Russian Chemical Bulletin, International Edition, Vol. 58, No. 1, pp. 115—118, January, 2009

Synthesis and polymerization of sterically hindered 2,5-diphenyl- and 2,3,5-triphenyl-1-vinylpyrroles*

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Sterically hindered 2,5-diphenyl- and 2,3,5-triphenyl-1-vinylpyrroles have been obtained by the vinylation of the corresponding NH-pyrroles with acetylene in superbasic catalytic system KOH—DMSO in up to 78% yield. 2,3,5-Triphenyl-1-vinylpyrrole has also been obtained in 75% yield by the regioselective bromination of 2,3-diphenyl-1-vinylpyrrole with subsequent cross-coupling of 5-bromo-2,3-diphenyl-1-vinylpyrrole with phenylmagnesium bromide in the presence of dichloro[1,1′-bis(diphenylphosphino)ferrocene]palladium(II). 2,5-Diphenyl- and 2,3,5-triphenyl-1-vinylpyrroles undergo a free-radical polymerization (AIBN, 80 °C) to form oligomers in 11 and 27% yield, respectively.

Key words: pyrroles, 2,5-diphenyl-1-vinylpyrrole, 2,3,5-triphenyl-1-vinylpyrrole, vinylation, cross-coupling, polymerization.

In the last decades, a growing interest to pyrroles is cased by their wide synthetic possibilities and enormous biological and technical potential.[1—4] A variety of medicines, including antitumor, are obtained on the basis of pyrroles.[5,6] Pyrroles are also the basis for development of light-emitting diodes, electroconducting coatings,[7] they are used as components for modern rechargeable lithium batteries.[8]

The presence of a vinyl group in the structure of pyrrole derivatives increases their synthetic potential, allows one to obtain unique polymers: electroconducting materials and organic metals for the processes of transformation of the solar energy and recording of information,[9—11] sorbents for noble metals,[12] biologically active compounds and plant protection substances,[11,13] modificators of polymers and polymeric azo dyes.[14,15]

Due to the discovery and systematic development of the reaction of ketoximes with acetylene in superbasic media (the Trofimov reaction), a possibility to synthesize a wide range of substituted pyrroles in one preparative stage has been appeared.[1—3]

Vinylation of phenylpyrroles with acetylene proceeds in the catalytic system KOH—DMSO at both high and atmospheric pressure.[1,16—18] In the latter case (100 °C, 5 h), 2,5-diphenyl-1-vinylpyrrole is formed only in the trace amounts (2—3%), whereas vinylation of 2,3-diphenylpyrrole under the same conditions leads to 2,3-diphenyl-1-vinylpyrrole in 35% yield (see Ref. 18). Conducting the reaction under pressure of acetylene (the initial pressure of 10—12 atm) allows one to increase the yield of 2,3-diphenyl-1-vinylpyrrole up to 72% (100 °C, 3 h).[16,17]

The present work deals with the synthesis of 2,5-diphenyl- and 2,3,5-triphenyl-1-vinylpyrroles and their polymerization.

Experiments showed that, in contrast to 2,3-diphenylpyrrole, 2,5-diphenylpyrrole (**1**) is vinylated under more drastic conditions: 2,5-diphenyl-1-vinylpyrrole (**2**) is not detected (^{1}H NMR) in the reaction medium after 3 h at 90—110 °C (the initial pressure of acetylene was 11—12 atm). An increase in the temperature up to 140 °C (the initial pressure of acetylene was 11—12 atm, 3 h) allowed us to obtain vinylpyrrole **2** in 20% yield (Scheme 1). And only on conducting the reaction at 150 °C (the initial pressure of acetylene was 11—12 atm, 3 h), the yield of vinylpyrrole **2** reached 78%.

The low vinylation rate of 2,5-diphenylpyrrole (**1**), obviously, results from the strong steric hindrance of the anionic center on the nitrogen atom by two phenyl substituents.

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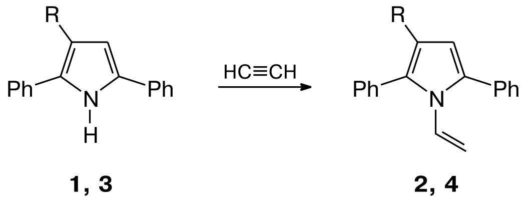
* Dedicated to Academician A. I. Konovalov on his 75th birthday.

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 1, pp. 115—118, January, 2009.

Scheme 1



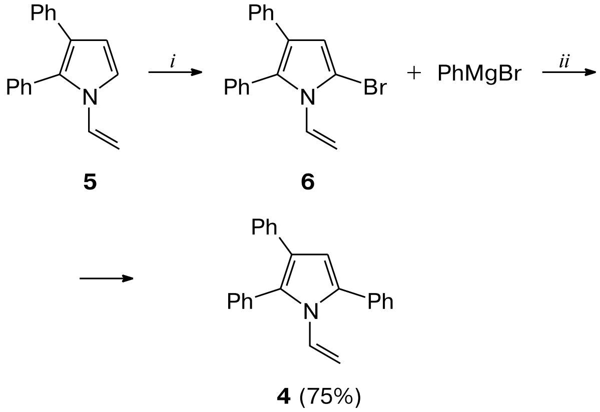
R = H (**1**, **2**), Ph (**3**, **4**)

Conditions: KOH—DMSO, 150 °C.

Under comparable conditions (150 °C, the initial pressure of acetylene of 11—12 atm, 4 h), 2,3,5-triphenylpyrrole (**3**) reacts with acetylene to form 2,3,5-triphenyl-1-vinylpyrrole (**4**) (77% yield).

2,3,5-Triphenyl-1-vinylpyrrole (**4**) was also obtained by the bromination of 2,3-diphenyl-1-vinylpyrrole (**5**) with *N*-bromosuccinimide[19] with subsequent Pd-catalyzed cross-coupling[20] of thus obtained 5-bromo-2,3-diphenyl-1-vinylpyrrole (**6**) with phenylmagnesium bromide (Scheme 2).

Scheme 2



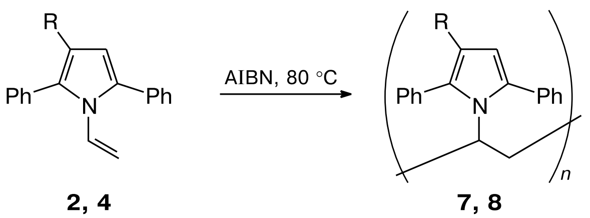
Reagents and conditions: *i*. *N*-bromosuccinimide (NBS), THF, 8—12 °C; *ii*. dichloro[1,1´-bis(diphenylphosphino)ferrocene]-palladium(II) ($PdCl_2$•dppf) (1 mol.%), THF, 60—63 °C.

It was found that 1-vinylphenylpyrroles **2** and **4** can polymerize in the presence of 2,2´-azo-bis(isobutyronitrile) (AIBN) with the formation of oligomers **7** and **8** (molecular mass 1400 and 1500, respectively, Scheme 3), well soluble in organic solvents (benzene, acetone, chloroform).

According to the IR and ^{1}H NMR spectroscopic data, the oligomers synthesized have the structure shown in Scheme 3.

In the IR spectra of polymers **7** and **8**, the absorption bands of the vinyl group (1640, 849, 577 cm^{-1})[1,21] are absent and the bands of stretching vibrations of the CH_2 and CH groups of the polymeric chain (2961, 2927, 2869 cm^{-1}) are present. The absorption bands of benzene and pyrrole rings remain in the IR spectra.

Scheme 3



R = H (**2**, **7**), Ph (**4**, **8**)

As it is characteristic of polymers, all the signals are broadened in the ^{1}H NMR spectra. The signals for the protons of the vinyl group (4.46—4.58, 4.70—4.80, and 6.67—6.81 ppm) disappear from them and the broad signals for the protons of the CH and CH_2 groups of the polymeric chain appear in the regions 3.73—4.27 and 2.13—3.17 ppm. The protons of the benzene rings resonate as a wide signal with the maximum at 7.25 ppm for **7** and 7.09 ppm for **8**, the signals for the protons of the pyrrole ring are observed at 6.05 and 6.50 ppm for compounds **7** and **8**, respectively.

2,5-Diphenyl- (**2**) and 2,3,5-triphenyl-1-vinylpyrroles (**4**) under study are characterized by low activity in the radical polymerization. Thus, the yield of poly(2,5-diphenyl-1-vinylpyrrole) (**7**) on polymerization in benzene (the ratio monomer : benzene was 2 : 1, 80 °C, 160 h) is only 2%. In the absence of benzene (polymerization in the neat mass), the yield of polymer **7** did not exceed 6%. The use of special method, *viz*., the addition of an initiator into the melted monomer, allowed us to raise the yield up to 11%.

Poly(2,3,5-triphenyl-1-vinylpyrrole) (**8**) was obtained under similar conditions in 27% yield for 115 h (Fig. 1).

The reduced activity of 2,5-diphenyl-1-vinylpyrrole in the polymerization, obviously, is caused by the limited

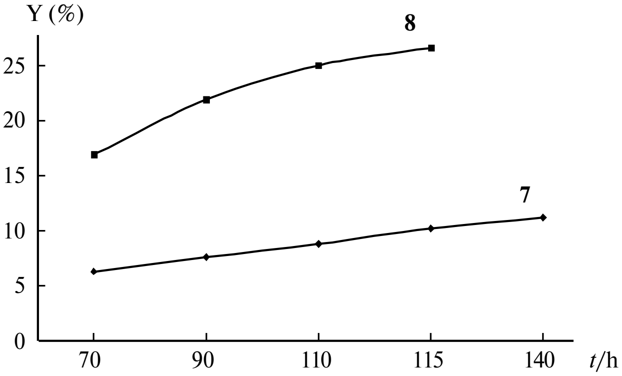


Fig. 1. Yields (*Y*) of poly(2,5-diphenyl-1-vinylpyrrole) (**7**) and poly(2,3,5-triphenyl-1-vinylpyrrole) (**8**) versus polymerization time.

accessibility of the monomer to the growing radical shielded by the phenyl groups.

The more than two-fold increase in the yield of polymer **8** in comparison with polymer **7**, apparently, is caused by the positive steric effect, namely, by the increase in the rotation angle of the phenyl substituent at position 2 with respect to the pyrrole ring due to the steric interaction with the phenyl substituent at position 3. This should lead to the steric deshielding of the *N*-vinyl group.

The formation of oligomers characteristic of 1-vinylphenylpyrroles **2** and **4** under study, as in the case of other 1-vinylpyrroles, probably, should be referred to the processes of the chain transfer on a monomer.[1,22]

In conclusion, we found conditions for the vinylation of sterically hindered 2,5-diphenyl- and 2,3,5-triphenylpyrroles with acetylene. Alternatively 2,3,5-triphenyl-1-vinylpyrrole was prepared by bromination of 2,3-diphenyl-1-vinylpyrrole with subsequent cross-coupling of 5-bromo-2,3-diphenyl-1-vinylpyrrole with phenylmagnesium bromide. The possibility to obtain oligomers of 2,5-diphenyl- and 2,3,5-triphenyl-1-vinylpyrroles under conditions of radical polymerization was demonstrated.

Experimental

IR spectra of compounds synthesized in the region 400—4000 cm^{-1} were recorded in KBr pellets on a Bruker IFS-25 spectrometer. NMR spectra were recorded on a Bruker DPX 400 spectrometer (400.13 (^{1}H) MHz, 100.6 MHz (^{13}C)); $CDCl_3$ was the solvent, HMDS was the internal standard. 2,3-Diphenyl-1-vinylpyrrole (**5**) was synthesized according to the procedure described earlier,[17] the catalyst $PdCl_2 \cdot$dppf was obtained according to the known procedure.[23]

Polymerization of 2,5-diphenyl-1-vinylpyrrole and 2,3,5-triphenyl-1-vinylpyrrole was carried out under argon in the presence of 2,2´-azo-bis(isobutyronitrile) (AIBN), which was recrystallized twice from methanol. Solvents (benzene, hexane) were purified by known procedures.[24]

Molecular mass of poly(2,5-diphenyl-1-vinylpyrrole) (**7**) was determined on a Waters gel chromatograph with the channel refractometric detector at 25 °C with THF being the eluent. Molecular mass of poly(2,3,5-triphenyl-1-vinylpyrrole) (**8**) was determined by the isopiestic method in benzene at 60 °C with azobenzene being the standard.[25]

2,5-Diphenyl-1-vinylpyrrole (2). 2,5-Diphenylpyrrole (**1**) (2.0 g, 9.1 mmol), KOH (0.60 g, 30 wt%, 10.7 mmol), and DMSO (50 mL) were placed in a 0.25 L rotary steel autoclave, acetylene was passed through until saturation, and the mixture was heated for 3 h at 150 °C. After cooling, the reaction mixture was diluted with water (250 mL) and extracted with hexane (5×50 mL). The extracts were washed with water (2×70 mL), and dried with K_2CO_3. After the solvent was evaporated, the residue was distilled *in vacuo* ($7 \cdot 10^{-2}$ Torr, 142—162 °C) to obtain 2,5-diphenyl-1-vinylpyrrole (**2**) (1.74 g, 78%), beige crystals, m.p. 80—81 °C. Found (%): C, 88.20; H, 6.08; N, 5.69. $C_{18}H_{15}N$. Calculated (%): C, 88.13; H, 6.16; N, 5.71. IR, ν/cm^{-1}: 3049, 1947, 1882, 1813, 1641, 1600, 1548, 1486, 1448, 1407, 1388, 1324, 1297, 1239, 1122, 1075, 968, 915, 896, 849, 838, 781, 765, 754, 729, 697, 653, 577. ^{1}H NMR, δ: 7.42 (m, 4 H, Ph); 7.36 (m, 4 H, Ph); 7.28 (m, 2 H, Ph); 6.81 (dd, 1 H, H_x, J_{H_a,H_x} = 8.8 Hz, J_{H_b,H_x} = 15.9 Hz); 6.31 (s, 2 H, H(3), H(4)); 4.81 (d, 1 H, H_a, J_{H_a,H_x} = 8.8 Hz); 4.58 (d, 1 H, H_b, J_{H_b,H_x} = 15.9 Hz). ^{13}C NMR, δ: 135.4, 133.6, 131.5, 129.4, 128.2, 127.0, 110.9, 109.5.

2,3,5-Triphenyl-1-vinylpyrrole (4). ***A.*** 2,3,5-Triphenylpyrrole (**3**) (2.1 g, 7.1 mmol), KOH (0.63 g, 30 wt%, 11.2 mmol), and DMSO (50 mL) were placed in a 0.25 L rotary steel autoclave, acetylene was passed through until saturation, and the mixture was heated for 4 h at 150 °C. After cooling, the reaction mixture was extracted with hexane (5×50 mL), the extracts were washed with water (2×70 mL), and dried with $MgSO_4$. After the solvent was evaporated, the product was isolated by column chromatography (Al_2O_3, hexane) to obtain 2,3,5-triphenyl-1-vinylpyrrole (**4**) (1.76 g, 77%), white crystals, m.p. 76—78 °C. Found (%): C, 89.86; H, 5.89; N, 4.20. $C_{24}H_{19}N$. Calculated (%): C, 89.68; H, 5.96; N, 4.36. IR, ν/cm^{-1}: 3057, 3030, 1950, 1888, 1813, 1640, 1603, 1503, 1485, 1468, 1406, 1388, 1358, 1298, 1115, 1072, 963, 913, 762, 696. ^{1}H NMR, δ: 7.49 (m, 2 H, Ph); 7.38—7.30 (m, 7 H, Ph); 7.17 (m, 5 H, Ph); 7.09 (m, 1 H, Ph); 6.67 (dd, 1 H, H_x, J_{H_a,H_x} = 8.8 Hz, J_{H_b,H_x} = 15.8 Hz); 6.50 (s, 1 H, H(4)); 4.70 (d, 1 H, H_a, J_{H_a,H_x} = 8.8 Hz); 4.46 (d, 1 H, H_b, J_{H_b,H_x} = 15.8 Hz). ^{13}C NMR, δ: 136.6, 135.1, 134.1, 133.7, 132.5,132.1, 130.1, 129.2, 129.1, 128.9, 128.5, 127.9, 126.4, 124.9, 112.1, 109.7.

B. Dichloro[1,1´-bis(diphenylphosphino)ferrocene]palladium(II) (0.042 g, 1 mol.%, $5.7 \cdot 10^{-5}$ mmol) and a solution of PhMgBr in THF (1 *M*, 13.2 mL) were sequentially added to a solution of 5-bromo-2,3-diphenyl-1-vinylpyrrole (**6**) (1.85 g, 5.7 mmol) in THF (5 mL) under argon and the mixture was heated for 3 h (60—63 °C). After cooling, the reaction mixture was diluted with water (60 mL), extracted with ether (3×20 mL), and dried with Na_2SO_4. After the solvent was evaporated, the product was isolated by column chromatography (Al_2O_3, hexane) to obtain 2,3,5-triphenyl-1-vinylpyrrole (**4**) (1.37 g, 75%).

5-Bromo-2,3-diphenyl-1-vinylpyrrole (6). *N*-Bromosuccinimide (NBS) (1.07 g, 6.0 mmol) was added to a solution of 2,3-diphenyl-1-vinylpyrrole (**5**) (1.23 g, 5.0 mmol) in THF (20 mL) cooled to 10—12 °C, the mixture was stirred until complete dissolution (10—15 min) and kept in refrigerator (8—10 °C) for 18 h. After that, sodium sulfate (1.0 g) was added to the mixture, the solvent was evaporated under reduced pressure, the residue was diluted with CCl_4 (5 mL), a precipitate was filtered off and washed with CCl_4 (3 mL). After evaporation of the solvent, the residue was passed through a layer of Al_2O_3 (light petroleum) to obtain 5-bromo-2,3-diphenyl-1-vinylpyrrole (**6**) (1.06 g, 65%), beige crystals, m.p. 70—71 °C. Found (%): C, 66.46; H, 4.25; Br, 23.98; N, 4.38. $C_{18}H_{14}BrN$. Calculated (%): C, 66.68; H, 4.35; Br, 24.65; N, 4.32. IR, ν/cm^{-1}: 3081, 3048, 2923, 2851, 1957, 1888, 1636, 1600, 1489, 1459, 1447, 1406, 1351, 1319, 1294, 1179, 1069, 1029, 958, 916, 896, 786, 761, 739, 697, 576. ^{1}H NMR, δ: 7.40 (m, 3 H, Ph); 7.24 (m, 2 H, Ph); 7.15 (m, 2 H, Ph); 7.10 (m, 3 H, Ph); 6.62 (dd, 1 H, H_x, J_{H_a,H_x} = 9.0 Hz, J_{H_b,H_x} = 15.6 Hz); 6.51 (s, 1 H, H(4)); 4.98 (d, 1 H, H_a, J_{H_a,H_x} = 9.0 Hz); 4.96 (d, 1 H, H_b, J_{H_b,H_x} = 15.6 Hz). ^{13}C NMR, δ: 135.9, 133.1, 132.2, 132.0, 131.6, 129.3, 129.0, 128.9, 128.8, 126.8, 113.8, 111.4.

Polymerization (general procedure). 2,2´-Azo-bis(isobutyronitrile) (5 wt%) was added to the melted monomer **2** or **4** with stirring and the mixture was incubated at 80 °C. The polymers **7**

and **8** obtained were purified by reprecipitation from benzene with hexane and dried *in vacuo* until the weight is constant.

Poly(2,5-diphenyl-1-vinylpyrrole) (7). Polymer **7** (0.28 g, 11%) was obtained from 2,5-diphenyl-1-vinylpyrrole (**2**) (2.54 g, 10.4 mmol) in the presence of AIBN (0.127 g, 0.77 mmol) after 140 h, white powder, m.p. 136—142 °C, molecular mass 1400. Found (%): C, 88.63; H, 5.96; N, 5.38. $C_{18}H_{15}N$. Calculated (%): C, 88.13; H, 6.16, N, 5.71. IR, ν/cm^{-1}: 3056, 3026, 2964, 2927, 2864, 1602, 1575, 1485, 1465, 1445, 1374, 1313, 1222, 1202, 1176, 1156, 1071, 1027, 968, 915, 757, 701, 667. ^1H NMR, δ: 7.26—7.11 (Ph); 6.01 (H(3), H(4)); 4.27 (N—CH); 2.12 (CH_2).

Poly(2,3,5-triphenyl-1-vinylpyrrole) (8). Polymer **8** (0.66 g, 27%) was obtained from 2,3,5-triphenyl-1-vinylpyrrole (**4**) (2.44 g, 7.6 mmol) in the presence of AIBN (0.122 g, 0.75 mmol) after 115 h, beige powder, m.p. 244—254 °C, molecular mass 1500. Found (%): C, 89.78; H, 5.90; N, 4.30. $C_{24}H_{19}N$. Calculated (%): C, 89.68; H, 5.96; N, 4.36. IR, ν/cm^{-1}: 3054, 2964, 2926, 2867, 1601, 1501, 1485, 1466, 1447, 1406, 1389, 1298, 1277, 1115, 1072, 913, 760, 696. ^1H NMR, δ: 7.49—7.09 (Ph); 6.50 (H(4)); 3.73 (N—CH); 3.17 (CH_2).

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Received March 12, 2008;
in revised form November 6, 2008