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Electrophilic Addition of Alcohols to 1-Vinyl-2-phenylazopyrroles and Unexpected Formation of 2-Methylquinoline

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Abstract—1-Vinyl-2-phenylazopyrroles react with alcohols in the presence of acids or $PdCl_2$ to give 1-(1-alkoxyethyl)-2-phenylazopyrroles in up to 49% yield. In the presence of trifluoroacetic acid 2-methylquinoline unexpectedly formed (yield up to 26%) involving into the reaction the phenylazo and 1-vinyl groups.

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Recently aryl- and hetarylazopyrrole dyes attracted superior attention due to their growing role in the development of materials for advanced technology [1–8].

Notwithstanding significant achievements in this field the arylazopyrroles N-vinyl derivatives remain practically unexplored. Only several C-vinyl compounds of this series were described [9–12]. We developed recently a simple and efficient procedure for introducing an arylazo group into the position *2* of the pyrrole ring of 1-vinylpyrroles [13] readily obtained from ketoximes and acetylene [14, 15]. 1-Vinyl substituent significantly increases the synthetic potential of pyrrole azo dyes for it provides a possibility of further functionalization.

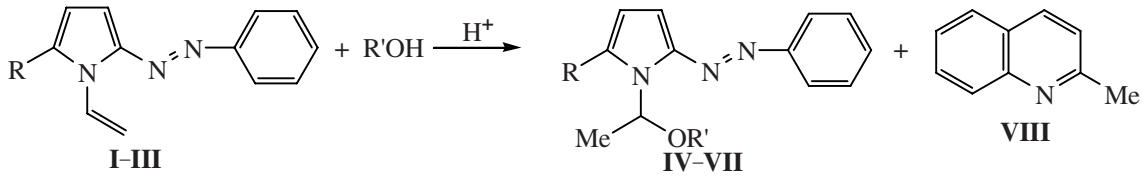
This study treats the trends in the electrophilic addition of alcohols to 1-vinyl-2-phenylazopyrroles with various substituents attached to the pyrrole ring. This reaction may be one among the routes of azopyrroles functionalization and also may serve a model for investigation of the competing reactivity of two nucleophilic sites: the vinyl substituent and the phenylazo group.

1-Vinylpyrroles are known [14–16] to take up alcohols in the presence of acids forming 1-(1-alkoxyethyl)pyrroles. Experiments demonstrated that 1-vinyl-2-phenylazopyrroles **I–III** in the presence of trifluoroacetic acid added alcohols to the 1-vinyl group providing the expected 1-(1-alkoxyethyl)-2-phenylazopyrroles **IV–VII** in preparative yields up to 49% (Scheme 1).

We tested also as catalysts also the trifluoromethanesulfonic acid and metal salts ($AgNO_3$, $PdCl_2$, $NiCl_2$), but the best results were obtained with CF_3COOH. The reaction was carried out in excess alcohol at the boiling point. The reaction did not occur at the equimolar ratio compound **I–III**:alcohol. The reaction progress was monitored by analysis of the reaction mixtures by GLC and ^1H NMR spectroscopy.

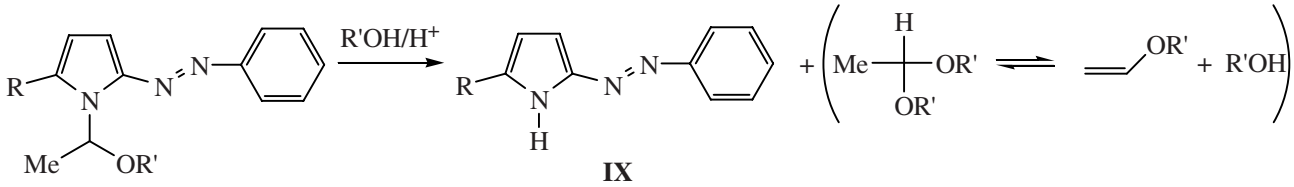
A selective formation of adduct **IV** was observed only in the reaction of 1-vinyl-2-phenylazopyrrole (**I**) with methanol. At the use of other alcohols (see the table) we

Scheme 1.



R = H (**I**), Me (**II**), Ph (**III**); R = H, R' = Me (**IV**), Et (**V**), *i*-Pr (**VI**), *t*-Bu (**VII**).

Scheme 2.



unexpectedly isolated 2-methylquinoline (**VIII**) as a side product. Alongside the adducts and 2-methylquinoline (**VIII**) virtually in all events products of alcoholysis, 2-phenylazopyrroles **IX**, were detected in amounts sometimes reaching 7% (Scheme 2).

Also polymer products formed in the reaction (a considerable difference was observed between the conversion of initial pyrroles **I–III** and the overall yield of the isolated products).

The experimental data compiled in the table show that the yield of adducts decreased with the lower acidity of the alcohol (with the growing donor effect of the R' radical) in keeping with the electrophilic characted of the addition reaction. In the same direction operated the expected steric effect of these substituents. The readiness of 2-methylquinoline (**VIII**) formation also grew in the same succession (Me → *t*-Bu).

Presumable reasons of the lower reactivity of 1-vinyl-2-phenylazopyrroles in electrophilic addition to alcohols compared with that of 1-vinylpyrroles [16] are the partial bonding of the catalyzing proton by the unshared electron pair of a nitrogen of the azo group and the electron-withdrawing effect of the azo substituent which decreases the nucleophilicity of the double bond. By an example of the reaction of 1-vinyl-2-phenylazopyrrole (**I**) conditions were developed for preparation of adducts **IV–VII** in higher yields. It was attained by fractional addition of CF_3COOH (~ 1% to the weight of azopyrrole **I** in each hour during the total reaction time). In another case (single addition of the acid in amount of 1% to the weight of azopyrrole **I**) the reaction stopped already after 1 h. This fact is in good agreement with the concurrent formation of 2-methylquinoline (**VIII**) that neutralizes the acid catalyst.

One more experimental feature of the reaction was revealed: At the one-shot addition of CF_3COOH in equimolar quantity with respect to azopyrroles **I–III** the reaction shifted completely to the formation of 2-methylquinoline (**VIII**).

Conditions of reaction between 1-vinyl-2-phenylazopyrroles **I–III** and alcohols R'OH, and yields of reaction products (molar ratio alcohol–pyrrole 100:1, acid concentration ~ 3–6% to the weight of compounds **I–III**)

Compd. no.	R'	Temperature, °C	Reaction time, h	Composition of crude reaction product, % (GLC data)[a]			Azopyrrole conversion, %
				adduct	2-methylquinoline	2-phenylazopyrrole	
I	Me	65	3.5	97 (49)	–	3	100
	Et	78	3.5	73 (33)	20 (10)	5	98
	i-Pr	82	3.5	50 (20)	25 (12)	2	77
	t-Bu	83	3.5	31 (12)	36 (16)	5	72
II	Me	65	3.5	21	74	3	98
	Et	78	3.5	–	88	4	92
	i-Pr	82	3.5	–	83	7	90
	t-Bu	83	3.5	–	79	2	81
III	Me	65	6	–	30	2	32
	Et	78	6	–	29	2	31
	i-Pr	82	6	–	14	1	15
	t-Bu	83	6	–	8	1	9

[a] Preparative yields are presented in parentheses.

The introduction of a methyl substituent into the position *2* of the pyrrole ring led to increased content of 2-methylquinoline in the reaction mixture, and therewith the adducts formation was detected only by GLC. Phenyl substituent in keeping with its electron-withdrawing effect significantly decreases the reactivity of the vinyl group. This was proved by the fact that even in 6 h the conversion of initial azopyrrole did not exceed 32%, the main reaction product was 2-methylquinoline, and the adducts formation was not detected even by GLC.

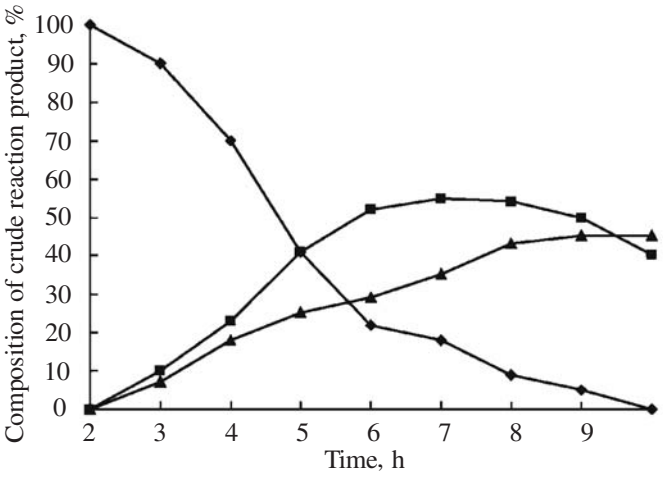
The prolonging of the process (from 3.5 to 7 h) in case of reactions between azopyrroles **I** and **II** with 2-propanol and *tert*-butanol resulted in a complete conversion of the initial pyrroles, but the preparative yields of adducts and also of 2-methyl-quinoline here decreased due to the development of side polymerization processes.

The Lewis acids and their certain combinations are known to catalyze alcohols addition to 1-vinylpyrroles [16]. By an example of methanol addition to 1-vinyl-2-phenylazopyrrole (**I**) we found that among the tested salts only palladium chloride in amount of 10% to the weight of azopyrrole **I** catalyzed the reaction, but the conversion of pyrrole **I** within 12 h was only 30%, and therewith the GLC analysis revealed the presence in the reaction mixture only of adduct **IV**.

Special experiments demonstrated that 2-methylquinoline (**VIII**) did not form either from 1-vinyl-2-phenylpyrrole or from 2-phenylazopyrrole. Therefore, firstly, its formation required the presence in the pyrrole ring both of 1-vinyl group and 2-phenylazo substituent, and secondly, the molecule of 2-methylquinoline was formed from phenylazo moiety. The latter conclusion was unambiguously proved by isolation from the reaction products of 1-vinyl-5-methyl-2-(4-ethoxyphenylazo)-pyrrole (**X**) with 2-propanol of 2-methyl-6-ethoxyquinoline (**XI**) in 26% yield (Scheme 3).

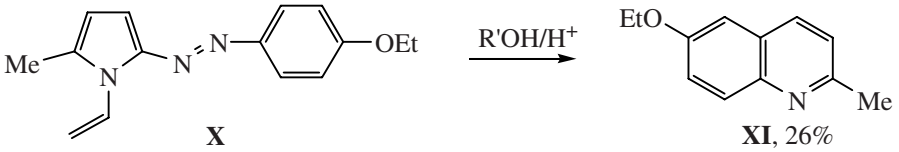
The route of 2-methylquinolines formation from pyrroles **I–III** is likely to include three stages (Scheme 4). 1. Pyrrole **IX** (alcoholysis product, Scheme 2) in a tautomeric form reacts with acetal (the second alcoholysis product, Scheme 2) in the presence of acid to give cation **A** that decomposes into 2-(alkoxyimino)-2*H*-pyrrole **B** and iminium cation **C**. 2. Cation **C** adds to the vinyl ether (the product of acetal decomposition catalyzed with acid, Scheme 2) and further thus arising cation **D** closed into tetrahydroquinoline cation **E**. 3. Further aromatization of the latter with alcohol elimination and oxidation effected by alkoxyimine **B** gives quinoline **VIII**, and the unstable 2-imino-2*H*-pyrrole (**XII**) that suffers polymerization.

Kinetic monitoring (GLC, ^{1}H NMR) of the reaction between pyrroles **I–III** and alcohols gave direct proofs that adducts **IV–VII** are precursors of quinoline **VIII**. Typical kinetic curves (see the figure) show that as initial pyrrole **I** is consumed the concentration of adduct **IV** grows, goes through a maximum and starts to decrease, whereas the concentration of 2-methylquinoline **VIII** continuously grows. This pattern of kinetic curves is characteristics of successive reactions of comparable rates.



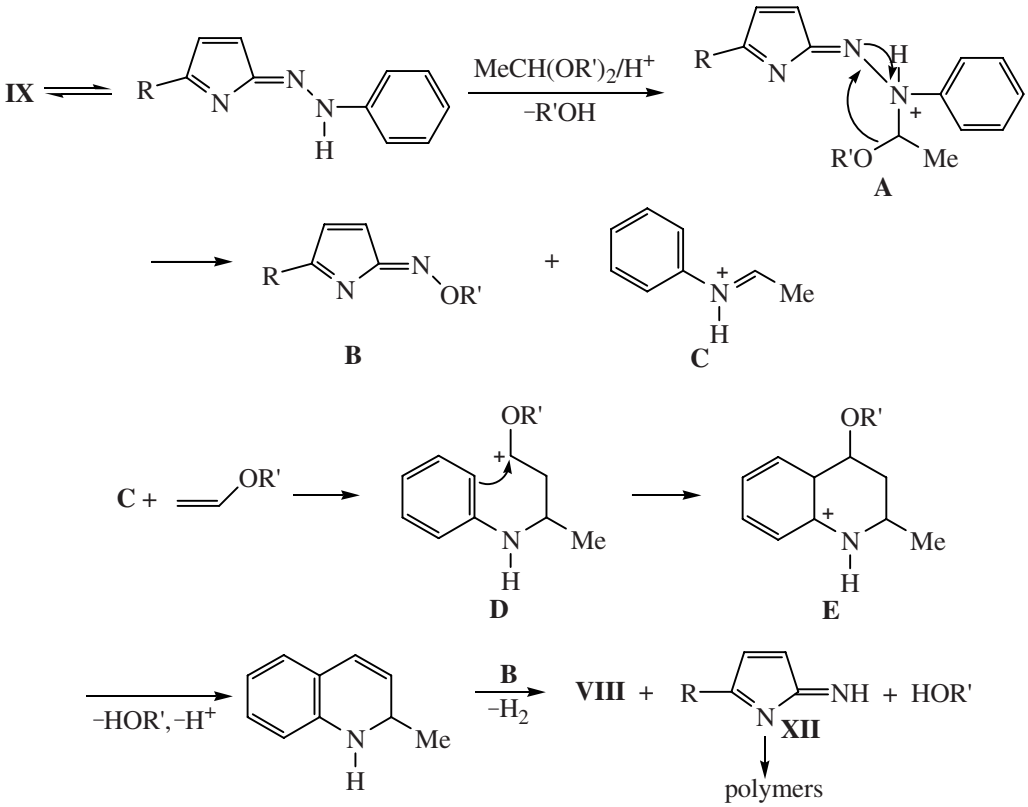
Variation of concentration of pyrrole **I** (a), adduct **IV** (b), and 2-methylquinoline **VIII** (c) in the course of pyrrole **I** reaction with 2-propanol [molar ratio (**I**); 2-propanol 1:100, boiling, 8 wt% of CF_3COOH with respect to compound **I**].

Scheme 3.



X **XI**, 26%

Scheme 4.



Inasmuch as the forming 2-methylquinoline bound the acid catalyst into a catalytically inactive salts, the fractional addition of acid into the reaction mixture was required to get the maximum adduct yield. It is consequently understandable why the one-shot addition of CF_3COOH to the reaction mixture provids the highest yield of 2-methylquinoline: all stages of its formation are acid-catalyzed (Scheme 4) and therefore are accelerated at the enhanced concentration of CF_3COOH.

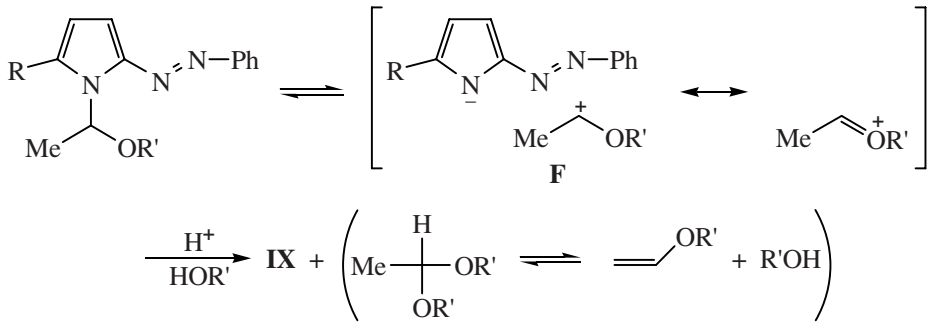
The effect of the reagents structure on the yield and products ratio in the reaction under study is also well consistent with the discussed Schemes. Actually, the key stage of 2-methylquinoline synthesis is apparently the acid-catalyzed adduct decomposition into NH-pyrrole **IX**, acetal (alcoholysis), and vinyl ether (acid-catalyzed thermolysis of acetal, Scheme 2). Consequently, the process would be facilitated by stabilization of cation **F** (i.e., at higher donor effect of the R' radical of the alcohol) and by higher stability of the departing pyrrole anion (i.e., at more electron-withdrawing substituents in the pyrrole ring, Scheme 5); just these trands are observed in experiments.

An alternative scheme of 2-methylquinoline (**VIII**) synthesis (Scheme 6) involves the protonation of azo group, formation of cyclic cation **G** with participation of 1-vinyl substituent, transfer of the vinyl group to the "aniline" nitrogen, prototropic transformation of cation **H** into iminium cation **I**, its addition to 1-vinyl group of the second molecule of 1-vinyl-2-phenylazopyrrole, and finally the closure of the arising cation **J** into tetrahydroquinoline cation **K**. The aromatization of the latter with elimination of 2-phenylazopyrrole and protonated 2-imino-2*H*-pyrrole results in 2-methylquinoline (**VIII**).

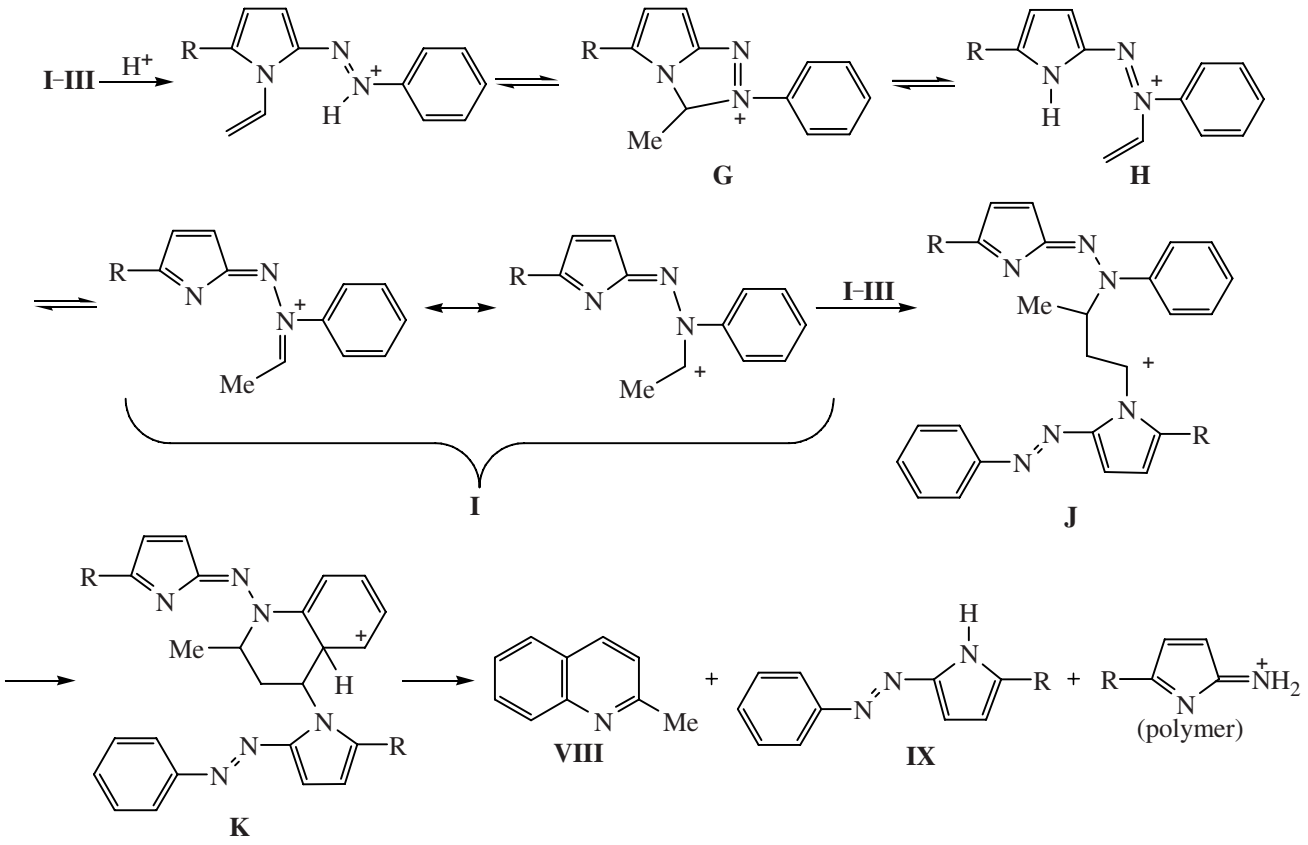
As shown experimentally, at the boiling of 1-vinyl-2-phenylazopyrrole (**I**) for 2 h in benzene with an equimolar quantity of CF_3COOH 2-methylquinoline actually formed (content in the reaction mixture 36%), however in alcoholic environment the reaction occurred predominantly by Scheme 4.

In some instances the reaction mixture contained (by GLC and ^1H NMR data) some aniline (9%), isopropyl acetate (7%) (in reaction with *i*-PrOH), and acetone (5%). These findings suggest an existence of one more alternative or concurrent route of 2-methylquinoline formation: the hydrolysis by water traces of adducts **IV–VII**, acetal and/or vinyl ether into acetaldehyde that is consumed in the reduction of phenylazo group to aniline

Scheme 5.



Scheme 6.



(oxidizing in its turn to acetic acid giving isopropyl acetate with *i*-PrOH). The aniline and acetaldehyde may condense further by Doebner–Miller reaction [17] into 2-methylquinoline. Phenylazo group may be apparently reduced with 2-propanol (and the latter oxidizes into acetone). At the same time the yield of 2-methylquinoline obviously exceeded the quantity of acetaldehyde that might form in reaction with water traces present in the specially dried alcohols used in the study, Therefore if this route exists, it is not the principal one.

EXPERIMENTAL

NMR spectra were registered on a spectrometer Bruker DPX-400 [400.13 (^{1}H), 101.61 MHz (^{13}C)] in $CDCl_3$, internal reference ГМDC. The chemical shifts of ^{15}N (40.53 MHz) were measured with respect to $MeNO_2$. The assignment of signals in ^{1}H and ^{13}C spectra was perfomed with the use of two-dimensional procedures COSY, NOESY, HSQC, and HMBC. IR spectra were recorded from films on a spectrophotometer Bruker

IFS-25. The analysis of reaction mixtures composition was done by GLC on a chromatograph Agilent Technologies 6890N. Initial 1-vinyl-2-phenylazopyrroles **I–III** were synthesized by procedure [13].

General synthetic procedure. In 100 mmol of alcohol was dissolved 1 mmol of 1-vinyl-2-phenyl-azopyrrole **I–III**, CF_3COOH was added (~1% to the weight of compound **I–III**), the mixture was heated at reflux, the acid was added dropwise each hour during the total reaction time. The reaction progress was monitored by GLC. Then the reaction mixture was neutralized with K_2CO_3, the alcohol was distilled off, the residue was treated with ethyl ether (10×2 ml), the extract was evaporated. The crude product obtained from pyrrole **I** and methanol containing solely adduct **IV** was purified by flash-chromatography (eluent hexane). In all other cases the crude product contained a mixture of initial azopyrrole **I–III**, adduct **V–VII**, and 2-methylquinoline (**VIII**), which were separated by column chromatograpy on basic alumina (pH 8.25), gradient elution with hexane–ethyl ether from 1:0 till 0:1.

1-(1-Methoxyethyl)-2-phenylazopyrrole (IV). Yield 0.11 g (49%). Light-yellow viscous fluid, n_D^{20} 1.5125. IR spectrum, cm^{-1}: 3128, 3062, 2990, 2936, 2829, 1643, 1511, 1483, 1455, 1419, 1363, 1327, 1282, 1231, 1209, 1198, 1131, 1097, 1053, 1021, 918, 861, 808, 765, 733, 690, 618, 594, 566, 529, 512, 462. ^{1}H NMR spectrum, δ, ppm: 7.77 m (2H, H_o), 7.43 m (2H, H_m), 7.32 m (1H, H_p), 7.14 d.d (1H, H^5, J_{4-5} 2.7, J_{3-5} 1.5 Hz), 6.71 d.d (1H, H^3, J_{3-4} 4.1, J_{3-5} 1.5 Hz), 6.36 d.d (1H, H^4, J_{4-5} 2.7, J_{3-4} 4.1 Hz), 6.24 q [1H, N–CH, J(CH–Me) 6.1 Hz], 3.22 s (3H, OMe), 1.68 d [3H, Me, J(CH–Me) 6.1 Hz]. ^{13}C NMR spectrum, δ, ppm: 153.5 (C_i), 146.7 (C^2), 129.5 (C_p), 129.0 (C_m), 122.1 (C_o), 121.6 (C^5), 111.3 (C^4), 99.5 (C^3), 82.4 (N–CH), 56.0 (OMe), 22.8 (Me). ^{15}N NMR spectrum, δ, ppm: –203.6 (N^1), 85.3 (N^2), 83.4 (N^3). Found, %: C 68.59; H 6.48; N 18.37. $C_{13}H_{15}N_3O$. Calculated, %: C 68.10; H 6.59; N 18.33.

2-Phenylazo-1-(1-ethoxyethyl)pyrrole (V). Yield 0.08 g (33%). Light-yellow viscous fluid, n_D^{20} 1.5115. IR spectrum, cm^{-1}: 3127, 2976, 2925, 2852, 1545, 1483, 1457, 1421, 1363, 1327, 1299, 1281, 1230, 1199, 1152, 1119, 1093, 1068, 1054, 1019, 948, 917, 849, 806, 764, 731, 690, 618, 595, 567, 525, 512, 494. ^{1}H NMR spectrum, δ, ppm: 7.77 m (2H, H_o), 7.44 m (2H, H_m), 7.34 m (1H, H_p), 7.19 d.d (1H, H^5, J_{4-5} 2.7, J_{3-5} 1.6 Hz), 6.72 d.d (1H, H^3, J_{3-4} 4.1, J_{3-5} 1.6 Hz), 6.38 d.d (1H, H^4, J_{4-5} 2.7, J_{3-4} 4.1 Hz), 6.36 q [1H, N–CH, J(CH–Me) 6.1 Hz], 3.40 m (2H, OCH_2), 1.69 d (3H, Me, J_{CH-Me} 6.1 Hz), 1.13 t [3H, CH_2–Me, J(CH_2–Me) 6.8 Hz]. ^{13}C NMR spectrum, δ, ppm: 153.6 (C_i), 146.6 (C^2), 129.6 (C_p), 129.1 (C_m), 122.1 (C_o), 121.8 (C^5), 111.3 (C^4), 99.6 (C^3), 80.8 (N–CH), 63.9 ($\underline{C}H_2$–Me), 23.1 (Me), 15.0 (CH_2–$\underline{Me}$). ^{15}N NMR spectrum, δ, ppm: –194.9 (N^1), 91.7 (N^2), 89.7 (N^3). Found, %: C 69.60; H 7.44; N 17.61. $C_{14}H_{17}N_3O$. Calculated, %: C 69.11; H 7.04; N 17.27.

1-(1-Isopropoxyethyl)-2-phenylazopyrrole (VI). Yield 0.05 g (20%). Light-yellow viscous fluid, n_D^{20} 1.5100. IR spectrum, cm^{-1}: 3127, 3063, 3031, 2973, 2931, 1510, 1483, 1456, 1421, 1362, 1329, 1300, 1281, 1229, 1199, 1186, 1139, 1121, 1096, 1058, 1036, 1020, 977, 917, 891, 874, 813, 765, 734, 690, 659, 619, 597, 569, 529, 515. ^{1}H NMR spectrum, δ, ppm: 7.76 m (2H, H_o), 7.48 m (2H, H_m), 7.33 m (1H, H_p), 7.20 d.d (1H, H^5, J_{4-5} 2.7, J_{3-5} 1.7 Hz), 6.70 d.d (1H, H^3, J_{3-4} 4.2, J_{3-5} 1.7 Hz), 6.46 q [1H, N–CH, J(CH-Me) 6.1 Hz] 6.35 d.d (1H, H^4, J_{4-5} 2.7, J_{3-4} 4.2 Hz), 3.56 m (1H, C$\underline{H}$–Me_2), 1.67 d [3H, Me, J(CH–Me) 6.1 Hz], 1.18 d, 1.00 d [6H, Me_2, J(CH–Me) 6.1 Hz]. ^{13}C NMR spectrum, δ, ppm: 153.7 (C_i), 146.5 (C^2), 129.6 (C_p), 129.1 (C_m), 122.2 (C_o, C^5), 111.6 (C^4), 99.8 (C^3), 78.4 (N-CH), 69.1 ($\underline{C}$H-Me_2), 23.8 (Me), 23.2, 21.5 (Me_2). ^{15}N NMR spectrum, δ, ppm: –201.5 (N^1), 83.9 (N^2), 79.8 (N^3). Found, %: C 70.41; H 7.74; N 15.91. $C_{15}H_{19}N_3O$. Calculated, %: C 70.01; H 7.44; N 16.33.

1-(1-*tert*-Butoxyethyl)-2-phenylazopyrrole (VII). Yield 0.03 g (12%). Light-yellow viscous fluid, n_D^{20} 1.5050. IR spectrum, cm^{-1}: 3103, 3062, 2923, 2853, 1731, 1599, 1482, 1459, 1365, 1328, 1299, 1283, 1229, 1196, 1156, 1107, 1059, 1037, 1020, 966, 916, 875, 807, 764, 733, 690, 620, 597, 567, 515. ^{1}H NMR spectrum, δ, ppm: 7.79 m (2H, H_o), 7.45 m (2H, H_m), 7.35 m (1H, H_p), 7.25 m (1H, H^5, J_{4-5} 2.7, J_{3-5} 1.3 Hz), 6.69 d.d (1H, H^3, J_{3-4} 4.0, J_{3-5} 1.3 Hz), 6.60 q [1H, N–CH, J(CH–Me) 6.0 Hz], 6.32 d.d (1H, H^4, J_{4-5} 2.7, J_{3-4} 4.0 Hz), 1.59 d [3H, Me, J(CH–Me) 6.0 Hz], 1.14 C (9H, Me_3). ^{13}C NMR spectrum, δ, ppm: 153.7 (C_i), 144.8 (C^2), 129.4 (C_p), 129.1 (C_m), 122.1 (C_o), 122.8 (C^5), 110.9 (C^4), 100.6 (C^3), 75.8 (N–CH), 75.7 ($\underline{C}$–Me_3), 28.1 (Me_3), 25.7 (Me). ^{15}N NMR spectrum, δ, ppm: –196.0 (N^1), 84.0 (N^2), 79.5 (N^3). Found, %: C 71.21; H 8.00; N 15.89. $C_{16}H_{21}N_3O$. Calculated, %: C 70.82; H 7.80; N 15.48.

2-Methyl-6-ethoxyquinoline (XI). In 6.00 g (100 mmol) of 2-propanol was dissolved 0.25 g (1 mmol) of 1-vinyl-5-methyl-2-(4-ethoxyphenylazo)pyrrole (**X**), 0.12 g (1 mmol) of CF_3COOH was added, the mixture was heated at reflux. After 6 h the reaction mixture was neutralized with K_2CO_3, the alcohol was distilled off,

the residue was treated with ethyl ether (10×2 ml), the extract was evaporated. The crude reaction product containing quinoline **XI** was purified from polymer products by flash-chromatography [basic alumina (pH 8.25), eluent hexane]. Yield 0.05 g (26%). Light-yellow fluid, n_D^{20} 1.6090. IR spectrum, cm^{-1}: 3059, 2979, 2921, 2849, 1621, 1602, 1511, 1500, 1474, 1393, 1377, 1344, 1308, 1234, 1173, 1155, 1113, 1045, 968, 941, 853, 828, 770, 641. ^1H NMR spectrum, δ, ppm: 7.90 d (1H, H^4, J_{3-4} 8.3 Hz), 7.88 d (1H, H^8, J_{7-8} 9.0 Hz), 7.31 d.d (1H, H^7, J_{7-8} 9.0, J_{6-7} 2.7 Hz), 7.20 d (1H, H^3, J_{3-4} 8.3 Hz), 7.00 d (1H, H^5, J_{5-6} 2.7 Hz), 4.11 q [2H, CH_2, J(CH_2–Me) 7.1 Hz], 2.68 s (3H, Me), 1.46 s (3H, CH_2–Me). ^{13}C NMR spectrum, δ, ppm: 156.6 (C^6), 156.3 (C^2), 143.9 (C^{10}), 135.1 (C^4), 130.1 (C^8), 127.4 (C^9), 122.2 ($C^{3,7}$), 106.0 (C^5), 63.8 (CH_2), 25.1 (Me), 14.9 (CH_2–Me). Found, %: C 77.22; H 6.89; N 7.87. $C_{12}H_{13}NO$. Calculated, %: C 76.98; H 7.00; N 7.48.

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