Electronic supplementary materials *Mendeleev Commun.*, 2018, **28**, 551–552

Synthesis of 1-(hydroxyaryl)furo[3,4-*c*]pyridines from 1-amino(alkoxy)furo[3,4-*c*]pyridines and (poly)phenols

Lyudmila K. Kibardina, Alexey V. Trifonov, Alexey B. Dobrynin, Michael A. Pudovik and Alexander R. Burilov

Experimental
Spectroscopic measurements

NMR experiments were performed on a Bruker AVANCE-400 spectrometer at 303 K equipped with 5 mm broadband probehead working at 400.13 MHz in ^{1}H and 100.61 MHz in ^{13}C experiments. Chemical shifts in ^{1}H spectra were reported relative to the solvent as internal standard (DMSO-d_6). MALDI mass spectra were obtained on an ULTRAFLEX III mass spectrometer. Measurements were conducted with the use of plastic and metal plates. 2,5-Dihydroxybenzoic acid (2,5-DHB) was used as matrix. IR spectra were recorded on a Tensor-27 spectrometer of Bruker Company in the range of 400-3600 cm^{-1} in KBr pellets.

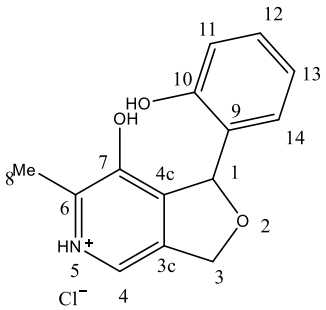
*The X-ray diffraction data for the compounds **2i**.*
Crystals ($C_{14}H_{13}BrNO_4^+$ $Cl^- \cdot 2(C_2H_6O)$ (M = 466.74) are monoclinic, at 296 K a= 12.076(3), b=8.222(2), c= 21.399(5) Å, β = 97.630(3)°, V = 2105.9(9) Å^3, Z= 4, space group P2$_1$/n, d_{calc} = 1.472 g cm^{-3}, μ = 2.111 mm^{-1}, F(000) = 960. The cell parameters and the experimental data were obtained on an automatic Bruker Smart APEX II CCD diffractometer [λ(MoK_α) = 0.71073 Å, ω-scanning], 2θ < 54°, R_{int} = 0.068. A total of 15694 reflections were collected, from which 4595 were independent; the number of the observed reflections with $I > 2\sigma(I)$ was 2406. The absorption correction was applied using the SADABS program.[33] The structure was solved by the direct method using the SIR program,[34] and it was refined by the full-matrix least-squares method using the SHELXL97 program package.[35] The hydrogen atoms of the hydroxyl groups were revealed by means of the difference electron density maps and refined in the isotropic approximation. The coordinates of the other hydrogen atoms were calculated geometrically and refined in a riding model. All the calculations were carried out using the WinGX [36] and APEX2 [37] programs; the final values of the divergence factors were R 0.0502, $_wR_2$ 0.1471, GOF = 1.02; the number of parameters to be refined was 300. The crystallographic data of the structures of **2i** have been deposited with the Cambridge Crystallographic Data Centre (http://www.ccdc.cam.ac.uk; the deposition code is CCDC 1845109)

Synthesis

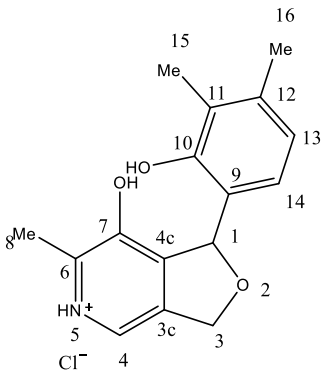
1-Diethylamino-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridine-7-ol (1). a) A mixture of pyridoxal (1 g, 5.99 mmol), diethylamine (0.44 g, 5.99 mmol), and benzene (10 ml) was refluxed for 2 h; the precipitate formed was separated, and the product was recrystallized from benzene. Yield 1.14 g (86%). Mp 91–92°C. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 0.96 t (6H, CH_3, J 7.1 Hz), 2.36 s (3H, CH_3), 2.59 m (4H, CH_2), 4.85 d (1H^a, CH_2O, J 12.5 Hz), 4.95 d (1H^b, CH_2O, J 12.5 Hz), 6.12 s (1H, CH), 7.90 c (1H, CH_{arom}). Found, %: C 64.66; H 8.26; N 12.39. $C_{12}H_{18}N_2O_2$. Calculated, %: C 64.84; H 8.16; N 12.60.

b) A mixture of pyridoxal hydrochloride (2.76 g, 13.53 mmol), diethylamine (3.95 g, 54.12 mmol), and benzene (20 ml) was refluxed for 2 h. Diethylamine hydrochloride was separated from hot solution, the filtrate was concentrated, and the residue was recrystallized from benzene. Yield 2.7 g (90%). Mp 91–92°C. Mass spectrum, *m/z*: 222 $[M]^+$.
c) A mixture of pyridoxal (1.0 g, 5.9 mmol) and *N*,*N*-diethyl-*N*-trimethylsilylamine (0.87 g, 5.9 mmol) of in toluene (10 ml) was maintained at 70°C for 0.5 h. The precipitate formed was recrystallized from diethyl ether. Yield 1.0 g (78 %). Mp 91–92°C. Found, %: C 64.71; H 8.11; N 12.42. $C_{12}H_{18}N_2O_2$. Calculated, %: C 64.84; H 8.16; N 12.60.

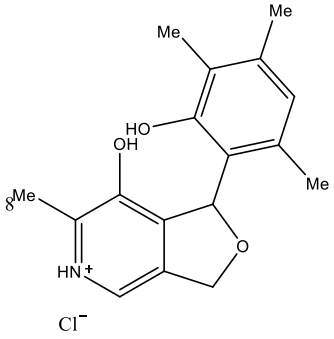
Synthesis of 1-(hydroxyaryl)furo[2,4-*c*]pyridines (2a-i) (general procedure). An equimolar mixture of furopyridine **1**, appropriate phenol, and concentrated hydrochloric acid (1 ml) in anhydrous ethanol (10 ml) was refluxed for 3–5 h. The mixture was cooled, the precipitate was separated, washed with ethanol and diethyl ether, and dried in vacuum. In the case compound 2**e**, the starting reactants were either aminal **1** or acetal **3**.



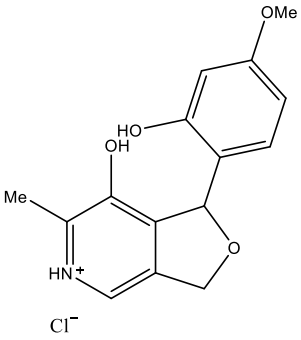
7-Hydroxy-1-(2-hydroxyphenyl)-6-methyl-1,3-dihydrofuro-[3,4-*c*]pyridin-5-ium chloride (2a). Yield 32%. Mp 248-250°C (dec.). IR spectrum, ν, cm^{-1}: 1060, 1555, 3424. ^{1}H NMR (400 MHz, DMSO–d_6), δ: 2.60 s (3H, CH_3), 5.17 d (1H^a, CH_2O, *J* 13.2 Hz), 5.25 dd (1H^b, CH_2O, *J* 13.5, 2.5 Hz), 6.61 d (1H, CH, *J* 1.7 Hz), 6.73 t (1H, CH_{arom}, *J* 6.9 Hz), 6.90 d (1H, CH_{arom}, *J* 8.1 Hz), 6.95 д (1H, CH_{arom}, *J* 7.6 Hz), 7.15 t (1H, CH_{arom}, *J* 8.5 Hz), 8.34 s (1H, CH_{arom}). ^{13}C NMR(DMSO-d_6), δ: 14.94 (C^8), 71.10 (C^3), 80.55 (C^1), 116.34 (C^{11}), 119.33 (C^{13}), 125.00 (C^{14}), 125.67 (C^{12}), 129.24 (C^9), 129.35 (C^4), 130.18 (C^{3c}), 139.49 (C^6), 142.32 (C^{4c}), 148.85 (C^{10}), 155.88 (C^7). MS (MALDI), *m/z*: 243 $[M - HCl]^+$. Found, %: C 60.04; H 4.97; Cl 12.63; N 4.96. $C_{14}H_{14}NO_3Cl$. Calculated, %: C 60.12; H 5.05; Cl 12.67; N 5.01.



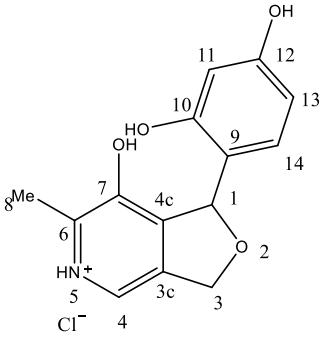
7-Hydroxy-1-(2-hydroxy-3,4-dimethylphenyl)-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2b). Yield 25%. Mp 221°C. IR spectrum, ν, cm^{-1}: 1059, 1553, 3396. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.10 s (3H, CH_3), 2.18 s (3H, CH_3), 2.60 s (3H, CH_3), 5.16 d (1H^a, CH_2O, *J* 13.3 Hz), 5.24 dd (1H^b, CH_2O, *J* 13.3, 1.9 Hz), 6.64 q (2H, CH_{arom}, *J* 7.9 Hz), 6.73 d (1H, CH, *J* 1.6 Hz), 8.33 s (1H, CH_{arom}). ^{13}C NMR spectrum (DMSO-d_6), δ: 12.76 (C^{15}), 14.86 (C^8), 20.43 (C^{16}), 70.87 (C^3), 80.73 (C^1), 121.87 (C^{11}), 124.34 (C^{13}), 125.09 (C^{14}), 125.48 (C^9), 125.60 (C^{12}), 138.47 (C^4), 139.34 (C^{3c}), 142.37 (C^6), 145.25 (C^{4c}), 148.91 (C^{10}), 152.94 (C^7). MS (MALDI), *m/z*: 271 $[M - HCl]^+$. Found, %: C 62.04; H 5.97; Cl 11.63; N 4.76. $C_{16}H_{18}Cl\,NO_3$. Calculated, %: C 62.43; H 5.84; Cl 11.54; N 4.55.



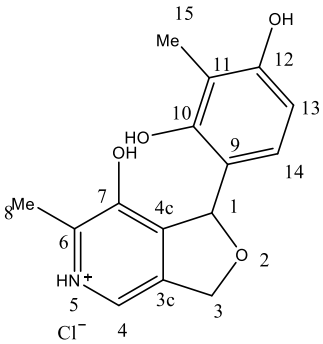
7-Hydroxy-1-(2-hydroxy-3,4,6-trimethylphenyl)-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2c). Yield 27%. Mp> 300°C. IR spectrum, ν, cm^{-1}: 729, 1061, 1530, 1617, 3264, 3340. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 1.96 s (3H, CH_3), 2.12 s (3H, CH_3), 2.21 s (3H, CH_3), 2.60 s (3H, CH_3), 5.17 d (1H^a, CH_2O, *J* 12.8 Hz), 5.35 d (1H^b, CH_2O, *J* 12.8 Hz), 6.53 s (1H, CH), 6.69 s (1H, CH_{arom}), 8.30 s (1H, CH_{arom}). MS (MALDI), *m/z*: 285 $[M-HCl]^+$. Found, %: C 63.22; H 6.17; Cl 11.27; N 4.20. $C_{17}H_{20}ClNO_3$. Calculated, %: C 63.45; H 6.22; Cl 11.04; N 4.35.



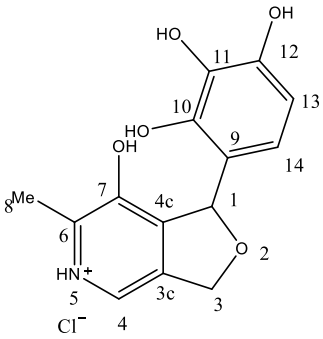
7-Hydroxy-1-(2-hydroxy-4-methoxyphenyl)-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2d). Yield 63%. Mp > 300°C. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.58 s (3H, CH_3), 3.69 s (3H, CH_3), 5.13 d (1H^a, CH_2O, *J* 13.4 Hz), 5.20 dd (1H^b, CH_2O, *J* 13.4, 2.1 Hz), 6.29 m (1H, CH_{arom}), 6.53 d (1H, CH, *J* 1.31 Hz), 6.72 m (1H, CH_{arom}), 8.70 s (1H, CH_{arom}). MS (MALDI), m/z: 274 $[M+H - HCl]^+$.



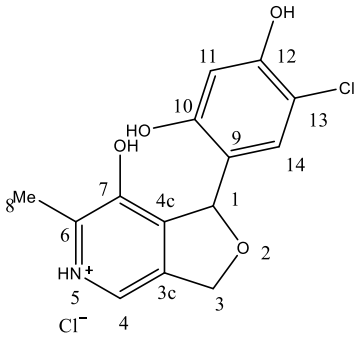
1-(2,4-Dihydroxyphenyl)-7-hydroxy-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2e).
Yield 60% (from **1**) and 73% (from **3**).
Mp > 300°C. IR spectrum, ν, cm^{-1}: 1059, 1597, 1615, 3254. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.60 s (3H, CH_3), 5.10 d (1H^a, CH_2O, *J* 13.3 Hz), 5.19 dd (1H^b, CH_2O, *J* 13.3, 1.7 Hz), 6.14 dd (1H, CH_{arom}, *J* 8.3, 2.3 Hz), 6.37 d (1H, CH_{arom}, *J* 2.3 Hz), 6.51 d (1H, CH, *J* 1.7 Hz), 6.69 d (1H, CH_{arom}, *J* 8.3 Hz), 8.31 s (1H, CH_{arom}), 9.40 s (1H, OH). ^{13}C NMR spectrum (DMSO-d_6), δ: 14.95 (C^8), 70.55 (C^3), 80.35 (C^1), 103.30 (C^{11}), 106.64 (C^{13}), 115.87 (C^9), 125.67 (C^{14}), 130.01 (C^4), 139.48 (C^{3c}), 142.21 (C^6), 145.34 (C^{4c}), 148.72 (C^{10}), 156.91 (C^{12}), 159.30 (C^7). MS (MALDI), m/z: 259 $[M-HCl]^+$. Found, %: C 56.81; H 4.63; Cl 12.31; N 4.72. $C_{14}H_{14}ClNO_4$.Calculated, %: C 56.70; H 4.73; Cl 12.10; N 4.73.



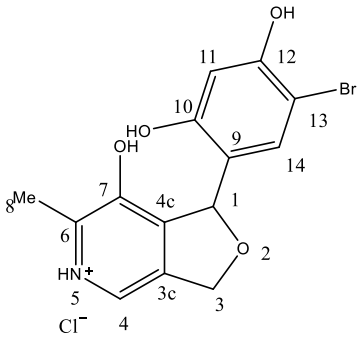
1-(2,4-Dihydroxy-3-methylphenyl)-7-hydroxy-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2f). Yield 36%. Mp > 300°C. IR spectrum, ν, cm^{-1}: 1059, 1606, 3258. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.00 s (3H, CH_3), 2.59 s (3H, CH_3), 5.12 d (1H^a, CH_2O, *J* 13.3 Hz), 5.19 dd (1H^b, CH_2O, *J* 13.3, 2.0 Hz), 6.31 d (1H, CH_{arom}, *J* 8.3 Hz), 6.52 d (1H, CH_{arom}, *J* 8.3 Hz), 6.64 d (1H, CH, *J* 1.6 Hz), 8.31 s (1H, CH_{arom}), 9.33 s (1H, OH). ^{13}C NMR spectrum (DMSO-d_6), δ: 9.54 (C^{15}), 14.92 (C^8), 70.47 (C^3), 80.71 (C^1), 107.11 (C^{13}), 112.41 (C^{11}), 117.39 (C^9), 125.46 (C^{14}), 126.01 (C^4), 139.37 (C^{3c}), 142.29 (C^6), 145.41 (C^{4c}), 148.90 (C^{10}), 154.37 (C^{12}), 157.11 (C^7). MS (MALDI), m/z: 273 $[M-HCl]^+$. Found, %: C 57.89; H 4.95; Cl 11.07; N 4.21. $C_{15}H_{16}ClNO_4$. Calculated, %: C 58.06; H 5.16; Cl 11.16; N 4.52.



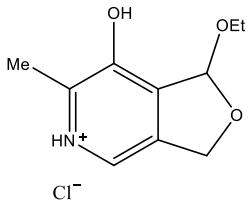
7-Hydroxy-6-methyl-1-(2,3,4-trihydroxyphenyl)-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2g). Yield 72%. Mp > 300°C. IR spectrum, ν, cm^{-1}: 1060, 3192. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.58 s (3H, CH_3), 5.11 d (1H^a, CH_2O, *J* 13.3 Hz), 5.18 dd (1H^b, CH_2O, *J* 13.3, 1.6 Hz), 6.23 m (2H, CH_{arom}), 6.54 d (1H, CH, *J* 1.9 Hz), 8.30 s (1H, CH_{arom}), 9.15 s (1H, OH). ^{13}C NMR spectrum (DMSO-d_6), δ: 14.93 (C^8), 70.54 (C^3), 80.58 (C^1), 107.17 (C^{13}), 117.14 (C^{14}), 118.84 (C^9), 125.59 (C^{11}), 133.76 (C^4), 139.42 (C^{3c}), 142.22 (C^6), 145.11 (C^{10}), 145.33 (C^{4c}), 147.19 (C^{12}), 148.79 (C^7). MS (MALDI), *m/z*: 275 $[M-HCl]^+$. Found, %: C 53.80; H 4.15; Cl 11.57; N 4.59. $C_{14}H_{14}ClNO_5$. Calculated, %: C 53.85; H 4.49; Cl 11.53; N 4.49.



1-(5-Chloro-2,4-dihydroxyphenyl)-7-hydroxy-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2h). Yield 36%. Mp > 300°C. IR spectrum, ν, cm^{-1}: 728, 1060, 1552, 3137, 3426. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.59 s (3H, CH_3), 5.10 d (1H^a, CH_2O, *J* 13.1 Hz), 5.22 dd (1H^b, CH_2O, *J* 13.1, 2.4 Hz), 6.46 d (1H, CH, *J* 1.6 Hz), 6.62 s (1H, CH_{arom}), 6.86 s (1H, CH_{arom}), 8.31 s (1H, CH_{arom}), 10.20 c (1H, OH). ^{13}C NMR spectrum (DMSO-d_6), δ: 14.98 (C^8), 70.89 (C^3), 80.10 (C^1), 104.52 (C^{11}), 109.83 (C^{13}), 117.33 (C^9), 125.72 (C^{14}), 129.95 (C^4), 139.49 (C^{3c}), 142.34 (C^6), 144.34 (C^{4c}), 148.77 (C^{12}), 154.43 (C^{10}), 155.77 (C^7). MS (MALDI), *m/z*: 294 $[M+H-HCl]^+$. Found, %: C 51.04; H 3.60; Cl 21.23; N 4.20. $C_{14}H_{13}Cl_2NO_4$. Calculated, %: C 50.91; H 3.94; Cl 21.52; N 4.24.



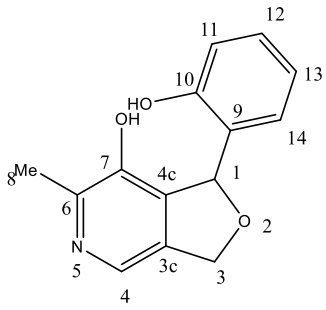
1-(5-Bromo-2,4-dihydroxyphenyl)-7-hydroxy-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (2i). Yield 35%. Mp 230°C (dec). IR spectrum, ν, cm^{-1}: 729, 1062, 1555, 3135, 3428. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.60 s (3H, CH_3), 5.11 d (1H^a, CH_2O, *J* 13.1 Hz), 5.23 dd (H^b, CH_2O, *J* 13.1, 2.1 Hz), 6.46 d (1H, CH, *J* 1.7 Hz), 6.66 s (1H, Ph), 7.01 s (1H, CH_{arom}), 8.31 s (1H, CH_{arom}), 10.31 s (1H, OH). ^{13}C NMR spectrum (DMSO-d_6), δ: 14.97 (C^8), 70.95 (C^3), 80.18 (C^1), 98.51 (C^{13}), 104.39 (C^{11}), 118.00 (C^9), 125.69 (C^{14}), 132.83 (C^4), 139.51 (C^{3c}), 142.30 (C^6), 144.91 (C^{4c}), 148.76 (C^{12}), 155.47 (C^{10}), 156.44 (C^7). MS (MALDI), *m/z*: 339.5 $[M+H-HCl]^+$. Found, %: C 44.62; H 3.35; Br 21.11; Cl 9.57; N 3.59. $C_{14}H_{13}BrCl\ NO_4$. Calculated, %: C 44.80; H 3.20; Br 21.33; Cl 9.47; N 3.73.



7-Hydroxy-1-ethoxy-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-5-ium chloride (3).

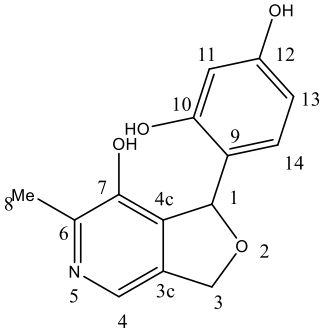
a) To a suspension of pyridoxal (1.3 g, 7.78 mmol) in anhydrous ethanol (10 ml), concentrated hydrochloric acid (1 ml) was added. The mixture was refluxed for 3 h, the solvent was removed, and the residue was washed with diethyl ether. Yield 1.26 g (83%). Mp >300°C. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 1.55 t (3H, CH_3, *J* 7.1 Hz), 2.95 s (3H, CH_3), 4.12 q (2H, CH_2, *J* 7.1 Hz), 5.45 d (1H^a, CH_2O, *J* 13.70 Hz), 5.52 d (1H^b, CH_2O, *J* 13.70 Hz), 6.90 s (1H, CH), 8.56 s (1H, CH_{arom}). MS (MALDI), *m/z*: 195 $[M-HCl]^+$. Found, %: C 51.72; H 6.03; Cl 15.30; N 6.03. $C_{10}H_{14}Cl\ NO_3$. Calculated, %: C 51.84; H 6.09; Cl 15.30; N 6.05.

b) A solution of diethylamino furopyridine **1** (0.44 g, 1.98 mmol) in ethanol (5 ml) was refluxed for 2 h with hydrochloric acid (1 ml), the solvent was removed. The residue was treated with diethyl ether. Yield 0.23 g (61%), mp >300°C. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 1.53 t (3H, CH_3, *J* 7.1 Hz), 2.91 s (3H, CH_3), 4.09 q (2H, CH_2, *J* 7.1 Hz), 5.43 д (1H^a, CH_2O, *J* 13.7 Hz), 5.44 d (1H^b, CH_2O, *J* 13.7 Hz), 6.86 s (1H, CH), 8.52 s (1H, CH_{arom}). MS (MALDI), *m/z*: 195 $[M-HCl]^+$.

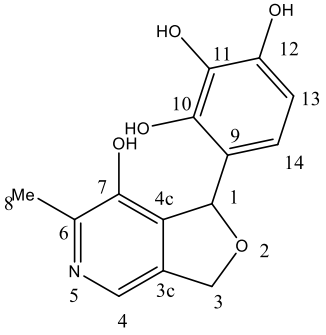


1-(2-Hydroxyphenyl)-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridine-7-ol (4a). A mixture of salt **2a** (0.59 g, 2.11 mmol), sodium hydride (0.11 g, 4.22 mmol), and anhydrous ethanol (10 ml) was refluxed for 1 h, the precipitate was separated, washed with distilled water, and dried in vacuum. Yield 84%. Mp > 300°C. IR spectrum, ν, cm^{-1}: 1056, 1596, 3097. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.35 s (3H, CH_3), 5.09 d (1H^a, CH_2O, J 12.2 Hz), 5.22 dd (1H^b, CH_2O, J 12.2, 0.7 Hz), 6.47 s (1H, CH), 6.74 t (1H, CH_{arom}, J 7.5 Hz), 6.88 d (1H, CH_{arom}, J 7.8 Hz), 6.96 d (1H, CH_{arom}, J 7.7 Hz), 7.11 t (1H CH_{arom}, J 7.7 Hz), 7.89 s (1H, CH_{arom}). ^{13}C NMR spectrum (DMSO-d_6), δ: 17.46 (C^8), 71.33 (C^3), 79.69 (C^1), 116.9 (C^{11}), 119.61 (C^{13}), 127.37 (C^{14}), 127.98 (C^{12}), 128.10 (C^9), 129.44 (C^{3c}), 136.63 (C^{4c}), 139.13 (C^4), 144.02 (C^6), 149.09 (C^7), 155.18 (C^{10}). MS (MALDI), *m/z*: 243.6 $[M]^+$. Found, %: C 69.36; H 5.43; N 5.27. $C_{14}H_{13}NO_3$. Calculated, %: C 69.12; H 5.39; N 5.76.

Compounds **4b,c** were prepared in a similar manner.



4-(7-Hydroxy-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-1-yl)benzene-1,3-diol(4b). Yield 61%. Mp > 300°C. IR spectrum, ν, cm^{-1}: 1054, 1555, 1617, 3184. ^{1}H NMR spectrum (400 МГц, DMSO–d_6), δ: 2.51 s (3H, CH_3), 5.07 d (1H^a, CH_2O, J 12.9 Hz), 5.17 d (1H^b, CH_2O, J 12.9 Hz), 6.16 dd (1H, CH_{arom}, J 8.3, 2.1 Hz), 6.41 d (1H, CH_{arom}, J 2.1 Hz), 6.46 d (1H, CH, J 1.4 Hz), 6.69 d (1H, CH_{arom}, J 8. Hz), 8.13 s (1H, CH_{arom}), 9.40 s (1H, OH). ^{13}C NMR spectrum (DMSO-d_6), δ: 16.24 (C^8), 70.63 (C^3), 79.91 (C^1), 103.42 (C^{11}), 106.76 (C^{13}), 116.76 (C^9), 127.32 (C^{14}), 129.44 (C^{3c}), 138.18 (C^{4c}), 142.48 (C^4), 143.08 (C^6), 148.41 (C^7), 156.51 (C^{10}), 159.03 (C^{12}). MS (MALDI), *m/z*: 259 $[M]^+$. Found, %: C 64.60; H 5.18; N 4.93. $C_{14}H_{13}NO_4$. Calculated, %: C 64.86; H 5.02; N 5.40.



4-(7-Hydroxy-6-methyl-1,3-dihydrofuro[3,4-*c*]pyridin-1-yl)benzene-1,2,3-triol (4c). Yield 81%. Mp > 300°C. IR spectrum, ν, cm^{-1}: 1033, 1584, 3482. ^{1}H NMR spectrum (400 MHz, DMSO–d_6), δ: 2.21 s (3H, CH_3), 5.05 d (1H^a, CH_2O, J 11.9 Γ Hz ц), 5.20 dd (1H^b, CH_2O, J 11.9, 2.2 Hz 5.97 (1H, CH_{arom}, J 8.4 Hz), 6.24 d (1H, CH, J 2.1 Hz), 6.39 d (1H, CH_{arom}, J 8.4 Hz), 6.69 d (1H, CH_{arom}, J 8.3 Hz), 7.53 s (1H, CH_{arom}). ^{13}C NMR spectrum (DMSO-d_6), δ: 18.87 (C^8), 71.50 (C^3), 80.70 (C^1), 105.02 (C^{13}), 114.86 (C^{14}), 121.46 (C^9), 127.27 (C^{3c}), 134.19 (C^{11}), 135.17 (C^{4c}), 136.85 (C^4), 144.35 (C^6), 144.89 (C^{10}), 147.33 (C^{12}), 151.40 (C^7). MS (MALDI), *m/z*: 275 $[M]^+$. Found, %: C 61.22; H 5.06; N 5.17. $C_{14}H_{13}NO_5$. Calculated, %: C 61.09; H 4.72; N 5.09.