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SHORT COMMUNICATIONS

Synthesis of *push-pull* Chromophores Including an *N*-[4-(4-Methoxyphenoxy)butyl]carbazole Fragment

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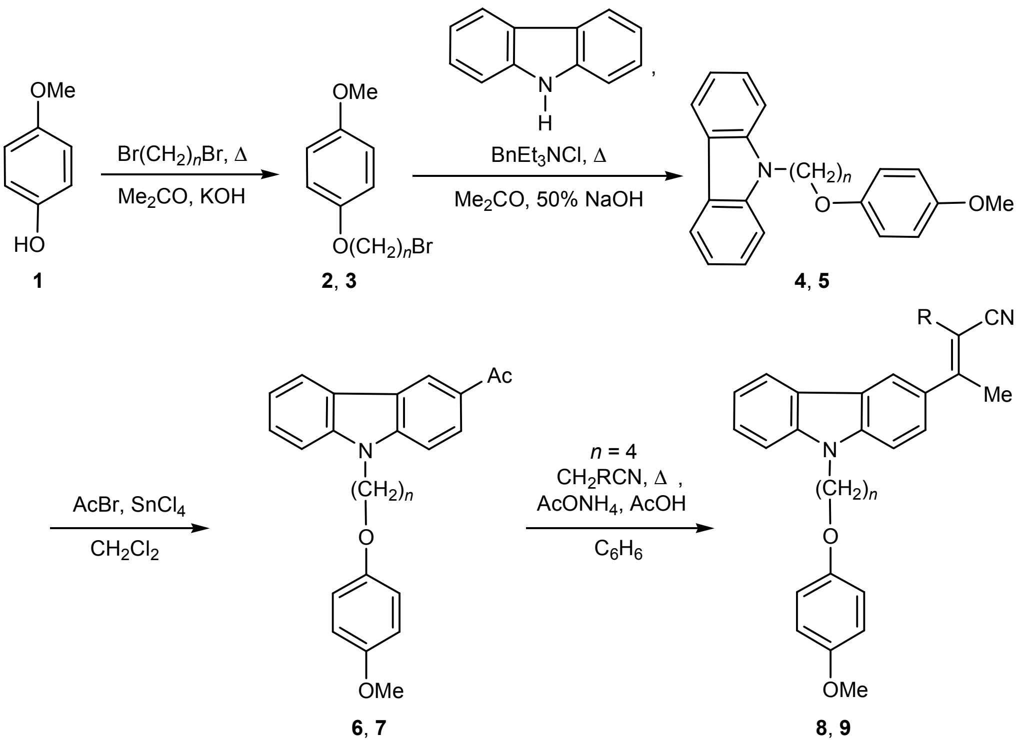
Organic substances with nonlinear optical properties of the second order are the promising materials for molecular electronics and photonics, that is, finally, for telecommunication and electro optical devices [1–6]. These materials possess some advantages if compared with their inorganic analogs: they have high values of nonresonance susceptibility, high nonlinear optic response, high transmissibility, low dielectric permittivity, besides they are sufficiently easy to process and economic [1, 7]. However certain drawbacks in their practical application also exist, e.g., the strong intermolecular electrostatic interaction, poor solubility in common organic solvents, thermal and photochemical instability. By altering the chemical structure of material, namely, by selecting the optimum electron-donor and electron-acceptor substituents it is possible to obtain the maximum nonlinearity on the molecular level and also affect the chemical, photochemical, and thermal stability. The introduction of electron-donor *ortho*- or *para*-alkoxyphenyl groups into the monomers is known to change the optical properties of chromophores. It results in increasing of the electro optic coefficient value (r_{33}) and molecular hyperpolarizability β [8–10]. When such a group is situated in the peripheral part of a long aliphatic chain and is incorporated into the chromophore structure both intermolecular and intramolecular interaction may occur between the donor and acceptor parts of the chromophore. Besides, the introduction of long-chain substituents in the structure of compounds affects the character of their packing in the solid state and their thermal stability [11].

In this work we continued to study carbazole-containing compounds including in their structure ω-(4-methoxyphenyl)alkyl substituents at the nitrogen atom of this heterocycle [12, 13] and have synthesized two new chromophores **8** and **9** including *N*-[4-(4-methoxyphenoxy)butyl]carbazole moiety as a donor fragment. The optical and electrochemical properties of all prepared during thes work compounds were examined. First from the corresponding α,ω-dibromoalkane and *p*-methoxyphenol **1** in acetone the starting bromides **2** and **3** were prepared [12]. The subsequent alkylation of carbazole with the synthesized bromides under the conditions of the phase-transfer catalysis in the system acetone–16 M water solution of NaOH–benzyltriethylammonium chloride led to the formation of the corresponding *N*-substituted carbazoles **4** and **5** [13]. The acetylation of the latter at room temperature in dichloroethane in the presence of $SnCl_4$ afforded 3-acetylcarbazoles **6** and **7**.

Further under the Knoevenagel condensation conditions acetylcarbazole **6** was treated with malonodinitrile or ethyl cyanoacetate in benzene media in the presence of a catalytic amount of ammonium acetate to obtain chromophores **8** and **9** – yellow viscous oils, well soluble in alcohols, alkanes, and haloalkanes.

UV and fluorescence spectra of compounds **6**–**9** were measured in chloroform. From the data of absorption and emission spectra the Stokes shifts were estimated (Δλ): 110 (**6**), 98 (**7**), 121 (**8**), 153 (**9**) nm. On the basis of the longest absorption wavelength value (λ_{onset}) the width of the forbidden bandgap (E_g^{opt})



n = 4 (**2**, **4**, **6**, **8**, **9**), 6 (**3**, **5**, **7**); R = CN (**8**), COOEt (**9**).

was calculated [14]: 3.02 (6), 3.18 (7), 2.61 (8), 2.69 (9) eV.

The electrochemical properties of compounds synthesized were explored by cyclic voltammetry. The cyclic voltammograms of compound **4** exhibit three oxidation peaks are present, at 910 (E^1_{ox}), 1235 (E^2_{ox}), and 1330 (E^3_{ox}) mV. Two reduction waves at 885 and 1140 mV (E^1_{red} and E^2_{red}) correspond to the first and to the second oxidation potentials – E^1_{ox} and E^2_{ox}. Analogous pattern was observed for compound **5**. Three oxidation peaks are clearly seen (1020, 1215 and 1320 mV), but in contrast to compound **4** only one reduction peak is pbserved at 1095 mV (E^1_{red}) which corresponds to E^1_{ox}. In both cases at each succeeding cycle the current value increases, and on the surface of the working electrode grows the blue film of the respective polymer [13].

The incorporation of electron-acceptor withdrawing groups into the structure of carbazoles **4** and **5** changes the pattern of the electrochemical oxidation and leads

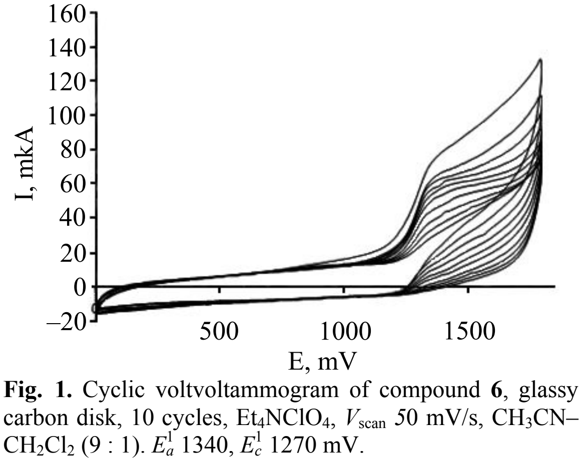


Fig. 1. Cyclic voltvoltammogram of compound **6**, glassy carbon disk, 10 cycles, Et_4NClO_4, V_{scan} 50 mV/s, CH_3CN–CH_2Cl_2 (9 : 1). E_a^1 1340, E_c^1 1270 mV.

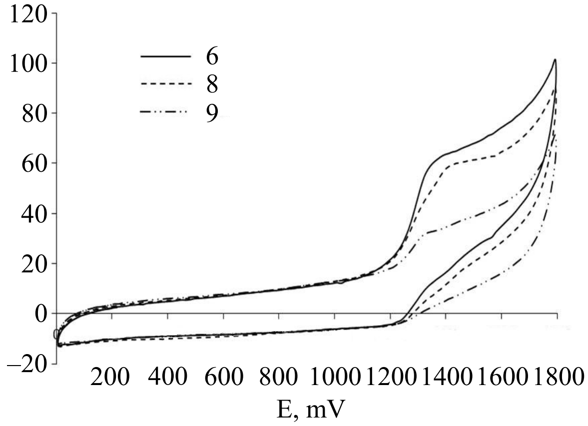


Fig. 2. Cyclic voltvoltammogram of compound **6**, **8**, **9**, glassy carbon disk, Et_4NClO_4, V_{scan} 50 mV/s, CH_3CN–CH_2Cl_2 (9 : 1). 6: E_a^1 1340, E_c^1 1270 mV. 8: E_a^1 1295, E_a^2 1380, E_c^1 1265 mV. 9: E_a^1 1295, E_c^1 1265 mV.

to the growth of the values of redox potentials. The only one clear reversible oxidation peak was observed in the cyclic voltammograms of compounds **6**, **8** and **9** at 1340 (**6**), 1295 (**8**), and 1295 (**9**) mV; the corresponding reduction peaks had blurred forms and appeared in the region 1260–1270 mV. The replacement of the carbonyl group by a vinyl fragment containing two strong electron-acceptor substituents results in a slight decrease in the oxidation potentials. In all cases at each succeeding cycle the current value increases, and on the surface of the working electrode grows the blue film of the respective polymer, consequently, the polymerization occurs at the positions *2* and *7* of the carbazole ring [15]. Examples of cyclic voltammograms of chromophores **6**, **8** and **9** are presented in Figs. 1, 2 (working electrode – glassy carbon disk).

1-(ω-Bromoalkoxy)-4-methoxybenzenes (2 and 3). To a solution of 0.1 mol of *p*-methoxyphenol **1** in 200 mL of acetone was added 6.72 g (0.12 mol) of KOH, after its dissolution 23.7 mL (0.2 mol) of 1,4-dibromobutane or 30.8 mL (0.2 mol) of 1,6-dibro-mohexabe was added dropwise, the mixture was refluxed for 1 h, cooled, the resulted precipitate (products of dimerization) was filtered off. The filtrate was evaporated, the residue was recrystallized from EtOH.

1-(4-Bromobutoxy)-4-methoxybenzene (2). Yield 68%, mp 43–45°C (46–47°C [16, 17]). ^{1}H NMR spectrum, δ, ppm: 1.90 quintet (2H, $C\underline{H}_2CH_2Br$, *J* 6.6 Hz), 2.05 quintet (2H, $OCH_2C\underline{H}_2$, *J* 6.3 Hz), 3.47 t (2H, $C\underline{H}_2Br$, *J* 6.3 Hz), 3.75 s (3H, OMe), 3.93 t (2H, $OC\underline{H}_2$, *J* 6.3 Hz), 6.81 s (4H, C_6H_4).

1-[6-Bromohexyloxy]-4-methoxybenzene (3). Yield 65%, mp 48–49°C [18]. ^{1}H NMR spectrum, δ, ppm: 1.43–1.47 m (4H, $OCH_2CH_2C\underline{H}_2C\underline{H}_2CH_2CH_2\cdot Br$), 1.72 quintet (2H, $C\underline{H}_2CH_2Br$, *J* 6.6 Hz), 1.87 quintet (2H, $OCH_2C\underline{H}_2$, *J* 6.3 Hz), 3.40 t (2H, $C\underline{H}_2Br$, *J* 6.9 Hz), 3.74 s (3H, OMe), 3.83 t (2H, $OC\underline{H}_2$, *J* 6.3 Hz), 6.76 s (4H, C_6H_4).

9-[ω-(4-Methoxyphenoxy)alkyl]-9*H*-carbazoles (4 and 5). To a solution of 0.024 mol of carbazole and 0.024 mol of bromoalkoxybenzene **2** or **3** in 80 mL of acetone was added 0.02 g of $BnEt_3NCl$ and 20 mL of 16 M water solution of NaOH, the mixture was refluxed for 12 h, cooled, the organic layer was separated, poured into 200 mL of water, and extracted with dichloromethane (3 × 50 mL). The combined organic extracts were washed with water (2 × 100 mL), dried with $MgSO_4$. The solvent was removed, the residue was recrystallized from methanol; the purification by column chromatography on silica gel was also possible (eluent acetone–hexane, 1 : 2).

9-[4-(4-Methoxyphenoxy)butyl]-9*H*-carbazole (4). Yield 75%, mp 71–72°C (70–72°C [13]). ^{1}H NMR spectrum, δ, ppm: 1.81 quintet (2H, $C\underline{H}_2CH_2N$, *J* 6.3 Hz), 2.07 quintet (2H, $OCH_2C\underline{H}_2$, *J* 6.9 Hz), 3.74 s (3H, OMe), 3.88 t (2H, $NC\underline{H}_2$, *J* 6.3 Hz), 4.38 t (2H, $OC\underline{H}_2$, *J* 6.9 Hz), 6.79 s (4H, C_6H_4), 7.23 d (2H, carbazole, *J* 6.3 Hz), 7.39–7.48 m (4H, carbazole), 8.09 d (2H, carbazole, *J* 7.8 Hz).

9-[6-(4-Methoxyphenoxy)hexyl]-9*H*-carbazole (5). Yield 70%, mp 69–70°C (70–71°C [13]). ^{1}H NMR spectrum, δ, ppm: 1.35–1.39 m (4H, $OCH_2CH_2\cdot C\underline{H}_2C\underline{H}_2CH_2CH_2N$), 1.60 quintet (2H, $C\underline{H}_2CH_2N$, *J* 6.9 Hz), 1.79 quintet (2H, $OCH_2C\underline{H}_2$, *J* 6.9 Hz), 3.67 s (3H, OMe), 3.81 t (2H, NCH_2, *J* 6.3 Hz), 4.39 t (2H, OCH_2, *J* 6.9 Hz), 6.79 s (4H, C_6H_4), 7.18 t (2H, carbazole, *J* 7.5 Hz), 7.44 t (2H, carbazole, *J* 7.5 Hz), 7.59 d (2H, carbazole, *J* 8.1 Hz), 8.14 (2H, carbazole, *J* 7.5 Hz).

Carbazoles (6 and 7). To a solution of 0.03 mol of carbazole **4** or **5** in 60 mL of anhydrous CH_2Cl_2 was added 7.0 mL (0.06 mol) of $SnCl_4$, the mixture was stirred for 10 min, then was added dropwise 2.2 mL (0.03 mol) of acetyl bromide. The reaction mixture was stirred for 24 h at room temperature, poured in the mixture of ice and hydrochloric acid, extracted with dichloromethane, the combined organic solutions were washed with water till neutral reaction, and evaporated. The solid residue was purified by column chromatography on silica gel (eluent dichloromethane–hexane, 1 : 1).

3-Acetyl-9-[4-(4-methoxyphenoxy)butyl]-9*H*-carbazole (6). Yield 85%, gray crystals, mp 77–78°C. IR spectrum, ν, cm^{-1}: 1626 (C=O). UV absorption spectrum, λ_{max}, nm (log ε): 322 (3.79), 330 (3.78); fluorescence spectrum, λ_{max}, nm: 388, 424, 440. Stokes shift Δλ 110 nm. ^{1}H NMR spectrum, δ, ppm: 1.81 quintet (2H, $C\underline{H}_2CH_2N$, *J* 6.3 Hz), 2.08 quintet (2H, $OCH_2C\underline{H}_2$, *J* 7.2 Hz), 2.71 s (3H, $COCH_3$), 3.74 s (3H, OMe), 3.89 t (2H, NCH_2, *J* 6.0 Hz), 4.40 t (2H, OCH_2, *J* 7.2 Hz), 6.78 s (4H, C_6H_4), 7.29 t (1H, carbazole, *J* 7.8 Hz), 7.39–7.53 m (3H, carbazole), 8.10 d (1H, carbazole, *J* 6.9 Hz), 8.14 d (1H, carbazole, *J* 7.8 Hz), 8.73 s (1H, carbazole). Mass spectrum, *m/z* (I_{rel}, %): 388 (8.1) $[M + H]^+$, 387 (28.3) $[M]^+$, 265 (13.8), 264 (67.9), 223 (16.1), 222 (100), 180 (24.9), 179 (23.2), 43 (16.1). Found, %: C 77.38; H 6.55; N 3.51. $C_{25}H_{25}NO_3$. Calculated, %: C 77.49; H 6.50; N 3.61. *M* 387.48.

3-Acetyl-9-[6-(4-methoxyphenoxy)hexyl]-9*H*-carbazole (7). Yield 80%, gray crystalline substance, mp 88–89°C. UV absorption spectrum, λ_{max}, nm (log ε): 320 (3.69), 330 (3.67); fluorescence spectrum, λ_{max}, nm: 378, 410, 428. Stokes shift Δλ 98 nm. ^{1}H NMR spectrum, δ, ppm: 1.45–1.49 m (4H, $OCH_2CH_2C\underline{H_2}C\underline{H_2}\cdot CH_2CH_2N$), 1.69 quintet (2H, $C\underline{H_2}CH_2N$, *J* 6.3 Hz), 1.91 quintet (2H, $OCH_2C\underline{H_2}$, *J* 7.2 Hz), 2.72 s (3H, $COCH_3$), 3.75 s (3H, OMe), 3.84 t (2H, NCH_2, *J* 6.3 Hz), 4.33 t (2H, OCH_2, *J* 6.9 Hz), 6.79 s (4H, C_6H_4), 7.29 t (1H, carbazole, *J* 7.2 Hz), 7.38–7.51 m (3H, carbazole), 8.11 d (1H, carbazole, *J* 8.4 Hz), 8.15 d (1H, carbazole, *J* 7.8 Hz), 8.74 s (1H, carbazole). Mass spectrum, *m/z* (I_{rel}, %): 416 (15.4) $[M + H]^+$, 415 (48.9) $[M]^+$, 223 (17.1), 222 (100), 180 (23.5), 179 (23.4), 124 (13.5), 43 (11.6). Found, %: C 77.87; H 6.98; N 3.21. $C_{27}H_{29}NO_3$. Calculated, %: C 78.04; H 7.03; N 3.37. *M* 415.52.

Chromophores (8 and 9). General procedure. A mixture of 5 mmol of carbazole **6**, 0.5 mL (5 mmol) of ethyl cyanoacetate or 0.3 g (5 mmol) of malonodinitrile, 1.14 mL of acetic acid, and 0.4 g of ammonium acetate in 50 mL of benzene was refluxed for 10 h with a Dean-Stark trap, then 0.5 mL (5 mmol) of $NCCH_2\cdot COOEt$ or 0.3 g (5 mmol) of $CH_2(CN)_2$, 1.14 mL of AcOH, and 0.4 g of $AcONH_4$ was added. The reaction mixture was again boiled for 10 h, cooled, washed with water, the organic layer was dried with Na_2SO_4, evaporated, the residue was purified by column chromatography on silica gel (elient CH_2Cl_2).

2-(1-{9-[4-(4-Methoxyphenoxy)butyl]-9*H*-carbazol-3-yl}ethylidene)malonodinitrile (8). Yield 70%, yellow viscous substance. IR spectrum, ν, cm^{-1}: 2217 (C≡N). UV absorption spectrum, λ_{max}, nm (log ε): 322 (3.79), 336 (3.78), 356 (3.77), 379 (3.77), 392 (3.74), 407 (3.66), 421 (3.66); fluorescence spectrum, λ_{max}, nm: 458, 542. Stokes shift Δλ 121 nm. ^{1}H NMR spectrum, δ, ppm: 1.83 quintet (2H, $C\underline{H_2}CH_2N$, *J* 6.3 Hz), 2.09 quintet (2H, $OCH_2C\underline{H_2}$, *J* 7.2 Hz), 2.76 s (3H, Me), 3.75 s (3H, OMe), 3.92 t (2H, NCH_2, *J* 6.0 Hz), 4.42 t (2H, OCH_2, *J* 6.9 Hz), 6.79 s (4H, C_6H_4), 7.31 t (1H, carbazole, *J* 7.5 Hz), 7.45–7.55 m (3H, carbazole), 7.77 d (1H, carbazole, *J* 8.4 Hz), 8.12 d (1H, carbazole, *J* 7.5 Hz), 8.39 s (1H, carbazole). ^{13}C NMR spectrum, δ, ppm: 14.2, 23.7, 25.9, 26.9, 28.9, 38.7, 43.2, 55.7, 68.1, 109.0, 109.4, 113.9, 114.7, 115.4, 120.4, 120.8, 120.9, 122.6, 123.2, 125.6, 126.2, 127.0, 128.8, 130.8, 141.1, 142.6, 152.9, 153.9. Mass spectrum, *m/z* (I_{rel}, %): 435 (15.4) $[M]^+$, 313 (23.5), 312 (100), 271 (12.1), 270 (52.8). Found, %: C 77.18; H 5.70; N 9.51. $C_{28}H_{25}N_3O_2$. Calculated, %: C 77.22; H 5.79; N 9.65. *M* 435.52.

Ethyl 2-cyano-3-{9-[4-(4-methoxyphenoxy)-butyl]-9*H*-carbazol-3-yl}but-2-enoate (9). Yield 75%, yellow viscous substance. IR spectrum, ν, cm^{-1}: 2259 (C≡N), 1722 (COOEt). UV absorption spectrum, λ_{max}, nm (log ε): 320 (3.45), 351 (3.47), 373 (3.51), 397 (3.37); fluorescence spectrum, λ_{max}, nm: 446, 550. Stokes shift Δλ 153 nm. ^{1}H NMR spectrum, δ, ppm: 1.39 t (3H, $COOCH_2C\underline{H_3}$, *J* 6.9 Hz), 1.85 quintet (2H, $C\underline{H_2}CH_2N$, *J* 6.3 Hz), 2.10 quintet (2H, $OCH_2C\underline{H_2}$, *J* 7.2 Hz), 2.83 s (3H, Me), 3.75 s (3H, OMe), 3.91 t (2H, NCH_2, *J* 6.0 Hz), 4.36 t (2H, OCH_2, *J* 7.5 Hz), 4.39 q (2H, $COOC\underline{H_2}CH_3$), 6.80 s (4H, C_6H_4), 7.26 t (1H, carbazole, *J* 7.8 Hz), 7.42–7.52 m (3H, carbazole), 7.63 d (1H, carbazole, *J* 7.5 Hz), 8.10 d (1H, carbazole, *J* 7.8 Hz), 8.25 s (1H, carbazole). ^{13}C, δ, ppm: 14.2, 23.8, 25.9, 27.0, 29.7, 43.0, 55.6, 55.7, 61.8, 68.1, 108.6, 109.1, 114.6, 114.7, 115.4, 117.4, 119.8, 120.4, 120.6, 122.7, 122.9, 125.6, 126.4, 126.5, 130.6, 140.9, 141.6, 152.9, 153.8, 163.0. Mass spectrum, *m/z* (I_{rel}, %): 483 (10.4) $[M + H]^+$, 482 (29.6) $[M]^+$, 360 (27.1), 359 (100), 318 (10.1), 317 (41.6), 314 (16.9), 313 (68.4), 289 (17.4), 271 (15.7), 243 (12.7), 242 (10.7), 241 (10.4), 229 (17.6). Found, %: C 74.56; H 6.21; N 5.70. $C_{30}H_{30}N_2O_4$. Calculated, %: C 74.67; H 6.27; N 5.81. *M* 482.57.

^{1}H NMR spectra were registered on a spectrometer Varian Mercury plus-300 in $CDCl_3$ (internal reference HMDS), mass spectra were measured on an instrument Agilent Technologies 6890N/5975B (ionizing electrons energy 70 eV). IR spectra were recorded on a spectrophotometer Specord 75 IR from solutions in chloroform. Elemental analysis was carried out on an analyzer CHNS-932 LECO Corp. UV spectra were obtained on a spectrophotometer SF-2000 in chloroform, cell 10 mm. Fluorescence spectra were registerred on a spectrofluorimeter Shimadzu RF-5301. The excitation wavelength 220 nm, cell size 10 × 10 mm, solvent $CHCl_3$. The reaction progress was monitored and the purity of compounds obtained was checked by TLC on Sorbfil plates. The mixtures were separated and the target products were purified by column chromatography on silica gel (Lancaster, Silica gel 60, 0.060–0.2 mm). Electrochemical measurements were carried out in monomer solutions (*c* 10^{-3} mol/L) in 9 : 1 acetonitrile–CH_2Cl_2 mixture, at room temperature using $Et_4N^+ClO_4^-$ as supporting electrolyte (*c* 0.1 mol/L). A potentiostate P-8 was used with electrochemical sensor Module EM-04 and three-electrode electrochemical cell: working electrode – glassy carbon disc, ITO-plate, or Pt wire, auxiliary electrode – Pt wire and Ag/

AgCl reference electrode. The measurements were performed at room temperature, potential scanning rate V_{scan} 50 mV/s.

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