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# CYCLIZATION PRODUCTS OF N-FLUOROPHENYL-β-ALANINES AND THEIR PROPERTIES

**R. Vaickelioniene and V. Mickevicius**

*Interaction of 2- and 4-fluorophenylamines with acrylic and itaconic acids leads to the synthesis of the corresponding N-substituted β-alanines, the cyclization of which leads to derivatives of dihydropyrimidinone, 4-carboxy-2-pyrrolidinone, and tetrahydropyridone. Compounds having benzimidazole, pyrazole, and hydrazine fragments in the molecule have been obtained from 4-carboxy-1-(4-fluorophenyl)-2-pyrrolidinone.*

**Keywords:** dihydropyrimidinones, N-substituted β-alanines, 4-carboxy-2-pyrrolidinones, tetrahydropyridone, condensation.

While continuing investigations on the chemistry of N-substituted amino acids [1, 2] we have synthesized in the present study fluorine-containing N-aryl-substituted β-amino acids, and have carried out heterocyclization of them into five- and six-membered rings. It is known that the presence of fluorine in a molecule often has a favorable influence on its biological activity.

N-(2- and 4-fluorophenyl)-β-alanines **2a,b** were obtained by the reaction of the appropriate aromatic amines **1a,b** with acrylic acid in 20% acetic acid. On boiling β-alanines **2a,b** with urea in glacial acetic acid N-carbamoyl-N-(2-fluorophenyl)- and N-carbamoyl-N-(4-fluorophenyl)-β-alanines **4a,b** were synthesized which, without isolation from the reaction mixture, were cyclized by the action of conc. HCl into the corresponding 2,4-(1H,3H)-dihydropyrimidinediones **3a,b**.

1-(2- and 4-fluorophenyl)-4-(1H,3H)-pyrimidinone-2-thiones **5a,b** were synthesized under analogous conditions as for compounds **3a,b** only using potassium thiocyanate in place of urea. For purification from N-substituted ureas compounds **3** and **5** were decyclized with 5% sodium hydroxide solution to the corresponding salts of N-aryl-N-carbamoyl(or thiocarbamoyl)-β-alanines **4, 6**, insoluble contaminants were filtered off, and salts **4** and **6** in the filtrate were cyclized once again with hydrochloric acid into compounds **3, 5** (Scheme 1).

On condensing β-alanine **2b** with the ethyl ester of acetoacetic acid 3-ethoxycarbonyl-1-(4-fluorophenyl)-2-methyl-1,4,5,6-tetrahydropyridone **7b** was formed, in the $^1$H NMR spectrum of which (Table 1) the most characteristic were the singlet signals of the methyl group protons at 1.84 and a triplet and quadruplet for the protons of the ethyl group at 1.20 and 4.09 ppm respectively.

On investigating the reactions of aromatic amines **1a,b** with itaconic acid the products of intramolecular cyclization of the amino acids, *viz*. 1-aryl-substituted 4-carboxy-2-pyrrolidinones **8a,b,** were isolated from the reaction mixture. We were unable to isolate the free N-substituted β,γ-amino acids. On condensing *o*-phenylenediamine and 1-aryl-substituted 4-carboxy-2-pyrrolidinones **8a,b** in hydrochloric acid (Phillips

Kaunas University of Technology, Kaunas LT-50254, Lithuania; e-mail: Vytautas.Mickevicius@ktu.lt.
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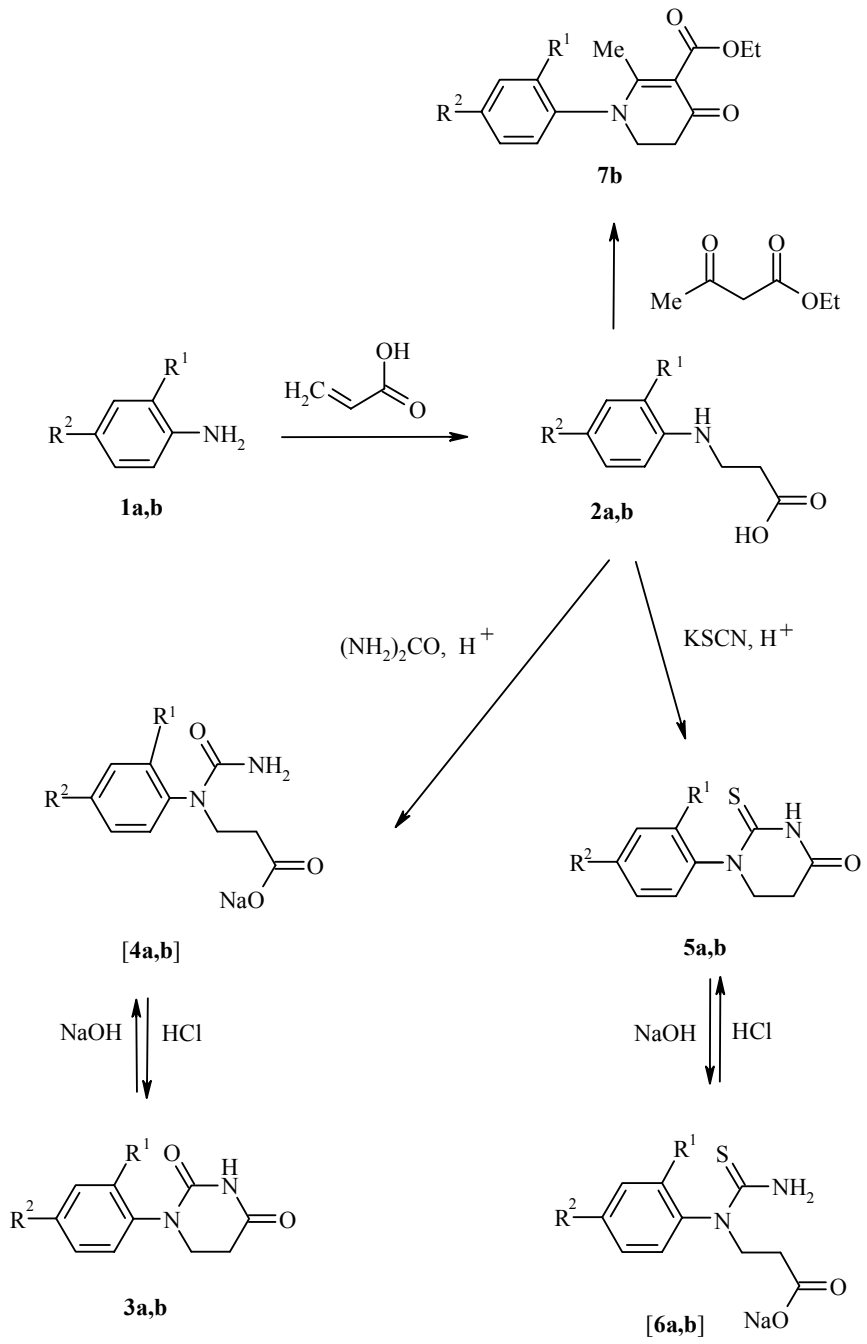
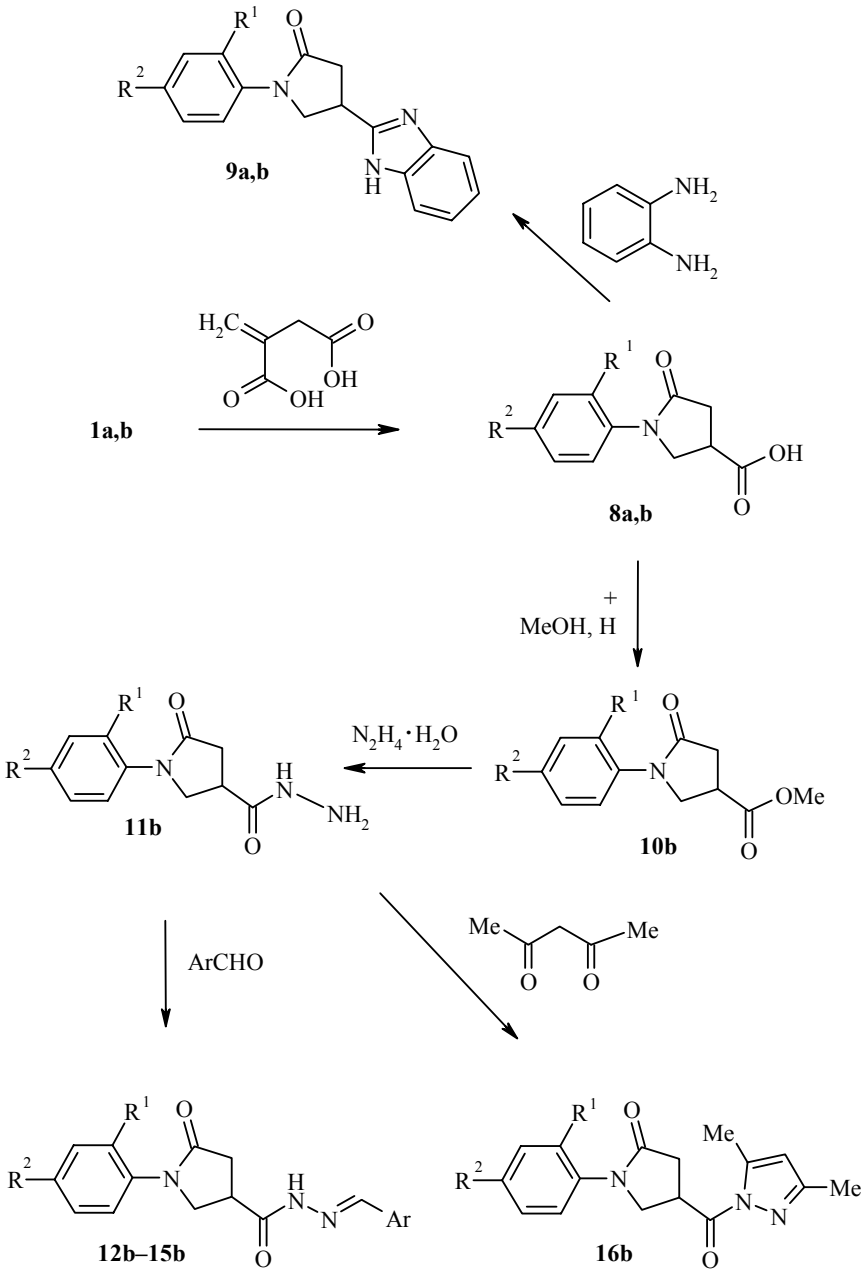
Scheme 1




**1a–6a** $R^1$ = 2-F, $R^2$ = H; **1b–7b** $R^1$ = H

method) compounds **9a,b** having a benzimidazole ring were synthesized. The action of methanol on compound **8b** in the presence of sulfuric acid gave the corresponding methyl ester, 1-(4-fluorophenyl)-4-methoxycarbonyl-2-pyrrolidinone (**10b**), which is then converted by reaction with hydrazine into hydrazide **11b** (Scheme 2).

Scheme 2




**8a, 9a** $R^1$ = 2-F, $R^2$ = H, **8b–16b** $R^1$ = H, $R^2$ = 4-F, **12b** Ar = 4-$MeOC_6H_4$, **13b** Ar = 4-$FC_6H_4$, **14b** Ar = 4-$BrC_6H_4$, **15b** Ar = 4-$Me_2NC_6H_4$

The condensation of hydrazide **11b** was then carried out in ethanol with the aromatic aldehydes 4-methoxybenzaldehyde, 4-fluorobenzaldehyde, 4-bromobenzaldehyde, and 4-dimethylaminobenzaldehyde. 1-(4-Fluorophenyl)-4-(4-methoxybenzylidenehydrazinocarbonyl)-, 4-(4-fluorobenzylidenehydrazinocarbonyl)-1-(4-fluorophenyl)-, 4-(4-bromobenzylidenehydrazinocarbonyl)-1-(4-fluorophenyl)-, and 4-(4-dimethylaminobenzylidenehydrazinocarbonyl)-1-(4-fluorophenyl)-2-pyrrolidinones **12b-15b** were isolated. It is evident from the $^1$H NMR spectra of these compounds that in solution in DMSO-$d_6$ they exist as a mixture of *Z/E*-isomers, the

TABLE 1. Characteristics and Data of Elemental Analysis of the Synthesized Compounds

| Compound | Empirical formula | Found, % / Calculated, % | | | mp, °C (solvent) | $^1$H NMR spectrum, δ, ppm (*J*, Hz)* | Yield, % |
|---|---|---|---|---|---|---|---|
| | | C | H | N | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| **2a** | $C_9H_{10}FNO_2$ | 59.42 / 59.01 | 5.39 / 5.50 | 7.42 / 7.65 | 102-103*[2] (acetone–hexane) | 2.67 (2H, t, *J* = 7.4, $CH_2CO$); 3.49 (2H, t, *J* = 7.3, $CH_2$); 6.6-7.0 (4H, m, arom H) | 55 |
| **2b** | $C_9H_{10}FNO_2$ | 59.34 / 59.01 | 5.33 / 5.50 | 7.78 / 7.65 | 86-87*[2] (acetone–hexane) | 2.65 (2H, t, *J* = 7.2, $CH_2CO$); 3.42 (2H, t, *J* = 7.2, $CH_2$); 6.02 (2H, s, $^+NH_2$); 6.5-7.0 (4H, m, arom H) | 79 |
| **3a** | $C_{10}H_9FN_2O_2$ | 57.51 / 57.69 | 4.57 / 4.36 | 13.68 / 13.46 | 185-186 (ethanol) | 2.82 (2H, t, *J* = 7.3, 5-$CH_2$); 3.85 (2H, t, *J* = 7.3, 6-$CH_2$); 7.1-7.5 (4H, m, arom H); 9.26 (1H, s, NH) | 74 |
| **3b** | $C_{10}H_9FN_2O_2$ | 57.58 / 57.69 | 4.19 / 4.36 | 13.66 / 13.46 | 238-239 (ethanol) | 2.65 (2H, t, *J* = 7.8, 5-$CH_2$); 3.72 (2H, t, *J* = 7.8, 6-$CH_2$); 7.1-7.5 (4H, m, arom H); 10.34 (1H, s, NH) | 77 |
| **5a** | $C_{10}H_9FN_2OS$ | 53.81 / 53.56 | 4.29 / 4.05 | 12.24 / 12.49 | 208-209 (ethanol) | 2.78 (2H, t, *J* = 7.7, 5-$CH_2$); 3.87 (2H, t, *J* = 7.7, 6-$CH_2$); 7.2-7.6 (4H, m, arom H); 11.38 (1H, s, NH) | 76 |
| **5b** | $C_{10}H_9FN_2OS$ | 53.76 / 53.56 | 4.21 / 4.05 | 12.28 / 12.49 | 291-292 (ethanol) | 2.82 (2H, t, *J* = 7.6, 5-$CH_2$); 3.90 (2H, t, *J* = 7.6, 6-$CH_2$); 7.2-7.5 (4H, m, arom H); 11.24 (1H, s, NH) | 81 |
| **7b** | $C_{15}H_{16}FNO_3$ | 64.56 / 64.97 | 5.43 / 5.82 | 5.38 / 5.05 | 225-226 (hexane) | 1.20 (3H, t, *J* = 7.9, $CH_2\underline{CH_3}$); 1.84 (3H, s, $CH_3$); 2.48 (2H, t, *J* = 8.1, $CH_2$); 3.83 (2H, t, *J* = 8.2, 6-$CH_2$); 4.09 (2H, q, *J* = 7.9, $\underline{CH_2}CH_3$); 7.2-7.6 (4H, m, arom H) | 35 |
| **8a** | $C_{11}H_{10}FNO_3$ | 59.31 / 59.19 | 4.74 / 4.52 | 6.51 / 6.28 | 161-162 (ethanol) | 2.78 (2H, d, *J* = 7.8, 3-$CH_2$); 3.4-3.7 (1H, m, CH); 4.05 (2H, d, *J* = 7.8, 5-$CH_2$); 7.2-7.5 (4H, m, arom H); 8.53 (1H, s, OH) | 59 |

TABLE 1 (continued)

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|
| **8b** | $C_{11}H_{10}FNO_3$ | 59.34<br>59.19 | 4.69<br>4.52 | 6.48<br>6.28 | 164-165<br>(ethanol) | 2.85 (2H, d, $J$ = 7.9, 3-$CH_2$); 3.3-3.6 (1H, m, CH); 4.11 (2H, d, $J$ = 7.8, 5-$CH_2$);<br>7.0-7.8 (4H, m, arom H); 8.54 (1H, s, OH) | 72 |
| **9a** | $C_{17}H_{14}FN_3O$ | 69.35<br>69.14 | 4.49<br>4.78 | 14.56<br>14.23 | 244-245<br>(1,4-dioxane) | 2.8-3.2 (2H, m, 3-$CH_2$); 3.9-4.1 (1H, m, CH); 4.2-4.4 (2H, m, 5-$CH_2$);<br>7.3-7.9 (8H, m, arom H); 9.5-10.0 (1H, br. s, NH) | 21 |
| **9b** | $C_{17}H_{14}FN_3O$ | 69.41<br>69.14 | 4.52<br>4.78 | 14.52<br>14.23 | 237-238<br>(2-propanol) | 2.6-3.1 (2H, m, 3-$CH_2$); 3.9-4.1 (1H, m, 4-CH); 4.2-4.4 (2H, m, 5-$CH_2$);<br>7.1-7.8 (8H, m, arom H); 9.5-9.9 (1H, br. s, NH) | 40 |
| **10b** | $C_{12}H_{12}FNO_3$ | 60.51<br>60.76 | 5.34<br>5.10 | 5.71<br>5.90 | 101-102<br>(ethanol) | 2.6-2.9 (2H, m, 3-$CH_2$); 3.3-3.6 (1H, m, CH); 3.71 (3H, s, $CH_3$);<br>4.0-4.3 (2H, m, 5-$CH_2$); 6.9-7.8 (4H, m, arom H) | 64 |
| **11b** | $C_{11}H_{12}FN_3O_2$ | 55.53<br>55.69 | 5.25<br>5.10 | 17.46<br>17.71 | 198-199<br>(ethanol) | 2.5-2.8 (2H, m, 3-$CH_2$); 3.0-3.3 (1H, m, CH); 3.7-4.1 (2H, m, 5-$CH_2$); 4.27 (2H, s, $NH_2$);<br>7.1-7.7 (4H, m, arom H); 9.24 (1H, s, NH) | 80 |
| **12b** | $C_{19}H_{18}FN_3O_3$ | 64.42<br>64.22 | 5.32<br>5.11 | 11.64<br>11.82 | 211-212<br>(1,4-dioxane) | Mixture of *Z*/*E*-isomers (55:45); 2.7-3.1 (2H, m, 3-$CH_2$); 3.3-3.6 (1H, m, CH);<br>3.8 (3H, s, $CH_3$); 3.8-4.3 (2H, m, 5-$CH_2$); 6.9-8.5 (9H, m, arom H + N=CH);<br>11.42 and 11.48 (1H, 2s, NH) | 96 |
| **13b** | $C_{18}H_{15}F_2N_3O_2$ | 62.69<br>62.97 | 4.65<br>4.40 | 12.46<br>12.24 | 224-226<br>(1,4-dioxane) | Mixture of *Z*/*E*-isomers (75:25); 2.6-2.9 (2H, m, 3-$CH_2$); 3.2-3.6 (1H, m, CH);<br>4.1-4.4 (2H, m, 5-$CH_2$); 7.1-8.5 (9H, m, arom H + N=CH); 11.58 and 11.67 (1H, 2s, NH) | 92 |
| **14b** | $C_{18}H_{15}BrFN_3O_2$ | 53.61<br>53.48 | 3.92<br>3.74 | 4.89<br>4.70 | 258-259<br>(1,4-dioxane) | Mixture of *Z*/*E*-isomers (70:30); 2.4-2.7 (2H, m, 3-$CH_2$); 2.6-2.9 (1H, m, CH);<br>3.8-4.3 (2H, m, 5-$CH_2$); 7.1-7.8 (9H, m, arom H + N=CH); 11.59 and 11.65 (1H, 2s, NH) | 92 |
| **15b** | $C_{20}H_{21}FN_4O_2$ | 65.32<br>65.20 | 5.60<br>5.75 | 15.42<br>15.21 | 233-235<br>(1,4-dioxane) | Mixture of *Z*/*E*-isomers (60:40); 2.6-2.9 (2H, m, 3-$CH_2$); 2.95 (6H, s, –$N(CH_3)_2$);<br>3.2-3.5 (1H, m, CH); 3.8-4.2 (2H, m, 5-$CH_2$); 6.7-7.8 (9H, m, arom H + N=CH);<br>11.24 and 11.31 (1H, 2s, NH) | 94 |
| **16b** | $C_{16}H_{16}FN_3O_2$ | 63.61<br>63.78 | 5.68<br>5.35 | 14.16<br>13.95 | 124-125<br>(1,4-dioxane) | 2.24 (3H, s, $CH_3$); 2.55 (3H, s, $CH_3$); 2.9-3.1 (2H, m, 3-$CH_2$); 4.0-4.8 (3H, m, $CH_2CH$);<br>6.0 (1H, s, C=CH); 6.9-7.7 (8H, m, arom H) | 86 |

* $^1$H NMR spectra were taken in $(CD_3)_2CO$ (compounds **2a**, **3a, 8a,b**), $CDCl_3$ (compounds **2b**, **16b**), and DMSO-$d_6$ (compounds **3b**, **5a**, **6b**, **7b**, **9a**, **9b-15b**).

*[2] According to [4] the melting points of compounds **2a** and **2b** are equal to 100.5-101.5 and 77.5-78.5°C respectively.

TABLE 2. Data of IR and Mass Spectra of the Synthesized Compounds

| Compound | IR spectrum, ν, $cm^{-1}$ | Mass spectrum, *m/z* $[M]^{+\cdot}$ |
|---|---|---|
| **2a** | 3388, 1723, 1685, 1624, 1453, 1288, 1203, 736 | — |
| **2b** | 3246, 1713, 1509, 1342, 1298, 1211, 1159, 1057, 834, 746 | — |
| **3a** | 3195, 1726, 1678, 1501, 1479, 1447, 1366, 1269, 1232, 827, 763 | 209.3 |
| **3b** | 3211, 1700, 1507, 1482, 1375, 1264, 1213, 846 | 209.3 |
| **5a** | 3226, 1711, 1506, 1432, 1364, 1217, 757 | 225.3 |
| **5b** | 3186, 1701, 1502, 1368, 1280, 1213, 1151, 844, 822 | 225.3 |
| **7b** | 1696, 1641, 1550, 1510, 1198, 1180, 1079, 856 | 278.3 |
| **8a** | 3074, 2905, 1724, 1653, 1605, 1508, 1429, 1289, 1273, 1223, 819, 758, 672 | 224.3 |
| **8b** | 3091, 2927, 1741, 1634, 1510, 1410, 1322, 1220, 1197, 838 | 224.3 |
| **9a** | 3434, 2706, 1701, 1506, 1461, 1416, 1227, 820, 766, 756, 749 | 296.3 |
| **9b** | 3052, 2749, 1691, 1513, 1433, 1337, 1237, 833, 747 | 296.3 |
| **10b** | 1737, 1700, 1511, 1396, 1281, 1226, 1132, 840, 817 | 238.3 |
| **11b** | 3308, 3281, 1680, 1636, 1510, 1402, 1319, 1240, 1232, 1126, 831 | 238.3 |
| **12b** | 3246, 1669, 1654, 1609, 1512, 1254, 1240, 1032, 829 | 356.3 |
| **13b** | 3122, 1680, 1665, 1512, 1404, 1323, 1231, 827 | 344.34 |
| **14b** | 3130, 1666, 1662, 1511, 1405, 1396, 1235, 830, 815 | 406.1 |
| **15b** | 3222, 2891, 1674, 1612, 1600, 1507, 1270, 1224, 1180, 844 | 369.3 |
| **16b** | 1719, 1585, 1511, 1485, 1392, 1377, 1328, 1227, 964, 829, 764 | 302.3 |

proton signals of which have different intensities. As a result of stronger shielding the signals of the protons of the *Z*-isomers are observed at higher field [3]. On the basis of the intensities of the signals of the NH group proton it is concluded that in DMSO-$d_6$ solution the *Z*-isomer always predominates.

The interaction of 1-(4-fluorophenyl)-4-hydrazinocarbonyl-2-pyrrolidinone (**11b**) with acetylacetone led to the synthesis of 4-(3,5-dimethyl-1-pyrazolylcarbonyl)-1-(4-fluorophenyl)-2-pyrrolidinone (**16b**). In the $^1$H NMR spectrum of compound **16b** (Table 1), apart from the signals of the protons of the aromatic and pyrrolidone rings, a characteristic signal is observed for the CH=C fragment of the pyrazole ring at 6.0, and also signals of the two methyl groups at 2.24 and 2.55 ppm.

## EXPERIMENTAL

The $^1$H NMR spectra were obtained on a Jeol FX 100 (100 MHz) spectrometer, internal standard was TMS. The IR spectra were recorded on a Perkin-Elmer FT-IR instrument in KBr disks, and the mass spectra on a Waters ZQ 2000 spectrometer, chemical ionization, ionizing voltage 15 eV.

A check on the progress of reactions and the purity of the compounds obtained was effected by TLC on Silufol UV-254 plates, visualization was in UV light or with iodine vapor.

**N-(2-Fluorophenyl)-β-alanine (2a).** A mixture of 2-fluoroaniline **1a** (11.1 g, 0.1 mol), acrylic acid (7.2 g, 0.1 mol), and 20% acetic acid (60 ml) was boiled for 8 h, cooled, the crystals of compound **2a** which had separated were filtered off, and crystallized from acetone–hexane, 1:1.

**N-(4-Fluorophenyl)-β-alanine (2b).** A mixture of 4-fluoroaniline **1b** (11.1 g, 0.1 mol), acrylic acid (7.2 g, 0.1 mol), and 20% acetic acid (60 ml) was boiled for 8 h, cooled, 25% NaOH (50 ml) was added, and the unreacted 4-fluoroaniline was extracted with ether (2 × 100 ml). The extract was acidified with 10% hydrochloric acid to pH 7, the crystals of **2b**, which separated on standing, were filtered off, washed with water, dried, and crystallized from acetone–hexane, 1:1.

**Preparation of 1-(2-Fluorophenyl)- and 1-(4-Fluorophenyl)dihydro-2,4-(1H,3H)-pyrimidinediones 3a,b (General Procedure).** A mixture of the appropriate β-alanine **2** (3.66 g, 0.02 mol), urea (2.4 g, 0.04 mol), and acetic acid (15 ml) was boiled for 12 h, acidified with conc. HCl to pH 1, boiled for 20 min more, and diluted with water (30 ml). The crystals, which separated on standing at 4°C were filtered off, dissolved with heating in 5% NaOH solution (15 ml), cooled, the solution filtered, the filtrate was heated to boiling, carefully acidified to pH 1 with conc. HCl, and boiled for 5 min. The crystals of dihydropyrimidinediones **3a,b**, which separated on cooling, were filtered off, and washed with water.

**1-(2-Fluorophenyl)- and 1-(4-Fluorophenyl)dihydro-(1H,3H)-pyrimidin-4-one-2-thiones 5a,b** were obtained analogously to compounds **3a,b**, using potassium thiocyanate instead of urea.

**3-Ethoxycarbonyl-1-(4-fluorophenyl)-2-methyl-1,4,5,6-tetrahydro-(1H)-pyrid-4-one (7b).** A mixture of β-alanine **2b** (3.66 g, 0.02 mol), ethyl acetoacetate (7.81 g, 0.06 mol), and conc. HCl (0.5 ml) was boiled for 8 h, cooled to room temperature, 5% $Na_2CO_3$ solution (200 ml) was added, and the reaction mixture brought to boiling. The crystals of **7b**, which separated on cooling, were filtered off, washed with water, with ethanol, and dried.

**Preparation of 4-Carboxy-1-(2-fluorophenyl)- and 4-Carboxy-1-(4-fluorophenyl)-2-pyrrolidinones 8a,b (General Procedure).** A mixture of the appropriate aromatic amine **1a,b** (11.0 g, 0.1 mol), itaconic acid (15.6 g, 0.12 mol), and water (100 ml) was boiled for 24 h, 4 M HCl solution (10 ml) was added, the mixture cooled, the crystals of compound **8**, which had separated, were filtered off, and washed with water.

**Preparation of 4-(1H-Benzimidazol-2-yl)-1-(2-fluorophenyl)- and 4-(1H-Benzimidazol-2-yl)-1-(4-fluorophenyl)-2-pyrrolidinones 9a,b (General Procedure).** 2-Pyrrolidinone **8a** or **8b** (11.16 g, 0.05 mol), and *o*-phenylenediamine (6.48 g, 0.06 mol) in 4 M HCl solution (100 ml) were boiled for 24 h. The mixture was cooled, the separated solid was filtered off, 5% $Na_2CO_3$ solution (100 ml) was added, and the mixture brought to boiling. The solid compound **9a** or **9b** was filtered off, washed with water, and crystallized from an appropriate solvent.

**1-(4-Fluorophenyl)-4-methoxycarbonyl-2-pyrrolidinone (10b).** 2-Pyrrolidinone **8b** (22.3 g, 0.1 mol) in methanol (85 ml) with conc. $H_2SO_4$ (5 ml) was boiled for 6 h, then the methanol was distilled in vacuum. The residue was treated with 5% $Na_2CO_3$ solution (200 ml) and the contents brought to boiling. The crystals of compound **10b**, which separated on cooling, were filtered off, washed with water, and dried.

**1-(4-Fluorophenyl)-4-hydrazinocarbonyl-2-pyrrolidinone (11b).** A mixture of 2-pyrrolidinone **10b** (11.86 g, 0.05 mol) and 99% hydrazine hydrate (7.5 g, 0.15 mol) in 2-propanol (80 ml) was boiled for 30 min, cooled to 4°C, the crystals of compound **11b**, which had separated, were filtered off, washed with 2-propanol, and with ether.

**Preparation of 4-Arylidenehydrazinocarbonyl-1-(4-fluorophenyl)-2-pyrrolidinones 12b-15b (General Procedure).** A mixture of hydrazide **11b** (1.19 g, 0.005 mol) and the appropriate benzaldehyde, 4-methoxybenzaldehyde (1.02 g, 0.0075 mol), 4-fluorobenzaldehyde (0.93 g, 0.0075 mol), 4-bromobenzaldehyde (1.2 g, 0.0065 mol), or N,N-dimethylaminobenzaldehyde (0.9 g, 0.0065 mol), in ethanol (60 ml) was boiled for 1 h. The crystals of hydrazone, which separated on cooling, were filtered off, washed with ethanol, and with ether.

**4-(3,5-Dimethyl-1-pyrazolyl)carbonyl-1-(4-fluorophenyl)-2-pyrrolidinone (16b).** A mixture of hydrazide **11b** (2.38 g, 0.01 mol), acetylacetone (2 g, 0.02 mol), and conc. HCl (1 ml) in 2-propanol (30 ml) was boiled for 2 h. The solvent was distilled in vacuum, water (100 ml) was added to the residue, and the mixture brought to boiling. The crystals of compound **16b**, which separated on cooling, were filtered off, and washed with water.

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