydroisoquinoline with 1-dimethylaminomethyl-2-naphthols. In the case of o-hydroxybenzyl alcohols products of a Michael aza reaction, 2-[(6,7-dimethoxy-3,4-dihydroisoquinolin-2(1H)-yl)methyl]phenols were obtained.

Keywords: *o*-hydroxybenzyl alcohols, Mannich bases, 6,7-dimethoxy-3,4-dihydroisoquinoline, *o*-quinone methides.

Among the condensed benzo[5,6][1,3]oxazino[2,3-*a*]isoquinolines highly selective antagonists of muscarinic M4 receptors are known [1, 2]. In addition, compounds of this type are of interest as structural analogs of certain isoquinoline alkaloids, primarly tetrahydroprotoberberines [3].

There are only a few examples in the literature of [4+2] cycloaddition of *o*-quinone methides to azomethines [1, 2, 4–8]. At the same time this reaction opens a route to various polyaminals of heterocyclic series.

We have shown that on interacting Mannich bases of the naphthalene series **1a–f** with 6,7-dimethoxy-3,4-dihydroisoquinoline **2** in boiling ethanol or *o*-xylene, 9,10-dimethoxy-12,13-dihydro-7a*H*,15*H*-naphtho-[1',2':5,6][1,3]oxazino[2,3-*a*]isoquinolines **3a–f** were formed.



1a–f **3a–f**

a R = H, **b** R = Ph , **c** R = 4-MeOC$_6$H$_4$, **d** R = 2-FC$_6$H$_4$, **e** R = 3-O$_2$NC$_6$H$_4$, **f** R = 2-thienyl

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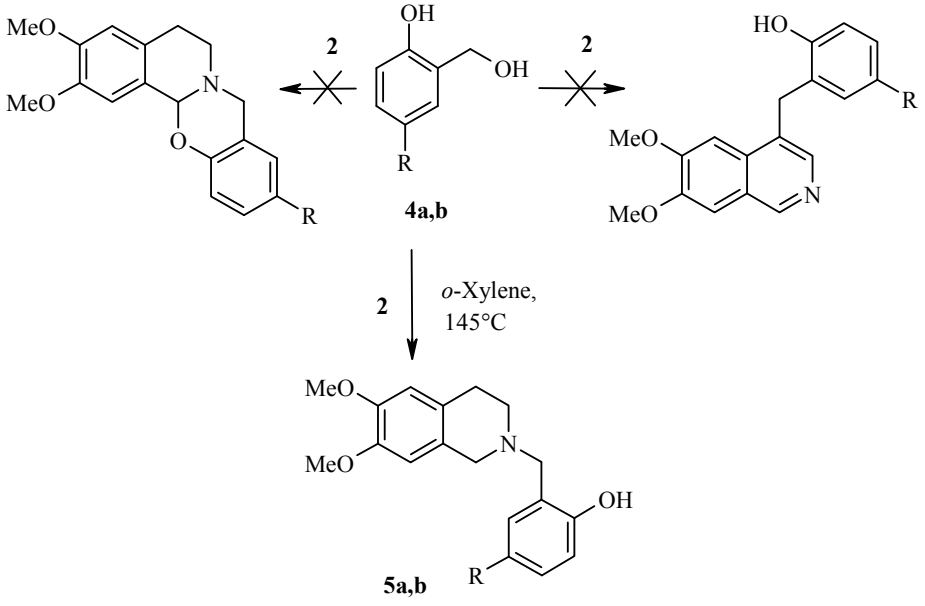
The formation of the cyclic system 12,13-dihydro-7a*H*,15*H*-naphtho-[1',2':5,6][1,3]oxazino[2,3-*a*]isoquinoline may be interpreted as the result of a [4+2] cycloaddition reaction with inverted electronic requirements [9]. *o*-Quinone methide, generated from the Mannich base, plays the role of diene component and 6,7-dimethoxy-3,4-dihydroisoquinoline – the role of heterodienophile.

The obtained oxazino[2,3-*a*]isoquinolines **3a–f** are high-melting, thermostable crystalline substances, readily soluble in chloroform and ethyl acetate, and poorly soluble in water and lower alcohols.

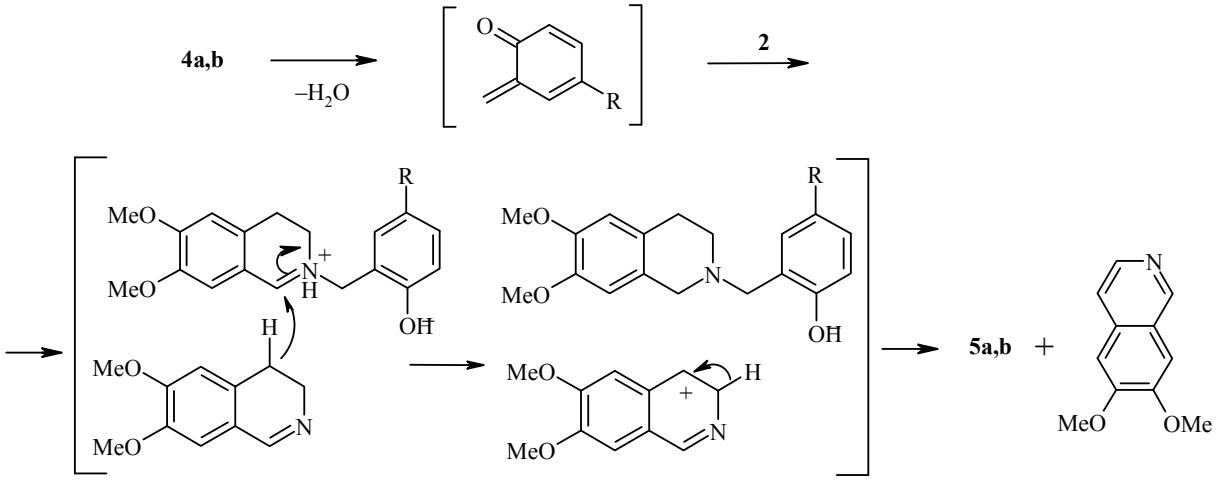
In the mass spectra of compounds **3a–f,** the peaks for the molecular ions are of low intensity. The main direction of fragmentation is retro-Diels–Alder reaction, leading to 6,7-dimethoxy-3,4-dihydroisoquinoline and corresponding *o*-quinone methide. In the ^{1}H NMR spectra, the protons of the methoxy group were displayed as singlets in the 3.84–3.95 region, signals were present for the protons of the methylene units at 2.74-4.78, and also the signals of the methine protons as singlets in the 5.40–5.82 ppm region. The aromatic protons of the isoquinoline fragment resonate at 6.63–6.98 ppm.

The interaction of 3,4-dihydroisoquinoline and salicylic alcohol described in the literature (170°C, sealed tube) [7] leads to 4-(2-hydroxybenzyl)isoquinoline, together with 1,2,3,4-tetrahydroisoquinoline. At the same time in the reaction of 6,7-dimethoxy-3,4-dihydroisoquinoline **2** with *o*-hydroxybenzyl alcohols **4a,b** in boiling *o*-xylene, in place of the products of C-alkylation or cycloaddition expected by us, 2-[(6,7-dimethoxy-3,4-dihydro-2(1*H*)-isoquinolinyl)methyl]phenols **5a,b** were isolated.

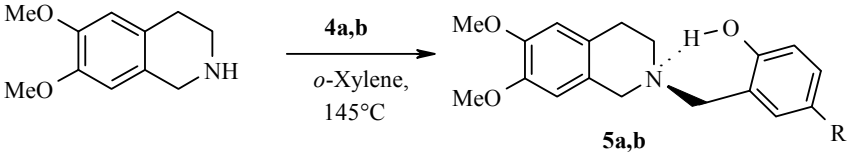


4, 5 a R = H, **b** R = Br

It might have been assumed that in boiling xylene, unlike in ethanol, disproportionation takes place initially of 6,7-dimethoxy-3,4-dihydroisoquinoline into 6,7-dimethoxyisoquinoline and 6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline [10] and then further interaction with *o*-quinone methide, leading to the formation of phenols **5a,b**. However, extended heating of 6,7-dimethoxy-3,4-dihydroisoquinoline (**2**) in boiling *o*-xylene in the absence of *o*-hydroxybenzyl alcohols does not lead to its disproportionation. Consequently, the mechanism of the reaction apparently includes initial quaternization of 6,7-dimethoxy-3,4-dihydroisoquinoline **2**, as a result of which its electrophilic character is increased and migration of hydrogen is facilitated, formally corresponding to a process of intermolecular hydride transfer.



Phenols **5a,b** were obtained by a counter synthesis in higher yields from 6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline and *o*-hydroxybenzyl alcohols **4a,b**.



In the IR spectra of compounds **5a,b,** bands were present for the stretching vibrations of the associated hydrogen bonds of the OH groups in the 2300–3300 cm^{-1} region, which refutes the formation of a cyclic structure. In the mass spectra peaks for molecular ions were of low intensity. The main direction of fragmentation was cleavage of a hydroxybenzyl fragment. In the ^1H NMR spectra the protons of the methylene units were displayed at 2.84–3.88 and signals were present for the protons of the methoxy groups at 3.81–3.86 ppm. Shift of the signal of the OH group protons to the low field region (>10 ppm) confirms the presence of an intramolecular hydrogen bond.

EXPERIMENTAL

The IR spectra were described on a Shimadzu FTIR 8400S instrument in KBr disks. The ^1H NMR spectra were recorded on a JEOL JNM-ECX 400 spectrometer (at 400 MHz) in $CDCl_3$, internal standard was TMS. Mass spectra were obtained on a Finnigan Trace DSQ instrument, energy of ionizing electrons was 70 eV. Elemental analysis was carried out on a EuroVector EA 3000 automatic CHNS analyzer.

The initial Mannich bases **1a–f** were obtained from 2-naphthol by the known procedure of [11, 12].

9,10-Dimethoxy-12,13-dihydro-7a*H*,15*H*-naphtho[1',2':5,6][1,3]oxazino[2,3-*a*]isoquinoline (3a). A mixture of dihydroquinoline **2** (1 g, 5.3 mmol) and Mannich base **1a** (1.05 g, 5.2 mmol) in ethanol (10 ml) was boiled for 1 h, cooled to room temperature, and maintained for 2 h at -20°C. The precipitated solid was filtered off, washed with ice-cold methanol, and recrystallized from an ethanol–DMF mixture. Compound **3a** (1.20 g, 66%) was obtained as colorless crystals, mp 186–187°C (lit., mp 185–187°C [4]). IR spectrum, ν, cm^{-1}: 3001, 2958, 2935, 2885, 2839, 1624, 1601, 1516, 1470, 1393, 1354, 1327, 1269, 1227, 1126, 1107, 1018, 860. ^1H NMR spectrum, δ, ppm (*J*, Hz): 2.91 (2H, br. s, CH_2); 3.08 (2H, br. s, CH_2); 3.91 (3H, s, CH_3); 3.95 (3H, s, CH_3); 4.78 (2H, s, CH_2); 5.75 (1H, s, CH); 6.71 and 6.98 (2H, s, H-8,11); 7.11 (1H, d, *J* = 8.8, H Ar); 7.36–7.41 (1H, m, H Ar); 7.50–7.54 (2H, m, H Ar); 7.68 (1H, d, *J* = 8.1, H Ar); 7.80 (1H, d, *J* = 7.3, H Ar). Mass spectrum, *m/z* (I_{rel}, %): 347 $[M]^+$ (27), 346 $[M-H]^+$ (9), 330 $[M-OH]^+$ (6), 191 $[C_{11}H_{13}NO_2]^+$ (100), 190

$[C_{11}H_{12}NO_2]^+$ (92), 176 $[C_{10}H_{10}NO_2]^+$ (73), 156 $[C_{11}H_8O]^+$ (31), 128 $[C_{10}H_8]^+$ (62). Found, %: C 76.11; H 6.13; N 3.98. $C_{22}H_{21}NO_3$. Calculated, %: C 76.06; H 6.09; N 4.03.

9,10-Dimethoxy-15-phenyl-12,13-dihydro-7a*H*,15*H*-naphtho[1',2':5,6][1,3]oxazino[2,3-*a*]isoquinoline (3b) was obtained analogously to compound **3a** from dihydroisoquinoline **2** (1 g, 5.2 mmol) and Mannich base **1b** (1.45 g, 5.2 mmol) in ethanol (15 ml). Yield 1.62 g (73%) of colorless crystals; mp 155–156°C (2-propanol–DMF). IR spectrum, ν, cm^{-1}: 3063, 3024, 2955, 2916, 2858, 2839, 1620, 1597, 1520, 1466, 1427, 1393, 1362, 1327, 1269, 1231, 1126, 1022, 976. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.81 (1H, dd, *J* = 15.4, *J* = 2.2, CH_2); 3.08 (1H, dd, *J* = 9.5, *J* = 5.9, CH_2); 3.20–3.41 (2H, m, CH_2); 3.88 (3H, s, CH_3); 3.89 (3H, s, CH_3); 5.46 (1H, s, CH); 5.63 (1H, s, CH); 6.70 and 6.85 (2H, s, H-8,11); 7.17 (1H, d, *J* = 8.8, H-6); 7.28–7.34 (7H, m, H Ar); 7.46 (1H, d, *J* = 7.3, H-1); 7.46–7.83 (2H, m, H Ar). Mass spectrum, *m/z* (I_{rel}, %): 423 $[M]^+$ (11), 406 $[M\text{-}OH]^+$ (4), 232 $[C_{17}H_{12}O]^+$ (35), 231 $[C_{17}H_{11}O]^+$ (100), 202 (28), 191 $[C_{11}H_{13}NO_2]^+$ (77), 190 $[C_{11}H_{12}NO_2]^+$ (26), 176 $[C_{10}H_{10}NO_2]^+$ (35). Found,%: C 79.49; H 6.02; N 3.36. $C_{28}H_{25}NO_3$. Calculated, %: C 79.41; H 5.95; N 3.31.

9,10-Dimethoxy-15-(4-methoxyphenyl)-12,13-dihydro-7a*H*15*H*-naphtho[1',2':5,6][1,3]oxazino-[2,3-*a*]isoquinoline (3c) was obtained analogously to compound **3a** from dihydroisoquinoline **2** (1 g, 5.2 mmol) and Mannich base **1c** (1.61 g, 5.2 mmol) in ethanol (15 ml). Yield 1.78 g (75%) of colorless crystals; mp 182-184°C (2-propanol–DMF). IR spectrum, ν, cm^{-1}: 3063, 2928, 2839, 1616, 1512, 1466, 1393, 1273, 1234, 1126, 1026, 976, 852. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.90 (1H, dd, *J* = 15.6, *J* = 2.3, CH_2); 3.05–3.09 (1H, m, CH_2); 3.21 (1H, td, *J* = 15.6, *J* = 5.5, CH_2); 3.30–3.36 (1H, m, CH_2); 3.76 (3H, s, CH_3); 3.88 (6H, s, $2CH_3$); 5.40 (1H, s, CH); 5.62 (1H, s, CH); 6.68 and 6.83 (2H, s, H-8,11); 6.80 (2H, d, *J* = 8.5, H Ar); 7.14 (1H, d, *J* = 8.8, H Ar); 7.20 (2H, d, *J* = 7.8, H Ar); 7.29–7.36 (2H, m, H Ar); 7.45 (1H, d, *J* = 7.8, H Ar); 7.73 (1H, d, *J* = 8.8, H Ar); 7.79 (1H, d, *J* = 7.3, H Ar). Mass spectrum, *m/z*, (I_{rel}, %): 453 $[M]^+$ (8), 436 $[M\text{-}OH]^+$ (1), 262 $[C_{18}H_{14}O_2]^+$ (43), 261 $[C_{18}H_{13}O_2]^+$ (88), 247 (26), 231 $[C_{17}H_{11}O]^+$ (100), 218 (22), 202 (7), 191 $[C_{11}H_{13}NO_2]^+$ (61), 190 $[C_{11}H_{12}NO_2]^+$ (28), 176 $[C_{10}H_{10}NO_2]^+$ (33). Found, %: C 76.85; H 5.96; N 3.14. $C_{29}H_{27}NO_4$. Calculated, %: C 76.80; H 6.00; N 3.09.

15-(2-Fluorophenyl)-9,10-dimethoxy-12,13-dihydro-7a*H*,15*H*-naphtho[1',2':5,6][1,3]oxazino-[2,3-*a*]isoquinoline (3d) was obtained analogously to compound **3a** from dihydroisoquinoline **2** (1 g, 5.2 mmol) and Mannich base **1d** (1.55 g, 5.2 mmol) in ethanol (15 ml). Yield 1.29 g (56%), colorless crystals; mp 160-161°C (2-propanol–DMF). IR spectrum.ν, cm^{-1}: 3063, 2951, 2839, 1620, 1601, 1520, 1485, 1466, 1393, 1335, 1269, 1231, 1126, 1022, 856. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.74 (1H, d, *J* = 15.6, CH_2); 3.12–3.21 (2H, m, CH_2); 3.31–3.40 (1H, m, CH_2); 3.84 (3H, s, CH_3); 3.87 (3H, s, CH_3); 5.68 (1H, s, CH); 5.71 (1H, s, CH); 6.64 and 6.85 (2H, s, H-8,11); 6.88–6.93 (2H, m, H Ar); 7.01–7.14 (2H, m, H Ar); 7.20–7.29 (4H, m, H Ar); 7.72–7.76 (2H, m, H Ar). Mass spectrum, *m/z*, (I_{rel}, %): 441 $[M]^+$ (40), 424 $[M\text{–}OH]^+$ (10), 250 $[C_{17}H_{11}FO]^+$ (46), 249 $[C_{17}H_{10}FO]^+$ (98), 231 $[C_{17}H_{11}O]^+$ (97), 220 (40), 202 (18), 191 $[C_{11}H_{13}NO_2]^+$ (100), 190 $[C_{11}H_{12}NO_2]^+$ (78), 176 $[C_{10}H_{10}NO_2]^+$ (84). Found, %: C 76.22; H 5.53; N 3.15. $C_{28}H_{24}FNO_3$. Calculated, %: C 76.17; H 5.48; N 3.17.

9,10-Dimethoxy-15-(3-nitrophenyl)-12,13-dihydro-7aH,15H-naphtho[1',2':5,6][1,3]oxazino[2,3-*a*]-isoquinoline (3e) was obtained analogously to compound **3a** from dihydroisoquinoline **2** (1 g, 5.2 mmol) and Mannich base **1e** (1.69 g, 5.2 mmol) in ethanol (15 ml). Yield 1.91 g (78%), colorless crystals; mp 203-204°C (2-propanol). IR spectrum, ν, cm^{-1}: 3063, 2928, 2858, 1620, 1528 (NO_2), 1466, 1392, 1350 (NO_2), 1269, 1231, 1126, 1022, 856. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.83 (1H, dd, *J* = 13.2, *J* = 2.9) and 3.12–3.44 (3H, m, $2CH_2$); 3.89 (6H, s, $2CH_3$); 5.48 (2H, s, 2CH); 6.70 and 6.83 (2H, s, H-8,11); 7.17 (1H, d, *J* = 8.8, H Ar); 7.27-7.35 (3H, m, H Ar); 7.43 (1H, dd, *J* = 8.1, *J* = 7.3, H Ar); 7.58 (1H, d, *J* = 7.3, H Ar); 7.78–7.84 (2H, m, H Ar); 8.13 (1H, d, *J* = 8.1, H Ar); 8.27 (1H, s, H Ar). Mass spectrum, *m/z* (I_{rel}, %): 468 $[M]^+$ (12), 451 $[M\text{-}OH]^+$ (3), 277 $[C_{17}H_{11}NO_3]^+$ (15), 276 $[C_{17}H_{10}NO_3]^+$ (23), 260 (37), 231 $[C_{17}H_{11}O]^+$ (42), 230 $[C_{17}H_{10}O]^+$ (79), 202 (52), 191 $[C_{11}H_{13}NO_2]^+$ (100), 190 $[C_{11}H_{12}NO_2]^+$ (47), 176 $[C_{10}H_{10}NO_2]^+$ (64). Found, %: C 71.82; H 5.13; N 6.04. $C_{28}H_{24}N_2O_5$. Calculated, %: C 71.78; H 5.16; N 5.98.

9,10-Dimethoxy-15-(2-thienyl)-12,13-dihydro-7a*H*,15*H*-naphtho[1',2':5,6][1,3]oxazino[2,3-*a*]isoquinoline (3f) was obtained analogously to compound **3a** from dihydroisoquinoline **2** (1 g, 5.2 mmol) and Mannich base **1f** (1.48 g, 5.2 mmol) in ethanol (15 ml). Yield 1.82 g (81%), colorless crystals; mp 199-200°C (ethanol). IR spectrum, ν, cm^{-1}: 3063, 2955, 2843, 1620, 1597, 1520, 1466, 1423, 1389, 1358, 1331, 1269, 1227, 1130, 1018, 976, 856. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.77 (1H, d, *J* = 11.9, CH_2); 3.02-3.08 (1H, m, CH_2); 3.19–3.31 (2H, m, CH_2); 3.88 (6H, s, $2CH_3$); 5.60 (1H, s, CH); 5.82 (1H, s, CH); 6.63 and 6.83 (2H, br. s, H thiophene); 6.69 and 6.87 (2H, s, H-8,11); 7.10 (1H, d, *J* = 8.7, H Ar); 7.26 (1H, d, *J* = 4.6, H thiophene); 7.33 (1H, dd, *J* = 8.3, *J* = 6.9, H Ar); 7.41 (1H, dd, *J* = 7.8, *J* = 6.9, H Ar); 7.64 (1H, d, *J* = 8.3, H Ar); 7.73 (1H, d, *J* = 8.7, H Ar); 7.79 (1H, d, *J* = 7.8, H Ar). Mass spectrum, *m/z* (I_{rel}, %): 429 $[M]^+$ (9), 412 $[M\text{-}OH]^+$ (1), 238 $[C_{15}H_{10}SO]^+$ (58), 237 $[C_{15}H_9SO]^+$ (100), 209 (12), 208 (22), 191 $[C_{11}H_{13}NO_2]^+$ (62), 190 $[C_{11}H_{12}NO_2]^+$ (28), 176 $[C_{10}H_{10}NO_2]^+$. Found, %: C 72.75; H 5.44; N 3.21; S 7.50. $C_{26}H_{23}NO_3S$. Calculated, %: C 72.70; H 5.40; N 3.26; S 7.46.

2-[(6,7-Dimethoxy-3,4-dihydroisoquinolin-2(1*H*)-yl)methyl]phenol (5a). A mixture of alcohol **4a** (0.65 g, 5.2 mmol) and dihydroisoquinoline **2** (2 g, 10.4 mmol) (method A) or 6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline (0.98 g, 5.2 mmol) (method B) in *o*-xylene was boiled for 6 h, the solvent was distilled in vacuum, the residue was dissolved with heating in methanol (10 ml) and the obtained solution was maintained for 1 day at -20°C. The precipitated solid was filtered off, washed with a small amount of ice-cold methanol, and recrystallized from methanol. The product 0.66 g (42%, method A) or 0.97 g (62%, method B) was obtained as colorless crystals; mp 155–156°C (lit. mp 200°C [13]). IR spectrum, ν, cm^{-1}: 2300–3300 (OH), 1609, 1589 (C=C), 1520, 1462, 1420, 1381, 1366, 1350, 1296, 1254, 1227, 1122, 1084, 1014, 987, 964, 860, 768. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.87 (4H, br. s, $2CH_2$); 3.68 (2H, s, CH_2); 3.82 (3H, s, CH_3); 3.85 (3H, s, CH_3); 3.87 (2H, s, CH_2); 6.50 and 6.61 (2H, s, H-5',8'); 6.79–6.86 (2H, m, H-4,6); 7.03 (1H, d, *J* = 7.3, H-3); 7.20 (1H, td, *J* = 7.3, *J* = 1.4, H-5); 10.91 (1H, br. s, OH). Mass spectrum, *m/z* (I_{rel}, %): 299 $[M]^+$ (33), 298 $[M\text{-}H]^+$ (35), 192 $[C_{11}H_{14}NO_2]^+$ (67), 176 $[C_{10}H_{10}NO_2]^+$ (11), 164 (100), 149 (17), 121 (17), 107 $[C_7H_7O]^+$ (30). Found, %: C 72.27; H 7.02; N 4.73. $C_{18}H_{21}NO_3$. Calculated, %: C 72.22; H 7.07; N 4.68.

4-Bromo-2-[(,7-dimethoxy-3,4-dihydroisoquinolin-2(1*H*)-yl)methyl]phenol (5b) was obtained analogously to compound **5a** from alcohol **4b** (1.06 g, 5.2 mmol) and dihydroisoquinoline **2** (2 g, 10.4 mmol, method A) or 6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline (0.98 g, 5.2 mmol, method B) in *o*-xylene (30 ml). Yield 0.97 g (49%) (method A) or 1.36 g (69%) (method B), colorless crystals; mp 154–155°C (methanol). IR spectrum, ν, cm^{-1}: 2300–3100 (OH), 1609, 1582 (C=C), 1516, 1474, 1450, 1346, 1300, 1258, 1231, 1126, 1084, 1018, 964, 825. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.87 (4H, br. s, $2CH_2$); 3.67 (2H, br. s, CH_2), 3.83 (3H, s, CH_3); 3.84 (2H, s, CH_2); 3.86 (3H, s, CH_3); 6.51 and 6.62 (2H, s, H-5',8'); 6.73 (1H d, *J* = 8.8, H-6); 7.15 (1H, d, *J* = 2.2, H-3); 7.27 (1H, dd, *J* = 8.8, *J* = 2.2, H-5); 10.82 1H, br. s, OH). Mass spectrum for ^{79}Br isotope, *m/z* (I_{rel}, %): 377 $[M]^+$ (32), 376 $[M\text{-}H]^+$ (28), 298 $[M\text{-}Br]^+$ (2), 192 $[C_{11}H_{14}NO_2]^+$ (82), 185 $[C_7H_6BrO]^+$ (12), 177 (17), 164 (100), 149 (24), 121 (28). Found, %: C 57.20; H 5.35; N 3.66. $C_{18}H_{20}BrNO_3$. Calculated, %: C 57.16; H 5.33; N 3.70.

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