Addition of acetylacetone and ethyl acetoacetate to carbodiimides promoted by nickel acetylacetonate

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The addition reactions of acetylacetone and ethyl acetoacetate with diphenylcarbodiimide and dicyclohexylcarbodiimide in the presence of nickel acetylacetonate afforded *N*,*N*′-substituted α,α-dioxoketene aminals. Adducts of acetylacetone with carbodiimides are readily deacetylated with MeONa in MeOH to form acetylketene aminals. Ketene aminals were used for the synthesis of functionalized 1,2,3-triazoles.

Key words: carbodiimides, acetylacetone, ethyl acetoacetate, addition to the C=N bond, catalysis, nickel acetylacetonate, deacetylation, α,α-dioxoketene aminals, 1-R-4-acetyl-5-(R-amino)-1,2,3-triazoles, biheterocycles.

α,α-Dioxoketene aminals are convenient starting compounds for the construction of heterocyclic systems.[1—6] In continuation of our investigations on the synthesis of this type of reagents from cyanamides,[7—10] we studied the reactions of acetylacetone and ethyl acetoacetate with diphenylcarbodiimide (DPC) and dicyclohexylcarbodiimide (DCC) catalyzed by nickel acetylacetonate $Ni(acac)_2$.

It is known that β-diketonates of a number of transition metals catalyze the addition of methylene-active β-diketones and ethyl acetoacetate to the C=N bond of isocyanates.[11—14] This fact gives grounds to expect that analogous reactions of β-dicarbonyl compounds with carbodiimides will afford *N*,*N*′-disubstituted α,α-dioxoketene aminals.

Examples of the addition of methylene-active compounds to carbodiimides in the presence of basic catalysts have been described earlier. More than a hundred years ago, an adduct was prepared by the reaction of malonic ester with DPC.[15] Much more later, this adduct was demonstrated[3] to be dianilinomethylenemalonic ester. *N*,*N*′-Substituted aminals were synthesized analogously from dibenzoylmethane and benzoylacetic ester.[3]

However, the reactions in the presence of bases are often accompanied by undesirable side processes due to instability of the starting compounds or final products with respect to the catalysts used. Presumably, that is the reason why data on the reactions of acetylacetone or ethyl acetoacetate with diphenylcarbodiimide are lacking in the literature, since the expected ketene aminals can be subjected to deacetylation in the presence of a base. For example, it is known that the reactions of acetylacetone with trihaloacetonitriles resulted finally in deacetylation of the adducts formed.[16,17] The reactions of DCC, which is less electrophilic than DPC, even with malonic ester or indane-1,3-dione in the presence of EtONa do not give ketene aminals, and only the reaction with Meldrum's acid affords the corresponding adduct.[18] Meldrum's acid reacts also with some other carbodiimides.[19]

We found that acetylacetone and ethyl acetoacetate readily added to DPC on refluxing in THF in the presence of catalytic amounts of $Ni(acac)_2$ to form ketene aminals **1a,b**, which were isolated in 70% and 75% yields, respectively (Scheme 1).* In the case of less reactive DCC, the addition of acetylacetone proceeded not so readily. To achieve the best results, a large excess of diketone and an equivalent amount of $Ni(acac)_2$ (with respect to DCC) are required. Under these conditions, ketene aminal **1c** was prepared in 67% yield with respect to DCC. In the reaction with the use of a ten times smaller amount of $Ni(acac)_2$, the yield of adduct **1c** decreased to 40% in spite of substantially more prolonged heating of the reaction mixture.

Colorless crystalline compounds **1a,b** are readily soluble in acetone, benzene, and chloroform but are poorly soluble in ethanol and light petroleum. The structures of **1a,b** were confirmed by physicochemical methods and elemental analysis. The mass spectra of ketene aminals **1a,b** are characterized by the presence of molecular ion peaks. In the ^1H NMR spectrum of compound **1a** in $CDCl_3$, both Me groups give a singlet at δ 2.50. The results of IR and ^1H NMR spectroscopy are also indica-

* We chose $Ni(acac)_2$ as the catalyst because it showed the highest efficiency compared to acetylacetonates of other metals in the reaction of acetylacetone with phenyl isocyanate.[13]

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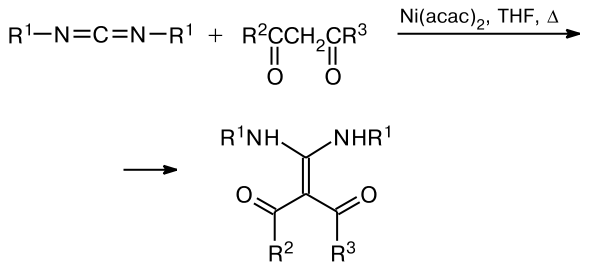
Scheme 1



1a—c

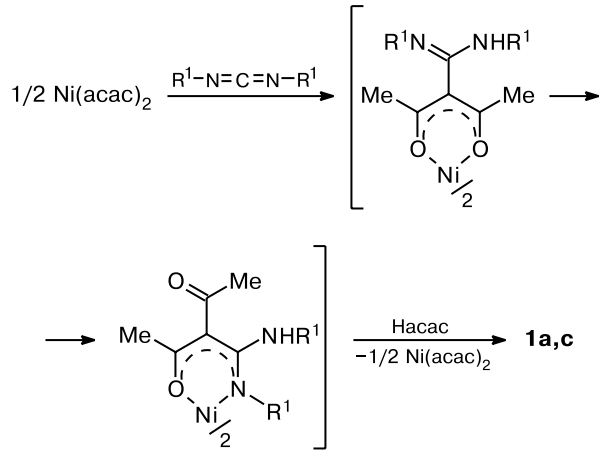
R^1 = Ph, R^2 = R^3 = Me (**a**); R^1 = Ph, R^2 = Me, R^3 = OEt (**b**); R^1 = *cyclo*-C_6H_{11}, R^2 = R^3 = Me (**c**)

tive of the formation of intramolecular N—H...O hydrogen bonds in solutions, which is typical of diacylketene aminals.[8,9]

The crystalline product prepared from DCC is poorly soluble in organic solvents (including DMSO), but the results of mass spectrometry and IR spectroscopy (in KBr) are in complete agreement with structure **1c**.

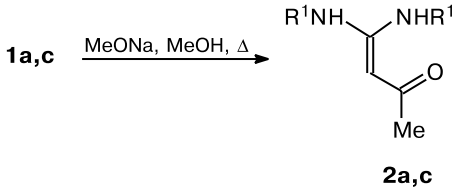
The role of $Ni(acac)_2$ in promotion of the reactions of acetylacetone with carbodiimides is illustrated in Scheme 2, which is analogous to the scheme of catalysis of the addition of acetylacetone to benzoylcyanamide proposed earlier.[8]

Scheme 2



Ethyl acetoacetate adds to carbodiimides as its Ni chelate formed from $Ni(acac)_2$ in the presence of an excess of the ester. As expected (see above), ketene aminals **1a,c** are readily deacylated with MeONa in boiling MeOH to give monoacetylketene aminals **2a,c** (Scheme 3) (*cf.* also deacetylation of diacylketene aminals derived from cyanamides[20]).

Scheme 3



2a,c

R^1 = Ph (**a**), *cyclo*-C_6H_{11} (**c**)

Compounds **2a,c** are colorless crystalline compounds, which are readily soluble in acetone, ethanol, chloroform, and benzene. Their structures were confirmed by spectroscopic methods (IR, ^{1}H NMR, and mass spectra). The ^{1}H NMR spectra (in $CDCl_3$) of ketene aminals **2a,c** are characterized by the presence of a singlet of HC=C at δ 4.91 and 4.54, respectively. According to the IR and ^{1}H NMR spectroscopic data, one of the NH groups is involved in an intramolecular N—H...O hydrogen bond.

Earlier,[21] compound **2a** has been synthesized by the reaction of aniline with β,β-dichlorovinyl methyl ketone.

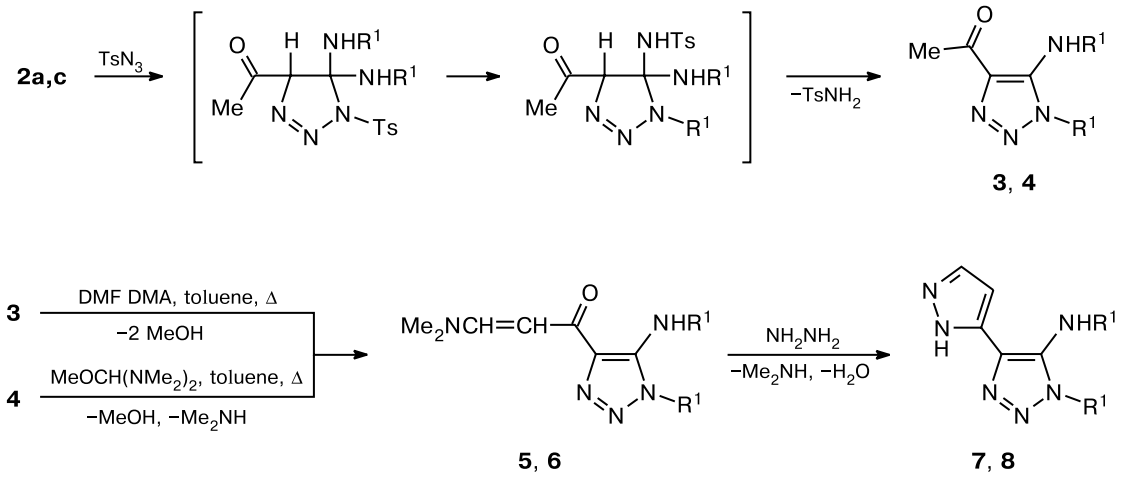
Ketene aminals prepared from carbodiimides can be used as chelating ligands and starting compounds for the construction of heterocyclic compounds. Earlier, we have demonstrated[22] that monoacylketene aminals containing the unsubstituted NH_2 group can be involved in the 1,3-dipolar addition to tosyl azide.[22] The subsequent Dimroth rearrangement and elimination of $TsNH_2$ afford 4-acyl-5-amino-1,2,3-triazole derivatives. Analogously, compounds **2a,c** are smoothly transformed into the corresponding 1,2,3-triazoles **3** and **4** (Scheme 4).

Acetyltriazoles can react with DMF dimethylacetal (DMF DMA) to give condensation products at the methyl group of the acetyl fragment.[22] Correspondingly, refluxing of triazole **3** with DMF DMA in toluene afforded enaminone **5**. Its treatment with hydrazine hydrate in boiling THF gave rise to 1-phenyl-5-phenylamino-4-(pyrazol-5-yl)-1,2,3-triazole (**7**).

It appeared that triazole **4** did not react with DMF DMA under analogous conditions and even on refluxing in xylene. We succeeded in preparing the corresponding enaminone **6** in good yield from compound **4** only with the use of a stronger electrophilic reagent, *viz.*, bis(dimethylamino)methoxymethane.[23] Subsequent cyclization with hydrazine hydrate in EtOH afforded biheterocycle **8**.

Crystalline 1,2,3-triazole derivatives **3—8** were isolated in 70—90% yields. Compounds **3**, **4**, **7**, and **8** are readily soluble in organic solvents (triazoles **3**, **7**, and **8** are poorly soluble only in light petroleum, and triazole **7** is poorly soluble also in MeCN). The spectroscopic characteristics of heterocycles **3—8** are in agreement with their structures (for pyrazolyltriazoles **7** and **8**, the most probable position of the proton of NH in the pyrazole ring is presented).

Scheme 4



R^1 = Ph (**2a**, **3**, **5**, **7**), *cyclo*-C_6H_{11} (**2c**, **4**, **7**, **8**)

Compounds **7** and **8** are of interest as potential chelating ligands.

Experimental

The ^{1}H NMR spectra were recorded on a Bruker WM-250 instrument. The ^{13}C NMR spectra were measured on a Bruker AM-300 spectrometer. The IR spectra were recorded on a Specord M-80 instrument. The mass spectra were obtained on a Kratos MS-30 instrument (EI, 70 eV, the temperature of an ionization chamber was 250 °C, a direct inlet system). Diphenylcarbodiimide was synthesized by dehydration of diphenylurea.[24] Tosyl azide[25] and bis(dimethylamino)methoxymethane[23] were prepared according to known procedures.

3-(Dianilinomethylene)pentane-2,4-dione (1a).* A mixture of DPC (12.25 g, 63 mmol), acetylacetone (7.7 mL, 75 mmol), and $Ni(acac)_2$ (1.67 g) in dry THF (30 mL) was refluxed for 12 h (TLC control), cooled to ~20 °C, and kept for 16 h. The precipitate that formed was filtered off and ketene aminal **1a** was obtained in a yield of 11 g. The filtrate was concentrated and the residue was recrystallized from EtOH to obtain additionally 2 g of compound **1a**, the total yield was 70%, m.p. 154—156 °C. Found (%): C, 73.56; H, 6.03; N, 9.88. $C_{18}H_{18}N_2O_2$. Calculated (%): C, 73.45; H, 6.16; N, 9.51. IR ($CHCl_3$), ν/cm^{-1}: 3300—3040 (NH, CH); 1615, 1595, 1570 (CO, C=C). ^{1}H NMR ($CDCl_3$), δ: 2.50 (s, 6 H, Me); 6.80—6.90 (m, 6 H, Ph); 7.00 (m, 4 H, Ph); 12.95 (br.s, 2 H, NH). MS, *m/z*: 294 $[M]^+$.

Ethyl 2-(dianilinomethylene)-3-oxobutanoate (1b). A mixture of DPC (4.47 g, 23 mmol), ethyl acetoacetate (2.9 mL, 23 mmol), and $Ni(acac)_2$ (0.4 g, 1.6 mmol) in dry THF (10 mL) was refluxed for 6—8 h (TLC control). The solvent was evaporated *in vacuo* and the residue was chromatographed on a column with SiO_2 ($CHCl_3$ as the eluent). After evaporation of the solvent, the residue was recrystallized from EtOH to prepare ketene aminal **1b** in a yield of 5.6 g (75%), m.p. 107—108 °C. Found (%): C, 70.44; H, 6.19; N, 9.02. $C_{19}H_{20}N_2O_3$. Calculated (%): C, 70.35; H, 6.21; N, 8.64. IR ($CHCl_3$), ν/cm^{-1}: 3300—3040 (NH, CH); 1625, 1597, 1585 (CO, C=C). ^{1}H NMR ($CDCl_3$), δ: 1.39 (t, 3 H, Me, J = 6.8 Hz); 2.51 (s, 3 H, MeCO); 4.31 (q, 2 H, CH_2, J = 6.8 Hz); 6.80—7.05 (m, 10 H, Ph); 12.60 (br.s, 2 H, NH). MS, *m/z*: 324 $[M]^+$.

3-(Dicyclohexylaminomethylene)pentane-2,4-dione (1c). A mixture of DCC (1.2 g, 5.8 mmol), $Ni(acac)_2$ (0.75 g, 2.9 mmol), and acetylacetone (10 mL, 97.5 mmol) in dry THF (9 mL) was refluxed for 13—14 h and cooled to ~20 °C. The precipitate that formed was filtered off and washed with THF (10 mL) and EtOH (10 mL). Ketene aminal **1c** was obtained in a yield of 1.2 g (67%), m.p. 263—265 °C. Found (%): C, 70.39; H, 9.75; N, 9.17. $C_{18}H_{30}N_2O_2$. Calculated (%): C, 70.55; H, 9.87; N, 9.14. IR (KBr), ν/cm^{-1}: 3300—3100 (NH, CH); 1630, 1560 (CO, C=C). MS, *m/z*: 306 $[M]^+$.

4,4-(Dianilino)but-3-en-2-one (2a). A mixture of ketene aminal **1a** (10 g, 34 mmol) and MeONa (34 mmol, from 0.78 g of Na) in MeOH (300 mL) was refluxed for 3—4 h (TLC control), cooled to ~20 °C, acidified with AcOH to pH ~7, and concentrated *in vacuo*. Chloroform (50 mL) was added to the residue, AcONa was filtered off, the filtrate was concentrated, and the residue was recrystallized from a benzene—hexane mixture. Compound **2a** was obtained in a yield of 6.30 g (73%), m.p. 114—115 °C (*cf.* lit. data[21]: 116—117 °C). ^{1}H NMR ($CDCl_3$), δ: 2.00 (s, 3 H, Me); 4.91 (s, 1 H, CH=); 6.19 (br.s, 1 H, NH); 7.12—7.50 (m, 10 H, Ph); 12.90 (br.s, 1 H, NH).

4,4-(Dicyclohexylamino)but-3-en-2-one (2c). Compound **2c** was synthesized analogously to ketene aminal **2a** from ketene aminal **1c** (11.4 g, 37 mmol) and MeONa in MeOH. The reaction was completed in 6—8 h (TLC control) to give an oil, which was crystallized by trituration with a mixture of hexane and diethyl ether and recrystallized from a mixture of hexane and benzene. The yield was 8.4 g (85%), m.p. 154—155 °C. Found (%): C, 72.63; H, 10.65; N, 10.28. $C_{16}H_{28}N_2O$. Calculated (%): C, 72.67; H, 10.67; N, 10.59. IR ($CHCl_3$), ν/cm^{-1}: 3448 (NH); 3300—3100 (NH, CH); 1605, 1570 (CO, C=C). ^{1}H NMR ($CDCl_3$), δ: 1.10—1.95 (m, 20 H, CH_2); 2.00 (s, 3 H,

* A. V. Zubarev participated in the synthesis of compounds **1a—c**.

Me); 3.10—3.38 (m, 2 H, CH); 4.00 (br.s, 1 H, NH); 4.54 (s, 1 H, CH=); 11.12 (br.s, 1 H, NH). MS, *m/z*: 264 $[M]^+$.

4-Acetyl-1-phenyl-5-phenylamino-1,2,3-triazole (3). A mixture of ketene aminal **2a** (1.01 g, 4 mmol) and TsN_3 (0.79 g, 4 mmol) in THF (15 mL) was stirred at ~20 °C for 24 h. The solvent was removed *in vacuo*, the residue was extracted with heptane heated to boiling (4×15 mL), the mixture was concentrated, and triazole **3** was obtained in a yield of 0.94 g (85%), m.p. 112—113 °C (from heptane). Found (%): C, 69.18; H, 5.24; N, 20.06. $C_{16}H_{14}N_4O$. Calculated (%): C, 69.05; H, 5.07; N, 20.13. IR (KBr), ν/cm^{-1}: 3270—3250 (NH); 1640 (CO), 1595, 1575, 1515. ^{1}H NMR ($CDCl_3$), δ: 2.75 (s, 3 H, Me); 6.70 (m, 2 H, Ph); 6.90 (m, 1 H, Ph); 7.00 (m, 2 H, Ph); 7.12—7.38 (m, 5 H, Ph); 8.78 (br.s, 1 H, NH). MS, *m/z*: 278 $[M]^+$.

4-Acetyl-1-cyclohexyl-5-cyclohexylamino-1,2,3-triazole (4). A mixture of ketene aminal **2c** (1.06 g, 4 mmol) and TsN_3 (0.79 g, 4 mmol) in THF (15 mL) was stirred at ~20 °C for 24 h. The solvent was removed *in vacuo* and light petroleum (20 mL) was added to the residue. The mixture was heated to boiling and cooled to ~20 °C. Then $TsNH_2$ that precipitated was filtered off, the filtrate was concentrated, and triazole **4** was obtained in a yield of 0.98 g (85%). An analytical sample was purified from a small amount of a TsN_3 impurity by column chromatography on SiO_2 (C_6H_6 and $CHCl_3$ were used successively as the eluents), m.p. 62—63 °C. Found (%): C, 66.20; H, 8.86; N, 19.23. $C_{16}H_{26}N_4O$. Calculated (%): C, 66.17; H, 9.02; N, 19.29. IR (KBr), ν/cm^{-1}: 3305 (NH); 1645 (CO); 1575. ^{1}H NMR ($CDCl_3$), δ: 1.20—2.15 (m, 20 H, CH_2); 2.60 (s, 3 H, Me); 3.29 (m, 1 H, CH); 4.02 (m, 1 H, CH); 6.42 (d, 1 H, NH, J = 9.8 Hz). ^{13}C NMR ($CDCl_3$), δ: 24.14, 24.87, 25.16, 25.52, 32.44, 33.82 (CH_2); 54.10, 58.28 (CH); 131.55 (q, C(4), ^3J = 3.2 Hz); 144.98 (C(5)); 193.86 (q, CO, ^2J = 6.0 Hz). MS, *m/z*: 290 $[M]^+$.

4-[3-(Dimethylamino)acryloyl]-1-phenyl-5-phenylamino-1,2,3-triazole (5). A mixture of triazole **3** (0.278 g, 1 mmol) and DMF DMA (0.20 mL, 1.5 mmol) in toluene (7 mL) was refluxed for 4 h and cooled to ~20 °C. Light petroleum (10 mL) was added and the precipitate that formed was filtered off to obtain triazole **5** in a yield of 0.23 g (70%), m.p. 201—202 °C. Found (%): C, 68.27; H, 5.82; N, 21.03. $C_{19}H_{19}N_5O$. Calculated (%): C, 68.45; H, 5.74; N, 21.01. ^{1}H NMR ($CDCl_3$), δ: 2.98 and 3.12 (both s, 6 H, NMe_2); 6.15 (d, 1 H, CH=, J = 13 Hz); 6.68 (m, 2 H, Ph); 6.82 (m, 1 H, Ph); 6.95 (m, 2 H, Ph); 7.12—7.30 (m, 3 H, Ph); 7.40—7.50 (m, 2 H, Ph); 7.88 (d, 1 H, CH=, J = 13 Hz); 9.17 (br.s, 1 H, NH).

1-Cyclohexyl-5-cyclohexylamino-4-[3-(dimethylamino)acryloyl]-1,2,3-triazole (6). A mixture of triazole **4** (0.29 g, 1 mmol) and bis(dimethylamino)methoxymethane (0.29 mL, 2 mmol) in toluene (5 mL) was refluxed for 5 h. The solvent was removed *in vacuo*, light petroleum (6 mL) was added to the residue, the precipitate that formed was filtered off, and triazole **6** was obtained in a yield of 0.31 g (90%), m.p. 96—97 °C. Found (%): C, 65.71; H, 9.09; N, 20.12. $C_{19}H_{31}N_5O$. Calculated (%): C, 66.05; H, 9.04; N, 20.27. ^{1}H NMR ($CDCl_3$), δ: 1.15—2.20 (m, 20 H, CH_2); 2.80—3.25 (m, 7 H, NMe_2 and CH); 3.95—4.12 (m, 1 H, CH); 6.10 (d, 1 H, CH=, J = 12.5 Hz); 6.51 (d, 1 H, NH, J = 10.7 Hz); 7.77 (d, 1 H, CH=, J = 12.5 Hz).

1-Phenyl-5-phenylamino-4-(pyrazol-5-yl)-1,2,3-triazole (7). A mixture of triazole **5** (0.167 g, 0.5 mmol) and hydrazine hydrate (0.1 mL, 2 mmol) in THF (4 mL) was refluxed for 1.5 h. The solvent and an excess of hydrazine were removed *in vacuo* (12 Torr) at ~100 °C. Benzene (2 mL) and light petroleum (3 mL) were added to the residue and the precipitate that formed was filtered off. The yield of triazole **7** was 0.133 g (88%), m.p. 192—193 °C (from MeCN). Found (%): C, 67.28; H, 4.81; N, 27.99. $C_{17}H_{14}N_6$. Calculated (%): C, 67.53; H, 4.67; N, 27.80. IR (KBr), ν/cm^{-1}: 3360—3120 (NH); 1615, 1598, 1520. ^{1}H NMR ($CDCl_3$), δ: 6.60—6.72 (m, 3 H, Ph and H(4) Pyr); 6.82 (m, 1 H, Ph); 6.93 (br.s, 1 H, NH); 7.10 (m, 2 H, Ph); 7.30—7.50 (m, 3 H, Ph); 7.52—7.75 (m, 3 H, Ph and H(3) Pyr). MS, *m/z* (I_{rel} (%)): 302 $[M]^+$ (63), 274 $[M - N_2]^+$ (87), 77 $[Ph]^+$ (100).

1-Cyclohexyl-5-cyclohexylamino-4-(pyrazol-5-yl)-1,2,3-triazole (8). A mixture of triazole **6** (0.173 g, 0.5 mmol) and hydrazine hydrate (0.1 mL, 2 mmol) in EtOH (4 mL) was refluxed for 1.5 h. The solvent and an excess of hydrazine were removed *in vacuo* (12 Torr) at ~100 °C. The residue was extracted with heptane (9 mL) heated to boiling, the extract was cooled, and the precipitate that formed was filtered off (operations were repeated five more times using the filtrate). Pyrazolyltriazole **8** was obtained in a yield of 0.11 g (72%), m.p. 127—128 °C. Found (%): C, 64.55; H, 8.20; N, 26.80. $C_{17}H_{26}N_6$. Calculated (%): C, 64.94; H, 8.33; N, 26.73. IR (KBr), ν/cm^{-1}: 3298, 3200—3120 (NH); 1607, 1545, 1505. ^{1}H NMR ($CDCl_3$), δ: 1.00—2.20 (m, 20 H, CH_2); 2.83—3.05 (m, 1 H, CH); 4.05—4.25 (m, 1 H, CH); 4.44 (br.s, 1 H, NH); 6.76 (d, 1 H, H(4) Pyr, J = 1.7 Hz); 7.61 (d, 1 H, H(3) Pyr, J = 1.7 Hz). ^{13}C NMR ($CDCl_3$), δ: 24.87, 25.19, 25.60, 25.77, 32.93, 33.95 (CH_2); 56.95, 57.29 (CH); 102.45 (dd, C(4) Pyr, ^1J = 178 Hz, ^2J = 8 Hz); 129.98 (C(4)); 131.24 (d, C(3) Pyr, ^1J = 185 Hz); 138.56 (C(5)); 143.03 (C(5) Pyr). MS, *m/z* (I_{rel} (%)): 314 $[M]^+$ (62), 55 (100).

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