Chemistry of Heterocyclic Compounds, Vol. 37, No. 4, 2001

NEW HETEROAROMATIC DERIVATIVES OF 6-AMINO-4-METHYLANGELICIN

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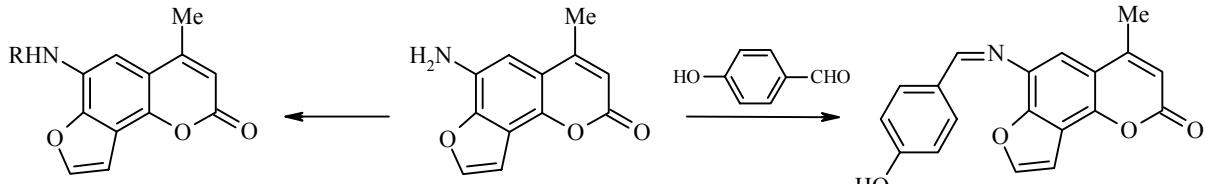
New heteroaromatic derivatives of 6-amino-4-methylangelicin, the pyrrolofurocoumarins and 6-pyrazolyl-4-methylangelicin, have been obtained.

Keywords: 6-azido-4-methylangelicin, aliphatic hydrazones, 6-amino-4-methylangelicin, 4-methyl-6-(3,5-dimethyl-1-pyrazolyl)angelicin, pyrrolofurocoumarins, 4-methyl-6-(*p*-toluenesulfonamido)-angelicin, Fischer method.

Amongst the furocoumarins the amino derivatives of angelicin are the most promising derivatives for studying biological activity. The high water-solubility provides active transport of a furocoumarin salt in biological substrates and a correspondingly more marked pharmacological effect. The number of studies on the synthesis of aminoangelicins is however small. 4-Aminoangelicins are obtained by condensing 5-aminomethyleno-6,7-dihydrobenzofuran-4-one [1] with dichloroacetic acid chloride in the presence of a tertiary amine into 3-chloro-3,4,5,6-tetrahydroangelicin with subsequent dehydrochlorination and aromatization. Aminomethyl derivatives of angelicins are obtained by substituting the halogen of a halomethyl group of the appropriate angelicin [2].

Investigation of the biological activity of a series of aminoangelicins has confirmed their promise as photochemotherapeutic agents [2, 4, 7]. Aminoangelicins studied in most detail contain an amino group on the lactone or furan ring.

We have reported previously the synthesis of 6-amino-4-methylangelicin in which the amino group is found on the benzene ring [8-13]. In the present work reactions of 6-amino-4-methylangelicin (**1**) are considered which enable new heteroaromatic derivatives of angelicin to be synthesized. 6-Aminoangelicin (**1**) possesses properties typical of aromatic amines. It is readily acylated at the amino group leading to 6-acetamido-, 6-formamido-, and 6-(*p*-toluenesulfonamido)-4-methylangelicins (compounds **2a,b** and **3** respectively), and forms Schiff's bases with aldehydes. For example 6-(*p*-hydroxyphenylaldimino)-4-methylangelicin (**4**) was obtained in good yield.



2a R = COMe
2b R = COH
3 R = Ts

1

4

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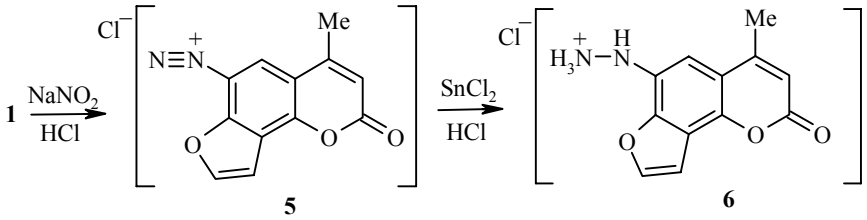
0009-3122/01/3704-0453$25.00

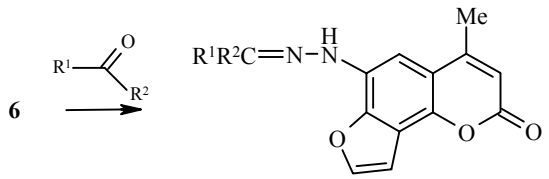
The amino group in 6-aminoangelicin **1** is subject to diazotization. The diazonium salt **5** obtained in this way is reduced with stannous chloride in hydrochloric acid at low temperature (-15°C) to the corresponding hydrazine **6**.



5 **6**

At higher temperatures (0°C) the reduction leads to obtaining of the initial amine **1**. Possibly the reaction passes through the intermediate formation of the hydrazine salt **6**, since the hydrochloride of hydrazine **6** obtained previously is reduced smoothly under the same conditions to amine **1**. The diazo group in compound **5** is readily substituted by other functional groups (for example azide). On reaction with sodium azide 6-azido-4-methylangelicin (**7**) is obtained.

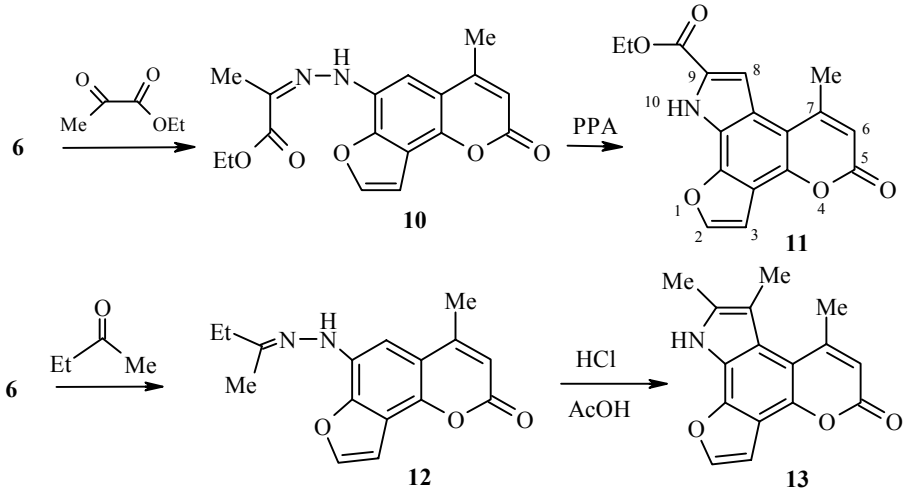
Reaction of hydrazine **6** with certain substituted benzaldehydes and ketones gives the corresponding hydrazones **8,9**.



8, 9

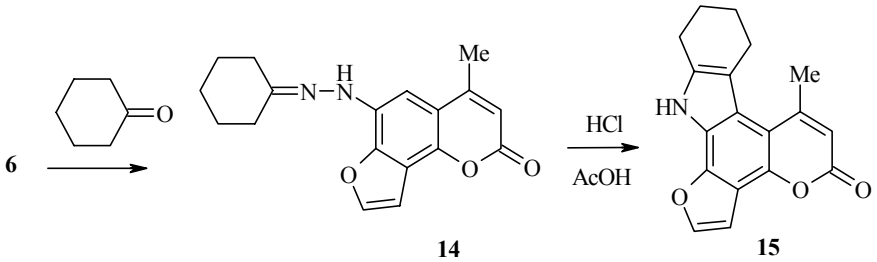
8 R^1 = 4-O_2N–C_6H_4, R^2 = H; **9** R^1 = 4-HO–C_6H_4, R^2 = H

Aliphatic hydrazones from compound **6**, in spite of their instability, are capable of cyclization by the Fischer method to the previously unknown pyrrolofurocoumarins. Due to the presence of donor groups in the benzene ring of angelicin position 5 is sufficiently reactive. Carbonyl components are most frequently used for Fischer cyclization [15], and the cyclizing reagents used, according to [14], are mixtures of sulfuric and acetic acids, hydrochloric and acetic acids, thionyl chloride and ethanol, and also polyphosphoric acid. When cyclizing hydrazone **10** the best yield was achieved using polyphosphoric acid, but for hydrazones **12** and **14** a mixture of hydrochloric and acetic acids was best.



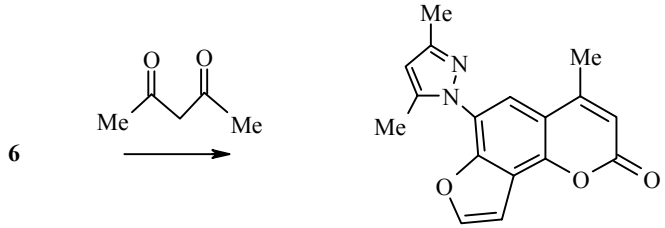
10 **11**

12 **13**



14 15

We also established that hydrazine **6** condensed with acetylacetone forming the corresponding 6-(3,5-dimethyl-1-pyrazolyl)-4-methylangelicin (**16**) in good yield.



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Probably the reaction occurs with the intermediate formation of the corresponding hydrazone and its subsequent cyclization. To carry out such a type of cyclization using acetoacetic ester was unsuccessful. In this case the reaction stopped at the stage of forming the corresponding hydrazone.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker WP 200 instrument with DMSO-d_6 or $CDCl_3$ as solvent and TMS as internal standard. Mass spectra were recorded on a Finnigan MAT SSQ 710 instrument at an ionizing voltage of 70 eV.

6-Acetamido-4-methylangelicin (2a). A mixture of compound **1** (0.1 g, 0.4 mmol) and acetic anhydride (5 ml) was heated for 10 min then cooled to precipitate a solid. The product obtained was recrystallized from acetic acid. Yield 0.1 g (90%); mp 279-281°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm, *J* (Hz): 2.16 (3H, s, NAc); 2.45 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.38 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.27 (1H, d, 9-H, $J_{9,8}$ = 2.1); 8.09 (1H, s, 5-H); 8.17 (1H, d, 8-H, $J_{8,9}$ = 2.1); 10.14 (1H, s, NH). Mass spectrum: 257 (47), 215 (-CH_2C=O, 100), 187 (-CH_2C=O, -CO, 70), 159 (-CH_2C=O, -2CO, 11). Found, %: C 65.20; H 4.55; N 5.39. $C_{14}H_{11}NO_4$. Calculated, %: C 65.37; H 4.31; N 5.44.

6-Formamido-4-methylangelicin (2b). Compound **1** (0.5 g, 2.3 mmol) was dissolved with heating in formic acid (1 ml). The precipitate obtained was filtered off, and recrystallized from DMF. Yield 0.3 g (65%); mp 238-240°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm, *J* (Hz): 2.50 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.32 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.23 (1H, d, 9-H, $J_{9,8}$ = 2.1); 8.11 (1H, s, 5-H); 8.35 (1H, s, CH); 8.41 (1H, d, 8-H, $J_{8,9}$ = 2.1); 10.51 (1H, s, NH). Mass spectrum: 243 (100), 215 (-CO, 21), 187 (-2CO,12), 159 (-3CO). Found, %: C 64.60; H 3.55; N 5.69. $C_{13}H_9NO_4$. Calculated, %: C 64.20; H 3.73; N 5.76.

4-Methyl-6-(4-toluenesulfonamido)angelicin (3). A mixture of compound **1** (0.2 g, 0.93 mmol), tosyl chloride (0.2 g, 1 mmol), pyridine (0.5 ml), and chloroform (3 ml) was heated under reflux for 1 h. The chloroform was distilled off and the residual reaction mixture was poured into water. The solid obtained was filtered off, dried, and recrystallized from chloroform. Yield 0.27 g (70%); mp 215-218°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm, *J* (Hz): 2.33 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.36 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.21 (1H, d, 9-H,

$J_{9,8}$ = 2.1); 7.25 (1H, s, 5-H); 7.32 (2H, d, 3'-H, 5'-H, $J_{3',2'} = J_{5',6'}$ = 7.8); 7.63 (2H, d, 2'-H, 6'-H, $J_{2',3'} = J_{6',5'}$ = 7.8); 8.04 (1H, d, 8-H, $J_{8,9}$ = 2.1); 10.47 (1H, s, NH). Mass spectrum: 369 (16), 214 (– 4-MePhSO$_2$, 100). Found, %: C 61.63; H 4.11; N 3.93. $C_{19}H_{15}NO_5S$. Calculated, %: C 61.78; H 3.71; N 3.79.

4-Methyl-6-angelicindiazonium Chloride (5). Compound **1** (0.22 g, 1 mmol) was dissolved with heating in conc. hydrochloric acid (2 ml), the solution cooled to 0 to 2°C, and sodium nitrite (0.075 g, 1.1 mmol) as a 30% solution was added with stirring. The mixture was stirred a further 30 min, the solution filtered, and then used for the further reactions.

6-Azido-4-methylangelicin (7). Sodium azide (0.7 g, 1.1 mmol) was added as a 30% aqueous solution to a solution of diazonium chloride **5** obtained by the method given above. The precipitated solid was filtered off, washed several times with water, dried, and recrystallized from DMF. Yield 1.45 g (60%); mp 204°C (decomp.). ^{1}H NMR spectrum (DMSO-d$_6$), δ, ppm, *J* (Hz): 2.48 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.41 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.32 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.37 (1H, s, 5-H); 8.23 (1H, d, 8-H, $J_{8,9}$ = 2.1). Mass spectrum: 241 (3), 213 (-CO, 100). Found, %: C 59.50; H 2.85; N 17.27. $C_{12}H_7N_3O_3$. Calculated, %: C 59.76; H 2.93; N 17.42.

6-(4-Hydroxybenzylideneimino)-4-methylangelicin (4). *p*-Hydroxybenzaldehyde (0.11 g, 0.87 mmol) was added to a solution of compound **1** (0.2 g, 0.87 mmol) in dioxane and the mixture was heated at 80°C for 15 min. The solid precipitated on cooling was filtered off, and recrystallized from dioxane. Yield 0.2 g (60%); mp 250°C (decomp.). ^{1}H NMR spectrum (DMSO-d$_6$), δ, ppm, *J* (Hz): 2.42 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.30 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 6.93 (2H, d, 2'-H, 6'-H, $J_{2',3'} = J_{6',5'}$ = 8.6); 6.96 (1H, s, 5-H); 7.20 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.75 (2H, d, 3'-H, 5'-H, $J_{3',2'} = J_{5',6'}$ = 8.6); 8.11 (1H, d, 8-H, $J_{8,9}$ = 2.1); 9.79 (1H, s, CH). Mass spectrum: 319 (100), 291 (-CO, 31), 171 (– 4-OHPhCH=N, – CO, 28), 144 (– CO, – HCO, – 4-OHPhCH=N, 40). Found, %: C 71.50; H 4.41; N 4.49. $C_{19}H_{13}NO_4$. Calculated, %: C 71.47; H 4.10; N 4.39.

6-Hydrazino-4-methylangelicin (6). Stannous chloride (0.57 g, 3 mmol) in conc. hydrochloric acid (1 ml) was added dropwise to a cooled solution of 4-methyl-6-angelicindiazonium chloride (1 mmol) so that the temperature did not exceed 10°C. The precipitated white solid hydrazine salt was filtered off and dried in the air. Yield 0.72 g (90%).

Hydrazones from 6-Hydrazino-4-methylangelicin (General Procedure). The appropriate ketone was added to a solution of 6-hydrazino-4-methylangelicin **6** (0.8 g, 1 mmol) in ethanol at room temperature. The precipitated solid was filtered off and recrystallized.

4-Methyl-6-(4-nitrobenzylidenehydrazino)angelicin (8). Yield 78%; mp 252-255°C (dioxane). ^{1}H NMR spectrum (DMSO-d$_6$), δ, ppm, *J* (Hz): 2.58 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.32 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.19 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.56 (1H, s, 5-H); 7.07 (1H, d, 8-H, $J_{8,9}$ = 2.1); 7.85 (2H, d, 2'-H, 6'-H, $J_{2',3'} = J_{6',5'}$ = 9.0); 7.94 (1H, s, CH); 8.28 (2H, d, 3'-H, 5'-H, $J_{3',2'} = J_{5',6'}$ = 9.0). Mass spectrum: 363 (73), 214 (– 4-NO$_2$PhCH=N, 96), 186 (– CO, – 4-NO$_2$PhCH=N, 100). Found, %: C 62.50; H 4.41; N 11.49. $C_{19}H_{15}N_3O_5$. Calculated, %: C 62.46; H 4.14; N 11.50.

6-(4-Hydroxybenzylidenehydrazino)-4-methylangelicin (9). Yield 82%; mp 247-250°C (dioxane). ^{1}H NMR spectrum (DMSO-d$_6$), δ, ppm, *J* (Hz): 2.49 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.63 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 6.82 (2H, d, 2'-H, 6'-H, $J_{2',3'} = J_{6',5'}$ = 8.6); 7.24 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.42 (1H, s, 5-H); 7.54 (2H, d, 3'-H, 5'-H, $J_{3',2'} = J_{5',6'}$ = 8.6); 8.14 (1H, s, CH); 8.16 (1H, d, 8-H, $J_{8,9}$ = 2.3); 9.64 (1H, d, OH); 10.37 (1H, d, NH). Mass spectrum: 334 (40), 214 (– 4-OHPhCH=N, 98), 186 (– CO, – 4-OHPhCH=N, 100). Found, %: C 68.50; H 4.41; N 8.49. $C_{19}H_{14}N_2O_4$. Calculated, %: C 68.26; H 4.22; N 8.38.

6-(1-Ethoxycarbonylethylidenehydrazino)-4-methylangelicin (10). Yield 80%; mp 198-200°C (dioxane). ^{1}H NMR spectrum (DMSO-d$_6$), δ, ppm, *J* (Hz): 1.30 (3H, t, COOCH$_2$<u>CH$_3$</u>); 2.18 (3H, s, CH$_3$C=N); 2.48 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 4.21 (2H, q, COO<u>CH$_2$</u>CH$_3$); 6.39 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.29 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.54 (1H, s, 5-H); 8.20 (1H, d, 8-H, $J_{8,9}$ = 2.1); 9.88 (1H, s, NH). Mass spectrum: 328 (67), 214 (-CH$_3$CNCOOC$_2$H$_5$, 100), 186 (-CH$_3$CNCOOC$_2$H$_5$, -CO, 92). Found, %: C 62.24; H 4.72; N 8.46. $C_{17}H_{16}N_2O_5$. Calculated, %: C 62.19; H 4.91; N 8.53.

6-(2-Butylidenehydrazino)-4-methylangelicin (12). Yield 82%; mp 144-146°C (dioxane). ^{1}H NMR spectrum (acetone-d_6), δ, ppm, *J* (Hz): 1.20 (3H, t, $CCH_2\underline{CH_3}$); 2.05 (3H, s, $CH_3C{=}N$); 2.38 (2H, q, $C\underline{CH_2}CH_3$); 2.51 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.25 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.18 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.53 (1H, s, 5-H); 7.97 (1H, d, 8-H, $J_{8,9}$ = 2.1); 8.00 (1H, s, NH). Mass spectrum: 284 (48), 214 (-$CH_3CNC_2H_5$, 94), 188 (-$CH_3CNC_2H_5$, -CO, 100). Found, %: C 67.59; H 5.67; N 9.85. $C_{16}H_{16}N_2O_3$. Calculated, %: C 67.39; H 5.81; N 9.43.

6-Cyclohexylidenehydrazino-4-methylangelicin (14). Yield 77%; mp 198-200°C (dioxane). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm, *J* (Hz): 1.63 (6H, s, 3'-CH_2, 4'-CH_2, 5'-CH_2); 2.48 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 2.50 (4H, m, 2'-CH_2, 6'-CH_2); 6.32 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.23 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.37 (1H, s, 5-H); 8.13 (1H, d, 8-H, $J_{8,9}$ = 2.1); 8.87 (1H, s, NH). Mass spectrum: 310 (48), 214 (-$(CH_2)_5$=N, 100), 186 (-$(CH_2)_5$=N, -CO, 94). Found, %: C 69.59; H 5.67; N 9.25. $C_{18}H_{18}N_2O_3$. Calculated, %: C 69.66; H 5.85; N 9.03.

9-Ethoxycarbonyl-7-methyl-5-oxopyrano[3,2-*e*]furo[3,2-*g*]indole (11). Hydrazone **10** (0.33 g, 1 mmol) was added to polyphosphoric acid (6 ml) and the mixture stirred at 75°C for 15 min, then poured into ice water. The precipitated solid was filtered off, washed with water, and recrystallized from acetic acid. Yield 0.093 g (30%); mp 280°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm, *J* (Hz): 1.38 (3H, t, $CH_2\underline{CH_3}$); 2.77 (3H, d, 7-Me, $J_{Me,7}$ = 1.1); 4.39 (2H, q, $\underline{CH_2}CH_3$); 6.37 (1H, d, 6-H, $J_{6,Me}$ = 1.1); 7.32 (1H, d, 3-H, $J_{3,2}$ = 2.1); 7.82 (1H, s, 8-H); 8.19 (1H, d, 2-H, $J_{2,3}$ = 2.1); 13.17 (1H, s, NH). Mass spectrum: 311 (35), 265 (-C_2H_5OH, 80), 237 (-C_2H_5OH, -CO, 100). Found, %: C 65.39; H 4.33; N 4.25. $C_{17}H_{13}NO_5$. Calculated, %: C 65.59; H 4.21; N 4.50.

7,8,9-Trimethyl-5-oxopyrano[3,2-*e*]furo[3,2-*g*]indole (13). Several drops of hydrochloric acid were added with stirring to a mixture of hydrazone **12** (0.28 g, 1 mmol) and acetic acid (5 ml). The mixture was stirred at room temperature for 3 h, the precipitated solid was filtered off, dried, and recrystallized from DMF. Yield 0.23 g (85%); mp 247°C (decomp.). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm, *J* (Hz): 2.41 (3H, s, 8-Me); 2.47 (3H, s, 9-CH_3); 2.73 (3H, d, 7-Me, $J_{Me,6}$ = 1.1); 6.21 (1H, d, 6-H, $J_{6,Me}$ = 1.1); 7.17 (1H, d, 3-H, $J_{3,2}$ = 2.1); 7.55 (1H, d, 2-H, $J_{2,3}$ = 2.1); 8.53 (1H, s, NH). Mass spectrum: 267 (100), 239 (-CO, 76), 225 (-CO, -CH_2, 33). Found, %: C 71.86; H 4.73; N 5.25. $C_{16}H_{13}NO_3$. Calculated, %: C 71.90; H 4.90; N 5.24.

7-Methyl-5-oxo-8,9,10,11-tetrahydropyrano[2,3-*c*]furo[2,3-*a*]carbazole (15). Several drops of hydrochloric acid were added to a solution of hydrazone **14** (0.33 g, 1 mmol) in acetic acid (5 ml). The mixture was heated under reflux for 1 h, then cooled, and poured onto ice. The precipitate was filtered off, dried, and recrystallized from toluene. Yield 0.26 g (90%); mp 253°C (decomp.). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm, *J* (Hz): 2.87-2.93 (8H, m, 8,9,10,11-CH_2); 2.74 (3H, d, 7-Me, $J_{Me,6}$ = 1.1); 6.21 (1H, d, 6-H, $J_{6,Me}$ = 1.1); 7.19 (1H, d, 3-H, $J_{3,2}$ = 2.1); 8.47 (1H, d, 2-H, $J_{2,3}$ = 2.1); 8.47 (1H, s, NH). Mass spectrum: 293 (71), 265 (-CO, 37), 237 (-2CO, 100). Found, %: C 73.86; H 5.23; N 4.65. $C_{18}H_{15}NO_3$. Calculated, %: C 73.71; H 5.15; N 4.78.

6-(3,5-Dimethyl-1-pyrazolyl)-4-methylangelicin (16). Acetylacetone (0.86 g, 1.0 mmol) was added to a solution of hydrazine **6** (0.8 g, 1.0 mmol) in alcohol at room temperature. The mixture was stirred until the formation of a white solid, which was filtered off, and recrystallized from chloroform. Yield 2.1 g (70%); mp 208-210°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm, *J* (Hz): 2.21 (3H, s, 3'-Me); 2.34 (3H, s, 5'-Me); 2.51 (3H, d, 4-Me, $J_{Me,3}$ = 1.1); 6.09 (1H, s, 4'-H); 6.33 (1H, q, 3-H, $J_{3,Me}$ = 1.1); 7.22 (1H, d, 9-H, $J_{9,8}$ = 2.1); 7.60 (1H, s, 5-H); 7.70 (1H, d, 8-H, $J_{8,9}$ = 2.1). Mass spectrum: 294 (100), 199 (– 3'-CH_3-pyrazolyl, – 5'-CH_3-pyrazolyl, 49). Found, %: C 69.30; H 4.61; N 9.28. $C_{17}H_{14}N_2O_3$. Calculated, %: C 69.38; H 4.79; N 9.52.

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