Synthesis of Podands of the 3,4-Dihydroisoquinoline Series

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Abstract—Podands containing 3,4-dihydroisoquinoline fragments were synthesized by reactions of 1,1'-{ethane-1,2-diylbis[oxy(3-methoxybenzene-4,1-diyl)]}bis(2-methylpropan-1-ol) and 1-(2-ethoxyethoxy)-2-methoxy-4-(2-methylprop-1-en-1-yl)benzene with ethyl cyanoacetate and methyl thiocyanate in concentrated sulfuric acid. Likewise, 1-substituted 3,4-dihydroisoquinolines having a crown ether fragment were obtained from 4-acetylbenzo-12-crown-4.

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Benzocrown ethers are widely used in organic and coordination chemistry [1, 2]. Numerous attempts were made to obtain heterocyclic compounds with both fused crown ether fragment [3, 4] and that connected through a rigid spacer [5]. Also, possible synthetic routes to heterocyclic compounds containing polyether chains, i.e., podands exhibiting interesting complexing properties, were explored in many publications [6, 7].

Both crown-containing heterocyclic compounds and podands are generally synthesized from functionalized aromatic compounds [1, 2]. Following this approach, we have synthesized 4-(2-ethoxyethoxy)-3methoxybenzaldehyde (I) by alkylation of vanillin with 2-ethoxyethyl methanesulfonate, and the subsequent Grignard reaction of I gave 1-(2-ethoxyethoxy)-2-methoxy-4-(2-methylprop-1-en-1-yl)benzene (II). The latter was brought into reactions with ethyl cyanoacetate, acetonitrile, and methyl thiocyanate to obtain the corresponding 3,4-dihydroisoquinoline derivatives **IIIa–IIIc** (Scheme 1).

An interesting pattern was observed in the synthesis of the corresponding carbinol from 4,4'-[ethane-1,2diylbis(oxy)]bis(3-methoxybenzaldehyde) (IV). Addition of the latter to isopropylmagnesium bromide afforded both the expected product, 1,1'-{ethane-1,2diylbis[oxy(3-methoxybenzene-4,1-diyl)]}bis(2-methylpropan-1-ol) (VI), and 1,1'-{ethane-1,2-diylbis[oxy-(3-methoxybenzene-4,1-diyl)]}bis(2-methylpropan-1one) (V) at a ratio of 1:1. Presumably, compound V was formed via Meerwein–Ponndorf–Verley-like reaction [8]. We failed to separate compounds V and VI by column chromatography, but the reduction of mixture V/VI with sodium tetrahydridoborate in ethanol afforded alcohol VI whose reactions with ethyl cyano-





 $R = Me(\mathbf{b}), MeS(\mathbf{c}).$



Scheme 3.



IX

acetate and methyl thiocyanate produced the corresponding 3,4-dihydroisoquinoline derivatives **VIIa** and **VIIb** (Scheme 2). Likewise, crown-containing compounds **VIIIa–VIIIc** were obtained from 4-acetyl-benzo-12-crown-4 (Scheme 3).

Podands containing a 3,4-dihydroisoquinoline fragment can also be synthesized via three-component condensation [9]. For instance, the reaction of 1,2-bis-(2-ethoxyethoxy)benzene with isobutyraldehyde and ethyl cyanoacetate in 96% sulfuric acid gave ethyl 2-[6,7-bis(2-ethoxyethoxy)-3,3-dimethyl-3,4-dihydroisoquinolin-1(2*H*)-ylidene]ethanoate (**IX**) (Scheme 4).

EXPERIMENTAL

The melting points were determined on a PTP melting point apparatus. The IR spectra were recorded on a Bruker IFS-66 spectrometer from solutions in chloroform (II, IIIa-IIIc, VI, VIIb) or Nujol mulls (VIIIa–VIIIc, IX). The ¹H and ¹³C NMR spectra were measured on a Varian Mercury Plus 300 instrument at 300 and 75 MHz, respectively, using CDCl₃ as solvent and hexamethyldisiloxane as internal reference. The mass spectra (electron impact, 70 eV) were obtained on an Agilent Technologies 6890N/5975B GC/MS system. Analysis of compounds IIIa and VIIIa revealed products of their deethoxycarbonylation (compounds **IIIb** and **VIIIb**, respectively), which were likely to be formed due to thermolysis in the injector. The elemental compositins were determined on a Leco Corporation CHNS-932 analyzer. The progress of reactions and the purity of products were monitored by TLC on Sorbfil UV-254 plates using ethyl acetate and ethyl acetate-hexane (1:1) as eluents; spots were visualized by treatment with a 0.5% solution of chloranil in toluene.

4-(2-Ethoxyethoxy)-3-methoxybenzaldehyde (I). Vanillin, 0.1 mol, was added to a solution of 0.1 mol of potassium hydroxide in 50 mL of dimethyl sulfoxide, and the mixture was heated on a boiling water bath. 2-Ethoxyethyl methanesulfonate, 0.1 mol, was then added, and the mixture was heated for 6 h at 40°C. The mixture was washed with a solution of potassium hydroxide and treated with diethyl ether, the extract was washed with water and dried over Na₂SO₄, and the solvent was distilled off. Yield 12.5 g (56%), oily substance. ¹H NMR spectrum, δ , ppm: 1.21 t (3H, OCH₂**Me**, *J* = 6.9 Hz), 3.59 q (2H, OCH₂, *J* = 7.0 Hz), 3.84 t (2H, OCH₂, *J* = 5.1 Hz), 3.90 s (3H, OMe), 4.24 t (2H, OCH₂, *J* = 5.1 Hz), 7.38–7.43 m (2H, H_{arom}), 7.00 d (1H, H_{arom}, *J* = 8.1 Hz), 9.83 s (1H, CHO). Mass spectrum, m/z (I_{rel} , %): 224 (38) [M]⁺, 152 (21), 151 (39). Found, %: C 63.99; H 7.31. C₁₂H₁₆O₄. Calculated, %: C 64.27; H 7.19. M 224.25.

1-(2-Ethoxyethoxy)-2-methoxy-4-(2-methylprop-1-en-1-yl)benzene (II). Yield 48%, bp 163–165°C (6 mm). IR spectrum, v, cm⁻¹: 2980, 2945, 2882, 2859, 1513. ¹H NMR spectrum, δ , ppm: 1.21 t (3H, OCH₂**Me**, J = 6.9 Hz), 1.85 d and 1.86 d (3H each, Me, J = 6.3 Hz), 3.58 q (2H, OCH₂, J = 7.05 Hz), 3.78–3.84 m (2H, OCH₂), 3.82 s (3H, OMe), 4.15 t (2H, OCH₂, J = 5.25 Hz), 6.19 s (1H, CH), 6.74–6.76 m (2H, H_{arom}), 6.84–6.87 m (1H, H_{arom}). Mass spectrum, m/z (I_{rel} , %): 250 (83) [M]⁺, 178 (100), 163 (23), 131 (26). Found, %: C 71.78; H 8.62. C₁₅H₂₂O₃. Calculated, %: C 71.97; H 8.86. M 250.33.

1-Substituted 3,4-dihydroisoquinolines IIIa–IIIc, VIIa, VIIb, VIIIa, and VIIIc (general procedure). A mixture of 0.01 mol of the corresponding alcohol or alkenylbenzene and 0.01 mol of nitrile was added dropwise to 5 mL of 96% sulfuric acid cooled to 5– 10°C. The mixture was stirred for 1.5 h at 20–25°C, poured into water, and neutralized to pH 7–8 with sodium carbonate. The product was extracted into methylene chloride, the extract was washed with water and dried over anhydrous MgSO₄, the solvent was distilled off, and the residue was purified by column chromatography or recrystallization.

Ethyl 2-[7-(2-ethoxyethoxy)-6-methoxy-3,3-dimethyl-3,4-dihydroisoquinolin-1(2H)-ylidene]ethanoate (IIIa). Yield 2.14 g (59%), light yellow crystals, mp 92–93°C (from EtOH). IR spectrum, v, cm⁻¹: 3274 (NH), 3189 (NH), 2972, 2933, 2870, 1647 (C=N), 1600, 1571, 1514. ¹H NMR spectrum, δ, ppm: 1.21–1.31 m (6H, OCH₂Me), 1.26 s (6H, Me), 2.74 s $(2H, CH_2), 3.59 q (2H, OCH_2, J = 7.0 Hz), 3.79-$ 3.80 m (2H, OCH₂), 3.87 s (3H, OMe), 4.11-4.19 m (4H, OCH₂), 5.01 s (1H, CH), 6.60 s (1H, H_{arom}), 7.22 s (1H, H_{arom}), 8.91 br.s (1H, NH). ¹³C NMR spectrum, δ_C, ppm: 14.6, 15.0, 28.3, 41.5, 49.2, 55.7, 58.3, 66.7, 68.7, 68.8, 76.4, 76.6, 110.5, 111.4, 120.4, 129.1, 146.9, 151.6, 155.3, 171.0. Found, %: C 65.76; H 7.96; N 3.59. C₂₀H₂₉NO₅. Calculated, %: C 66.09; H 8.04; N 3.85.

7-(2-Ethoxyethoxy)-6-methoxy-1,3,3-trimethyl-3,4-dihydroisoquinoline (IIIb). Yield 1.07 g (37%), light yellow crystals, mp 65–66°C (from hexane– EtOAc). IR spectrum, v, cm⁻¹: 2977, 2939, 2917, 2868, 1624 (C=N), 1604, 1571. ¹H NMR spectrum, δ , ppm: 1.18 s (6H, Me), 1.23 t (3H, OCH₂**Me**, *J* = 6.9 Hz), 2.31 s (3H, Me), 2.60 s (2H, CH₂), 3.60 q (2H, OCH₂, J = 6.9 Hz), 3.81 t (2H, OCH₂, J = 5.1 Hz), 3.88 s (3H, OMe), 4.19 t (2H, OCH₂, J = 5.1 Hz), 6.63 s (1H, H_{arom}), 7.11 s (1H, H_{arom}). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 14.9, 23.1, 27.7, 38.3, 53.2, 55.5, 66.5, 68.8, 69.1, 111.0, 112.3, 121.1, 130.4, 146.2, 151.5, 160.3. Mass spectrum, m/z ($I_{\rm rel}$, %): 291 (100) [M]⁺, 276 (42) [M - Me]⁺, 218 (11), 204 (31). Found, %: C 69.76; H 8.43; N 4.55. C₁₇H₂₅NO₃. Calculated, %: C 70.07; H 8.65; N 4.81. M 291.39.

7-(2-Ethoxyethoxy)-6-methoxy-3,3-dimethyl-1methylsulfanyl-3,4-dihydroisoquinoline (IIIc). Yield 1.42 g (44%), white crystals, mp 54-55°C (from EtOH). IR spectrum, v, cm⁻¹: 2968, 2925, 2870, 1596, 1563, 1512. ¹H NMR spectrum, δ, ppm: 1.18 s (6H, Me), 1.24 t (3H, OCH₂Me, J = 7.0 Hz), 2.40 s (3H, SMe), 2.61 s (2H, CH₂), 3.60 q (2H, OCH₂, J =7.0 Hz), 3.82 t (2H, OCH₂, J = 5.1 Hz), 3.87 s (3H, OMe), 4.19 t (2H, OCH₂, J = 5.1 Hz), 6.62 s (1H, H_{arom}), 7.23 s (1H, H_{arom}). ¹³C NMR spectrum, δ_{C} , ppm: 12.2, 15.1, 28.3, 38.9, 55.7, 55.9, 66.8, 68.8, 68.9, 110.7, 111.0, 121.2, 129.9, 146.5, 151.6, 159.4. Mass spectrum, m/z (I_{rel} , %): 323 (14) $[M]^+$, 308 (100) $[M - Me]^+$, 250 (16). Found, %: C 62.90; H 7.71; N 4.50; S 9.62. C₁₇H₂₅NO₃S. Calculated, %: C 63.13; H 7.79; N 4.33; S 9.91. M 323.45.

4,4'-[Ethane-1,2-diylbis(oxy)]bis(3-methoxybenzaldehyde) (IV) was synthesized by alkylation of vanillin with methylene bromide in DMSO according to [10]. Yield 47%, light yellow crystals, mp 179– 181°C; published data [10]: mp 169°C. ¹H NMR spectrum, δ , ppm: 3.90 s (6H, OMe), 4.54 s (4H, OCH₂CH₂O), 7.10 d (2H, H_{arom}, J = 8.1 Hz), 7.41– 7.46 m (4H, H_{arom}), 9.86 s (2H, CHO). Mass spectrum, m/z (I_{rel} , %): 330 (56) [M]⁺, 180 (11), 179 (100), 165 (12), 164 (22), 152 (15), 151 (48), 150 (13), 149 (32). Found, %: C 65.16; H 5.25. C₁₈H₁₈O₆. Calculated, %: C 65.45; H 5.49. *M* 330.33.

1,1'-{Ethane-1,2-diylbis[oxy(3-methoxybenzene-4,1-diyl)]}bis(2-methylpropan-1-ol) (VI). Yield 69%, white crystals, mp 112–113°C. IR spectrum, v, cm⁻¹: 3432 br (OH), 2957, 2930, 2871, 1592, 1513. ¹H NMR spectrum, δ , ppm: 0.76 d (6H, Me, J = 7.2 Hz), 1.01 d (6H, Me, J = 6.6 Hz), 1.88–1.95 m (4H, CH, OH), 3.85 s (6H, OMe), 4.27 d (2H, CH, J = 6.9 Hz), 4.40 s (4H, OCH₂), 6.80 d.d (2H, H_{arom}, J = 8.1, 1.8 Hz), 6.87 d (2H, H_{arom}, J = 1.8 Hz), 6.92 d (2H, H_{arom}, J = 8.1 Hz). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 18.4, 19.0, 35.2, 55.9, 67.6, 79.9, 110.2, 118.9, 137.3, 147.4, 149.5. Mass spectrum, m/z ($I_{\rm rel}$, %): 418 (31) [M]⁺, 382 (28), 357 (100), 205 (43), 190 (26), 179 (72), 178 (21). Found, %: C 68.61; H 8.25. C₂₄H₃₄O₆. Calculated, %: C 68.87; H 8.19. *M* 418.52.

Diethyl 7,7'-[ethane-1,2-diylbis(oxy)]bis{[6-me-thoxy-3,3-dimethyl-3,4-dihydroisoquinolin-1(2*H***)ylidene]ethanoate} (VIIa). Yield 1.16 g (19%), white crystals, mp 185°C (decomp.; from EtOH). ¹H NMR spectrum, \delta, ppm: 1.24–1.31 m (18H, Me), 2.74 s (4H, CH₂), 3.88 s (6H, OMe), 4.14 q (4H, OCH₂Me), 4.41 s (4H, OCH₂CH₂O), 5.02 s (2H, CH), 6.61 s (2H, H_{arom}), 7.25 s (2H, H_{arom}), 8.92 br.s (2H, NH). Mass spectrum,** *m***/***z* **(***I***_{rel}, %): 464 (14) [***M* **– 2CO₂Et + 2H]⁺, 450 (30), 449 (100) [***M* **– 2CO₂Et + 2H – Me]⁺, 219 (16), 218 (22). Found, %: C 66.85; H 7.22; N 4.43. C₃₄H₄₄N₂O₈. Calculated, %: C 67.09; H 7.29; N 4.60.** *M* **608.72.**

7,7'-[Ethane-1,2-diylbis(oxy)]bis[6-methoxy-3,3-dimethyl-1-(methylsulfanyl)-3,4-dihydroisoquinoline] (VIIb). Yield 0.74 g (14%), light yellow crystals, mp 196–197°C (from EtOH). IR spectrum, v, cm⁻¹: 2958, 2921, 2851, 1597, 1514. ¹H NMR spectrum, δ , ppm: 1.24 s (12H, Me), 2.41 s (6H, SMe), 2.62 s (4H, CH₂), 3.87 s (6H, OMe), 4.43 s (4H, OCH₂CH₂O), 6.63 s (2H, H_{arom}), 7.27 s (2H, H_{arom}). Mass spectrum, m/z (I_{rel} , %): 528 (13) [M]⁺, 515 (14), 514 (32), 513 (100) [M – Me]⁺, 250 (30), 249 (30). Found, %: C 63.86; H 6.83; N 5.36; S 11.89. C₂₈H₃₆N₂O₄S₂. Calculated, %: C 63.61; H 6.86; N 5.30; S 12.13. M 528.73.

Ethyl 2-{14,14,15-trimethyl-2,3,5,6,8,9,14,15octahydro[1,4,7,10]tetraoxacyclododeca[2,3-g]isoquinolin-12(13*H*)-ylidene}ethanoate (VIIIa) was purified by column chromatography (chloroform– acetone, 9:1). Yield 46%, oily substance, R_f 0.3. IR spectrum, v, cm⁻¹: 1630, 1595, 1512. ¹H NMR spectrum, δ, ppm: 1.13–1.31 m (12H, Me), 2.62 q (1H, CH, J = 7.2 Hz), 3.77 s (4H, O(CH₂)₂O]; 3.81–3.83 m, 3.87–3.90 m, and 4.21–4.23 m [10H, O(CH₂)₂O, OCH₂Me]; 4.97 s (1H, CH), 6.73 s (1H, H_{arom}), 7.26 s (1H, H_{arom}), 8.81 br.s (1H, NH). Found, %: C 65.39; H 7.47; N 3.26. C₂₂H₃₁NO₆. Calculated, %: C 65.17; H 7.71; N 3.45.

12,14,14,15-Tetramethyl-2,3,5,6,8,9,14,15-octahydro[1,4,7,10]tetraoxacyclododeca[2,3-g]isoquinoline (VIIIb) was synthesized by heating compound VIIIa for 2.5 h in boiling 10% H₂SO₄ and was purified by column chromatography (EtOAc–EtOH, 1:1). Yield 74%, oily substance, R_f 0.2. IR spectrum, v, cm⁻¹: 1624 (C=N), 1600, 1572, 1515. ¹H NMR spectrum, δ , ppm: 1.12 d (3H, Me, J = 7.2 Hz); 1.23 s, 1.30 s, and 2.52 s (3H each, Me); 2.68 q (1H, CH, J = 7.2 Hz), 3.77 s [4H, O(CH₂)₂O]; 3.80–3.83 m, 3.89– 3.92 m, and 4.18–4.23 m [8H, O(CH₂)₂O]; 6.78 s (1H, H_{arom}), 7.22 s (1H, H_{arom}). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 15.7, 18.2, 21.1, 23.0, 26.7, 40.5, 56.6, 57.8, 69.4, 69.7, 70.2, 70.6, 71.3, 73.4, 114.3, 118.4, 138.9, 148.3, 155.2, 164.7. Mass spectrum, *m/z* (*I*_{rel}, %): 333 (100) [*M*]⁺, 318 (24) [*M* – Me]⁺, 291 (7), 230 (10), 202 (13). Found, %: C 68.17; H 8.31; N 4.31. C₁₉H₂₇NO₄. Calculated, %: C 68.44; H 8.16; N 4.20. *M* 333.42.

14,14,15-Trimethyl-12-(methylsulfanyl)-2,3,5,6,8,9,14,15-octahydro[1,4,7,10]tetraoxacyclododeca[2,3-g]isoquinoline (VIIIc) was purified by column chromatography (hexane-ethyl acetate, 5:1). Yield 48%, oily substance, R_f 0.2. IR spectrum, v, cm⁻¹: 1590, 1555, 1505. ¹H NMR spectrum, δ, ppm: 1.11 d (3H, Me, J = 6.9 Hz), 1.13 s (6H, Me), 2.40 s (3H, SMe), 2.59 q (1H, CH, J = 6.9 Hz), 3.78 s [4H, O(CH₂)₂O]; 3.80-3.83 m, 3.86-3.89 m, and 4.16-4.23 m [8H, O(CH₂)₂O]; 6.75 s (1H, H_{arom}), 7.26 s (1H, H_{arom}). ¹³C NMR spectrum, δ_{C} , ppm: 12.1, 15.5, 24.2, 28.3, 40.6, 58.4, 70.0, 71.2, 71.5, 72.8, 72.9, 115.1, 115.5, 118.6, 121.2, 137.3, 148.3, 153.1, 158.7. Mass spectrum, m/z (I_{rel} , %): 365 (14) $[M]^+$, 350 (100) [M -Me]⁺. Found, %: C 62.67; H 7.62; N 3.67; S 9.01. C₁₉H₂₇NO₄S. Calculated, %: C 62.44; H 7.45; N 3.83; S 8.77. M 365.49.

Ethyl 2-[6,7-bis(2-ethoxyethoxy)-3,3-dimethyl-3,4-dihydroisoquinolin-1(2H)-ylidene]ethanoate (IX). A mixture of 2.54 g (0.01 mol) of 1,2-bis-(2-ethoxyethoxy)benzene, 0.72 g (0.01 mol) of isobutyraldehyde, and 1.13 g (0.01 mol) of ethyl cyanoacetate was added dropwise to 5 mL of 96% H₂SO₄ cooled to 5-10°C. The subsequent procedure was the same as the general one (see above). The product was purified by column chromatography (hexane-ethyl acetate, 5:1). Yield 1.98 g (47%), light yellow oily substance, $R_{\rm f}$ 0.2. IR spectrum, v, cm⁻¹: 1630, 1595, 1510. ¹H NMR spectrum, δ, ppm: 1.19–1.31 m (9H, Me), 1.25 s (6H, Me), 2.71 s (2H, CH₂); 3.56-3.63 m, 3.77-3.81 m, and 4.12-4.19 m [14H, O(CH₂)₂OCH₂, OCH₂Me]; 5.00 s (1H, CH), 6.65 s (1H, H_{arom}), 7.21 s (1H, H_{arom}), 8.90 br.s (1H, NH). ¹³C NMR spectrum, δ_{C} , ppm: 14.7, 15.1, 28.4, 41.6, 49.4, 58.4, 66.7, 66.8, 68.8, 69.0, 69.4, 76.7, 111.8, 114.0, 121.2, 129.3, 147.5, 151.3, 155.3, 171.1. Mass spectrum, m/z (I_{rel} , %): 349 (100) [$M - CO_2Et + H$]⁺, 334 (22) [$M - CO_2Et + H - Me$]⁺, 276 (16), 204 (10). Found, %: C 65.79; H 8.53; N 3.59. C₂₃H₃₅NO₆. Calculated, %: C 65.53; H 8.37; N 3.32.

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