# SYNTHESIS AND REACTIONS OF NEW DERIVATIVES OF FURO[3,2-*b*]PYRROLE

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**Abstract:** Synthesis of methyl 4*H*-furo[3,2-*b*]pyrrole-5-carboxylate is taken place in two step synthesis. Vilsmeier-Haack reaction of 4*H*-furo[3,2-*b*]pyrrole-5-carboxylate **1** led to methyl 2-formyl-4*H*-furo[3,2-*b*]pyrrole-5-carboxylate **4**, which served as starting compound for synthesis of furo[3,2-*b*]pyrrole-2-aldoxime **5** and methyl 2-[(4-oxo-2-thioxo-1,3-thiazolidin-5-ylidene)methyl]-4*H*-furo[3,2-*b*]pyrrole-5-carboxylate **7**. The hydrazinolysis of **1** was prepared by carbohydrazide **2**, which subsequently reacted with aldehydes to form of derivatives **3**. Pyrazole derivatives **11** were prepared by the reaction of azlactone **9** with derivatives **10**. Reactions were carried out under microwave irradiation or at classical heating.

Key words: aldoxime, carbohydrazide, furo[3,2-b]pyrrole, microwave irradiation, hippuric acid

### **1. Introduction**

During the past few decades many results have been published in the area of the synthesis of heterocyclic compounds containing furo[3,2-*b*]pyrrole skeleton (PUTEROVÁ *et al.*, 2004) due to their biological activity (KRUTOŠÍKOVÁ *et al.*, 1994; GORUGANTULA *et al.*, 2010).

Carboxhydrazides and their derivatives are interesting class of compounds, which exhibits antitubercular (JORDÃO *et al.*, 2011), antimicrobial (PIACZONKA *et al.*, 2013), antifungal (TELVEKAR *et al.*, 2012), anticonvulsant and anti-inflammatory (ULLOORA *et al.*, 2013) activities.

The present paper is a continuation of previous research, which dealt with the synthesis and reactions of furo[3,2-*b*]pyrrole system (GAŠPAROVÁ *et al.*, 2005; GAŠPAROVÁ *et al.*, 2007).

### 2. Material and methods

Melting points of products were determined on a Kofler hot place and are uncorrected. <sup>1</sup>H NMR spectra were obtained on a 300 MHz spectrometer VARIAN GEMINI 2000 in CDCl<sub>3</sub> or DMSO-d<sub>6</sub> with tetramethylsilane as the internal standard. The infrared spectra (IR) were taken on a FTIR IRAffinity-1 spectrophotometer using KBr technique. All microwave experiments were performed in a Panasonic NN-E205 type microwave oven. The apparatus was adapted for laboratory applications; n-hexane was used as coolant for the condenser. Methyl 4*H*-furo[3,2-*b*]pyrrole-5-carboxylate (**4**), 4*H*-furo[3,2-*b*]pyrrole-5-carbohydrazide (**5**) and methyl 2-formyl-4*H*-furo[3,2-*b*]pyrrole-5-carboxylate (**7**) were synthesized following the published procedures (KRUTOŠÍKOVÁ *et al.*, 1994; GAJDOŠ *et al.*, 2005).

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### 2.1. Synthesis of compounds 3a-d

The mixture of 4H-furo[3,2-*b*]pyrrole-5-carbohydrazide **2** (1.21 mmol), 5-substituted furan-2-karbaldehyde (1.21 mmol) in ethanol (5 ml), and catalytic amount of 4-methylbenzenesulfonic acid was refluxed at 70-80°C for 0.5-1 h (Scheme 1). After cooling, the solid product was filtered off, washed with ethanol and recrystallized from ethanol.



2.1.1 N'-{[5-(4-nitrophenyl)furan-2-yl]methylidene}-4H-furo[3,2-b]pyrrole-5-carbohydrazide (**3***a*)

Yield 48 %; m. p 279-282 °C. Calcd. for  $C_{18}H_{12}N_4O_5$  (364.31) C, 59.34; H, 3.32; N, 15.38. Found: C, 58.84; H, 3.24; N, 14.76 %. IR (KBr): 3350, 3140, 1650, 1594, 1510, 1330, 1275, 1137, 1075, 1022, 915, 852, 753, 656 cm<sup>-1</sup>. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 11.71 (s,1H, NH); 11.54 (s, 1H, NH); 8.75 (d, 2H, J = 7.8 Hz, H-11', H-7'); 7.78 (d, 1H, J = 4.5 Hz, H-2); 7.73 (d, 2H, J = 8.4 Hz, H-10', H-8'); 7.5 (s, 1H, H-7); 6.98 (s, 1H, H-6); 6.28 (d, 1H, J = 3 Hz, H-3'); 5.88 (s, 1H, H-3).

#### 2.1.2 N'-{[5-(4-methylphenyl)furan-2-yl]methylidene}-4H-furo[3,2-b]pyrrole-5carbohydrazide (**3b**)

Yield 53 %; m. p 207-210 °C. Calcd. for  $C_{19}H_{15}N_3O_3$  (333.34) C, 68.46; H, 4.54; N, 12.61. Found: C, 67.80; H, 4.39; N, 12.40 %. IR (KBr): 3453, 3235, 3113, 1634, 1616, 1571, 1490, 1431, 1251, 1137, 1072, 1028, 986, 819, 724 cm<sup>-1</sup>. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 11.58 (s,1H, NH); 11.59 (s, 1H, NH); 8.28 (s, 1H, H-7); 7.77 (d, 1H, J = 4.5 Hz, H-2); 7.70 (d, 2H, J = 7.8 Hz, H-11', H-7'); 7.31 (d, 2H, J = 8.4 Hz, H-10', H-8'); 7.07 (d, 1H, J = 3.3 Hz, H-4'); 7.02 (m, 2H, H-6, H-3'); 6.60 (d, 1H, J = 3.9 Hz, H-3); 2.36 (s, 3H, CH<sub>3</sub>).

2.1.3 N'-[(5-phenylfuran-2-yl)methylidene]-4H-furo[3,2-b]pyrrole-5-carbohydrazide (**3c**)

Yield 35 %; m. p 198-200 °C. Calcd. for  $C_{18}H_{13}N_3O_3$  (319.31) C, 67.71; H, 4.10; N, 13.16. Found: C, 67.38; H, 3.96; N, 13.62 %. IR (KBr): 3331, 3124, 1631, 1471, 1441, 1358, 1305, 1273, 1135, 1075, 1022, 914, 806, 762, 734, 688, 646 cm<sup>-1</sup>. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 11.53 (br, 2H, NH); 8.28 (s, 1H, H-7); 7.70 (m, 3H, H-2, H-7', H-11'); 7.49 (m, 3H, H-10', H-8'); 7.37 (m, 1H, H-9'); 7.02 (d, 1H, J = 3 Hz, H-4'); 7.04 (d, 1H, J = 3 Hz, H-3'); 6.99 (s, 1H, H-6); 6.60 (d, 1H, J = 3.9 Hz, H-3).

### 2.1.4 4-Methyl-2-{[2-({2-[3-(trifluoromethyl)phenyl]-4H-furo[3,2-b]pyrrol-5yl}carbonyl)hydrazinylidene]methyl}-4H-furo[3,2-b]pyrrole-5-carboxylic acid (**3d**)

Yield 40 %; m. p 244-246 °C. IR (KBr): 3257, 2962, 2596, 1674, 1614, 1533, 1448, 1332, 1261, 1222, 1161, 1072, 957, 796, 694 cm<sup>-1</sup>. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 11.84 (s,1H, NH); 11.66 (s, 1H, NH); 8.27 (s, 1H, H-7); 8.12 (m, 2H, H-7", H-9"); 7.67 (m, 2H, H-10", H-11"); 7.43 (s, 1H, H-6'); 7.25 (s, 1H, H-6); 7.04 (s, 1H, H-3'); 7.03(s, 1H, H-3); 6.82 (s, 1H, H-5'); 3.96 (s, 3H, CH<sub>3</sub>).

# 2.2 Synthesis of methyl 2-[(hydroxyimino)methyl]-4H-furo[3,2-b] pyrrole-5-carboxylate (5)

**Classical heating (A).** To mixture of ethanol (26 ml) and water (8 ml) was added hydroxylamine hydrochloride (0.6 g, 9 mmol), sodium hydroxide (2 g, 50 mmol) and methyl 2-formyl-4H-furo[3,2-b]pyrrole-5-carboxylate **4** (0.38 g, 2 mmol) (Scheme 1). The reaction mixture was stirred at room temperature for 24 h. The precipitate was filtered off, washed with water and recrystallized from methanol.

**Microwave method (B)**. To mixture of ethanol (26 ml) and water (8 ml) was added hydroxylamine hydrochloride (0.6 g, 9 mmol) and methyl 2-formyl-4*H*-furo[3,2-*b*]pyrrole-5-carboxylate **4** (0.38 g, 2 mmol) (Scheme 1). The reaction mixture was irradiated at 90 W for 13 min. After cooling, the solid product was filtered off, washed with water and recrystallized from methanol.

Yield: 50 % (A), 70 % (B); m. p 236-241 °C. Calcd. for  $C_9H_8N_2O_4$  (208.17): C, 51.93; H, 3.87; N, 13.46. Found: C, 51.88; H, 3.89; N, 13.61 %. IR (KBr): 3290, 3145, 3084, 2960, 1678, 1458, 1315, 1273, 1220, 1124, 985, 920, 769 cm<sup>-1</sup>. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 12.09 (d, 1H, J = 0.6 Hz, OH); 11.88 (s, 1H, NH); 7.59 (s, 1H, H-6); 7.26 (d, 1H, J = 0.9 Hz, H-3); 6.79 (d, 1H, J = 0.7 Hz, CH); 3.81 (s, 3H, OCH<sub>3</sub>).

### 2.3 Methyl 2-{[(benzyloxy)imino]methyl}-4H-furo[3,2-b]pyrrole-5carboxylate (**6**)

The mixture of methyl 2-[(hydroxyimino)methyl]-furo[3,2-*b*]pyrrole-5-carboxylate **5** (3.3 g, 1.6 mmol), benzyl chloride (3.8 g, 30 mmol) and catalytic amount of sodium carbonate in acetone (200 ml) was refluxed for 48 h (Scheme 1). Sodium carbonate

was filtered off and the excess of benzylchloride was distilled under reduced pressure. Ice water (100 ml) was added and the mixture was extracted with diethylether ( $2 \times 50$  ml). The organic layer was dried with sodium sulphate and the solvent was evaporated.

Yield: 20 %; m. p 224-227 °C. Calcd. for  $C_{16}H_{14}N_2O_4$  (298.29): C, 64.42; H, 4.73; N, 9.39. Found: C, 64.01; H, 4.69; N, 9.12 %. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 11.72 (s, 1H, NH); 7.77 (s, 1H, H-6); 7.42 (m, 6H, Ph); 6.77 (s, 1H, CH); 5.01 (s, 2H, CH<sub>2</sub>); 3.88 (s, 3H, OCH<sub>3</sub>).

# 2.4 Synthesis of methyl 2-[(4-oxo-2-thioxo-1,3-thiazolidin-5-ylidene) methyl]-4H-furo[3,2-b]pyrrole-5-carboxylate (7)

The mixture of methyl 2-formyl-4*H*-furo[3,2-b]pyrrole-5-carboxylate **4** (5 g, 26 mmol), rhodanine (7 g, 53 mmol) and sodium acetate (13 g) in glacial acetic acid (45 ml) was refluxed for 30 min. (Scheme 2). The reaction mixture was poured into water (260 ml). The separated precipitate was washed with water (140 ml), ethanol (50 ml), diethylether (20 ml), and the solid product was recrystallized from acetone.





Yield: 70 %; m. p 296-298 °C. Calcd. for  $C_{12}H_8N_2O_4S_2$  (308.33): C, 46.74; H, 2.62; N, 9.09. Found: C, 46.44; H, 2.60; N, 8.88 %. <sup>1</sup>H NMR  $\delta_H$  (DMSO-d<sub>6</sub>): 13.69 (brs, 1H, NH); 12.12 (s, 1H, NH); 7.51 (s, 1H, CH); 7.29 (s, 1H, H-6); 6.87 (s, 1H, H-3); 3.83 (s, 3H, OCH<sub>3</sub>).

## 2.5 Synthesis of 2-[(E)-2-carboxy-2-sulfanylethenyl]-4H-furo[3,2-b] pyrrole-5-carboxylic acid (8)

The mixture of 2-[(4-oxo-2-thioxo-1,3-thiazolidin-5-ylidene)methyl]-furo[3,2b]pyrrole-5-carboxylate **7** (4 g, 1.5 mmol) and aqueous sodium hydroxide (7 g NaOH in 18 ml H<sub>2</sub>O) was refluxed for 30 min. (Scheme 2). After cooling, 20 M hydrochloric acid (24 ml) was added into the reaction mixture. The formed solid product was filtered off and recrystallized from methanol.

Yield: 80 %; m. p >350 °C for  $C_{10}H_7NO_5S$  (253.23). <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 11.74 (s, 1H, NH); 7.56 (s, 1H, H-6); 6.99 (d, 1H, H-3); 6.74 (dd, 1H, J = 2.7 Hz, 1.1 Hz, H-7); 3.64 (brs, 1H, SH).

## 2.6 Synthesis of methyl 2-[(5-oxo-2-phenyl-1,3-oxazol-4(5H)-ylidene) methyl]-4H-furo[3,2-b] pyrrole-5-carboxylate (**9**)

**Classical heating** (A). The mixture of methyl 2-formyl-4*H*-furo[3,2-b]pyrrole-5-carboxylate **4** (0.5 g, 2.6 mmol), hippuric acid (0.46 g, 2.6 mmol) and catalytic amount of fused potassium acetate (0.38 g, 38 mmol) in acetanhydride (12.5 ml) was refluxed for 1 h (Scheme 3). After cooling, the reaction mixture was poured into ice water. The formed solid product was filtered off, washed with water and recrystallized from ethanol.

**Microwave method (B)**. The mixture methyl 2-formyl-furo[3,2-*b*]pyrrole-5-carboxylate **4** (0.5 g, 2.6 mmol), hippuric acid (0.46 g, 2.6 mmol) and catalytic amount of fused potassium acetate (0.38 g, 38 mmol) in acetanhydride (12.5 ml) was irradiated at 90 W for 12 min (Scheme 3). The work-up was the same as above method A.



Yield: 80 % (A), 92 % (B); m. p 324-326 °C. Calcd. for  $C_{18}H_{12}N_2O_5$  (336.30): C, 64.29; H, 3.60; N, 8.33. Found: C, 63.96; H, 3.55; N, 8.21 %. IR (KBr): 3286, 3140, 1786, 1768, 1689, 1631, 1500, 1450, 1396, 1288, 1247, 1219, 1132, 977, 854, 761, 702 cm<sup>-1</sup>. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 11.76 (s, 1H, NH); 7.61 (m, 5H, Ph); 7.18 (s, 1H, H-6); 7.01 (s, 1H, H-3); 6.82 (s, 1H, H-7); 3.93 (s, 3H, OCH<sub>3</sub>).

#### 2.7 Synthesis of compounds (11a-b)

The mixture of methyl 2-[(5-oxo-2-phenyl-1,3-oxazol-5(4*H*)-ylidene)methyl]-furo [3,2-*b*]pyrrole-5-carboxylate **9** (1.4 mmol) and corresponding carbohydrazide (1.4 mmol), acetic acid (10 ml) and catalytic amount of fused potassium acetate (0.4 g) was irradiated at 90 W for 17 min (Scheme 3). After cooling, the solid product was filtered off, washed with water and recrystallized from ethanol.

2.7.1 Methyl 2-{[1-{[(2,3-dimethyl-4H-furo[3,2-b]pyrrole-5-yl)carbonyl]imino}-5-oxo -2-phenyl-4H-imidazol-4-ylidene]methyl}-4H-furo[3,2-b]pyrrole-5-carboxylate (**11a**)

Yield: 65 %; m. p 285-287 °C. Calcd. for  $C_{27}H_{21}N_5O_6$  (511.49): C, 63.40; H, 4.14; N, 13.69. Found: C, 63.19; H, 4.35; N, 12.82 %. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 11.78 (s, 1H, NH); 11.30 (s, 1H, NH); 10.14 (s, 1H, NH); 8.09 (m, 2H, H-2', H-6'); 7.62 (m, 3H, H-3', H-4', H-5'); 7.26 (s, 1H, H-7); 6.87 (s, 1H, H-6''); 6.86 (d, 1H, J = 1.9 Hz, H-6); 6.68 (d, 1H, J = 1.8 Hz, H-3); 3.78 (s, 3H, OCH<sub>3</sub>); 2.28 (s, 3H, CH<sub>3</sub>); 2.04 (s, 3H, CH<sub>3</sub>).

#### 2.7.2 *Methyl* 2-{[1-(phenylcarbonylimino)-5-oxo-2-phenyl-4H-imidazol-4-ylidene] methyl}-4H-furo[3,2-b]pyrrole-5-carboxylate (**11b**)

Yield: 74 %; m. p 310-314 °C. Calcd. for  $C_{25}H_{18}N_4O_5$  (454.43): C, 66.07; H, 3.99; N, 12.33. Found: C, 65.78; H, 3.94; N, 11.78 %. IR (KBr): 3296, 1685, 1629, 1512, 1450, 1286, 1219, 1143, 817, 758, 698 cm<sup>-1</sup>. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): 12.07 (s, 1H, NH); 11.71 (s, 1H, NH); 8.05 (dd, 2H, J = 8.1 Hz. J = 1.5 Hz, H-2',H-6'); 7.91 (dd, 2H, J = 8.2 Hz, J = 1.5 Hz, H-3', H-5'); 7.66 (m, 6H, Ar); 7.53 (s, 1H, H-6); 7.26 (s, 1H, H-7); 6.88 (s, 1H, H-3); 3.85 (s, 3H, OCH<sub>3</sub>).

### 3. Results and discussion

Methyl 2-formyl-4*H*-furo[3,2-*b*]pyrrole-5-carboxylate **7** was routinely prepared by condensation of furan-2-carboxaldehyde with methyl azidoacetate in methanol in the presence of sodium methoxide giving methyl 2-azido-3-(furan-2-yl)propenoate, which underwent the cyclization in boiling toluene to give methyl 4*H*-furo[3,2-*b*]pyrrole-2-carboxylate **4** (KRUTOŠÍKOVÁ *et al.*, 1994).

Compounds **3a-d** were formed by reaction with carbohydrazide **2** using corresponding aldehydes in 35-53% yield. The structures of compounds **3a-d** were corroborated by the presence two singlets of NH protons at 11.84 and 11.53 ppm. IR spectra of **3a-d** exhibit absorption bands of carbonyl group at 1631-1674 cm<sup>-1</sup> and absorption bands of NH group at 3258-3453 cm<sup>-1</sup>.

One of the most interesting results in the study is synthesis of methyl 2-[(hydroxyimino)methyl]-4H-furo[3,2-b]pyrrole-5-carboxylate 5, which was prepared by reaction of methyl 2-formyl-4H-furo[3,2-b]pyrrole-5-carboxylate 4 with hydroxylamine hydrochloride in sodium hydroxide either by classical heating for 24 h in 50% yield or in microwave oven for 13 min. in 70% yield. The only one preparation of 4H-furo[3,2-b]pyrrole-2-aldoxime 5 has been described by GORUGANTULA et al., 2010. In their study there was described the synthesis of 4H-furo[3,2-b]pyrrole-2-4-nitro-5-(2-phenylethenyl)furan-2-aldoxime palladium-catalysed aldoxime via reductive cyclization. Reaction of aldehyde 4 with hydroxylamine has been described as useful method of nitrile synthesis (KRUTOŠÍKOVÁ et al., 1993), but in that case aldoxime 5 has not been isolated. <sup>1</sup>H NMR spectrum displayed doublet signal of =N-OH proton at 12.09 ppm. Doublet signal of CH proton occur at 6.79 ppm. IR spectrum of 5 shows the absorption bands of carbonyl group at 1678  $\text{cm}^{-1}$  and of NH group at 3290  $\text{cm}^{-1}$ .

Reaction of **5** with benzyl chloride in acetone led to methyl 2- $\{[(benzyloxy)imino]methyl\}$ -4H-furo[3,2-b]pyrrole-5-carboxylate **6** in yield 20 % after 48 h of refluxed. <sup>1</sup>H NMR spectra display singlet signal of NH proton at 11.72 ppm, multiplet signal of Ph group at 7.42 ppm, singlet signal of CH<sub>2</sub> bond at 5.01 ppm.

The reaction of methyl 2-formyl-4H-furo[3,2-b]-pyrrole-5-carboxylate **4** with rhodanine afforded compound **7**, which subsequently underwent hydrolysis to provide derivative **8** in 80 % yield. Compound **8** display singlet signal of NH group at 1.74 ppm and broad signal of SH group at 3.64 ppm. Signal COOH group is not occurring in the spectrum as a result of the rapid exchange hydrogen for deuterium.

The reactions of azlactone **9** with carbohydrazides **10** were carried in acetic acid under microwave irradiation in the presence of potassium acetate to give compounds **11a-b** in yields 65-74 %. Compounds **11a** and **11b** display two or three singlets at 12.01–10.14 ppm in their <sup>1</sup>H NMR spectra due to NH group. Three singlets of NH groups of compound **11a** appear at 11.78, 11.30 and 10.14 ppm. The characteristic bands observed at 1680 cm<sup>-1</sup> and 3296 cm<sup>-1</sup> in IR spectra correspond to the C=O and NH group.

The structures of the prepared compounds were confirmed by <sup>1</sup>H NMR and IR spectra.

### 4. Conclusions

Derivatives **3** were obtained by the reaction of carbohydrazide **2** with heterocyclic aldehydes. Methyl 2-formyl-4*H*-furo[3,2-b]pyrrole-5-carboxylate **4** served as the substrate for synthesis of furo[3,2-b]pyrrole-2-aldoxime **5** as well as derivatives **7**, **9**. The prepared furo[3,2-b]pyrrole-2-aldoxime **5** was used as starting compound for the condensation reaction with benzyl chloride to give a compound **6**. The hydrolysis of compound **7** gave 2-[2-carboxy-2-sulfanylethenyl]-4*H*-furo[3,2-b]pyrrole-5-carboxylic acid **8**. Pyrazole derivatives **11** were prepared by the reaction of azlactone **9** with derivatives **10**.

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