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Synthesis and properties of 2-substituted naphtho[2,3-d]imidazole and benzoxazole derivatives

Birutė Sapijanskaitė[1],

Vytautas Mickevičius[1]*,

Kristina Kantminienė[2] and

Gema Mikulskienė[3]

[1] Department of Organic Chemistry, Kaunas University of Technology, Radvilėnų 19, LT-50254 Kaunas, Lithuania

[2] Department of General Chemistry,

[3] Department of Bioorganic Compounds, Institute of Biochemistry, Vilnius, Lithuania

Condensation of 1-(9-alkyl-9*H*-carbazol-3-yl)-4-carboxy-2-pyrrolidinones with 2,3-diaminonaphthalene or *o*-aminophenol was carried out by melting a reaction mixture at 170–230 ˚C and provided new 2-substituted naphtho[2,3-d]imidazole and benzoxazole derivatives. Derivatives of 2-substituted naphtho[2,3-d]imidazole were alkylated with an excess of ethyl iodide in the presence of potassium hydroxide. The structure of the study compounds was elucidated by ^{1}H and ^{13}C NMR spectroscopy. Their hole-transporting properties were investigated.

Key words: carbazole, 2-substituted naphtho[2,3-d]imidazole, benzoxazole, alkylation, hole-transporting properties, NMR spectra

INTRODUCTION

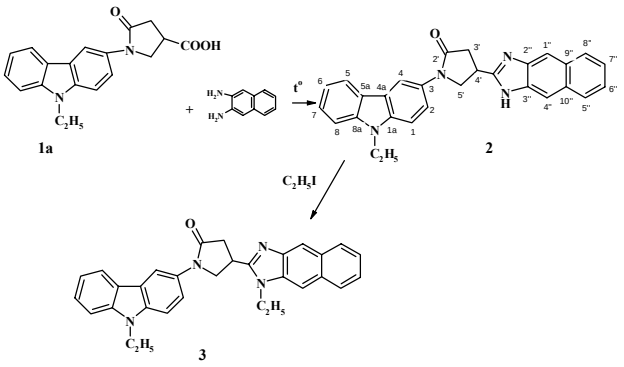
The interest in the chemistry of carbazoles is beginning to increase steadily, since functionalized carbazoles are synthetically interesting building blocks for certain carbazole-containing alkaloids and pharmacologically attractive carbazole derivatives [1-3], optical recording materials and organic photoconductors [4-7].

The aim of this work was synthesis of new carbazole derivatives containing naphthoimidazole and benzoxazole fragments, and investigation of their hole-transporting properties.

RESULTS AND DISCUSSION

The starting 1-(9-alkyl-9*H*-carbazol-3-yl)-4-carboxy-2-pyrrolidinones **1a, b** were prepared by reaction of the corresponding 3-amino-9-alkylcarbazoles with itaconic acid by the method described previously [8].

Naphtho[2,3-d]imidazole **2** was obtained by heating a mixture of 1-(9-ethyl-9*H*-carbazol-3-yl)-4-carboxy-2-pyrrolidinone **1a** and 2,3-diaminonaphthalene at 170–190 ˚C for 2 h and then at 220–230 ˚C for 30 min (Scheme 1). Alkylation of compound **2** in the presence of potassium hydroxide and sodium carbonate with excess of ethyl iodide without solvent at room temperature led to 1-(9-ethyl-9*H*-carbazol-3-yl)-4-(1-ethyl-1*H*-naphtho[2,3-d]imidazol-2-yl)-2-pyrrolidinone **(3)**.



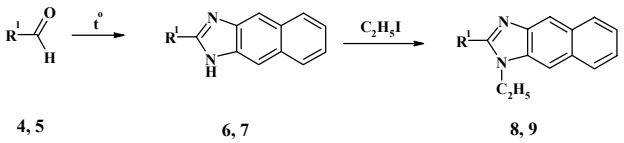
Scheme 1. Synthesis of 1,4-bisubstituted-2-pyrrolidinones

In order to compare the ionization potentials, compounds **6**, **7** were synthesized from the corresponding aldehydes **4**, **5** and 2,3-diaminonaphthalene in refluxing nitrobenzene. Alkylation of compounds **6** and **7** with ethyl iodide without solvent in the presence of potassium hydroxide provided naphtho[2,3-d]imidazole derivatives **8** and **9** (Scheme 2).

It was attempted to form a benzoxazole ring by the methodology [9] according to which a mixture of heterocyclylcarboxylic acid, o-aminophenol and orthoboric acid was refluxed in ethylene glycol. However, this method

* Corresponding author. E-mail: vytautas.mickevicius@ktu.lt

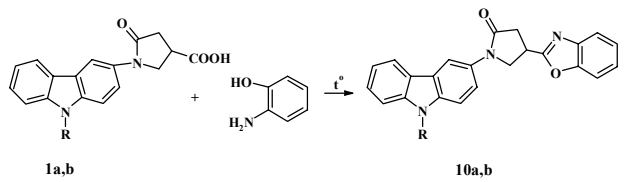
did not yield the objective products. Harsher conditions, i.e. melting of 1-substituted 4-carboxy-2-pyrrolidinones **1 a, b** and *o*-aminophenol at 190-220 ˚C gave 1-(9-alkyl-9*H*-carbazol-3-yl)-4-(1,3-benzoxazol-2-yl)-2-pyrrolidinones **10 a, b** in 25% (**10a**) and 36 % (**10b**) yields (Scheme 3).



4, 5 **6, 7** **8, 9**

4, 6, 8 R^1 = 9-ethylcarbazol-3-yl,
5, 7, 9 R^1 = 4-$(C_2H_5)_2$-N-C_6H_4

Scheme 2. Synthesis of 1- and 1,2-substituted naphtho[2,3-d]imidazoles



1a,b **10a,b**

a RC_2H_5, **b** RC_3H_7

Scheme 3. Synthesis of 1-(9-alkyl-9*H*-carbazol-3-yl)-4-(1,3-benzoxazol-2-yl)-2-pyrrolidinones

The structure of the synthesized compounds was determined by ^{1}H and ^{13}C NMR spectroscopy [10, 11]. Assignment of the resonances of the atoms was based on the chemical shift theory, using previously determined increments of substituents, and by considering the peak intensity, multiplicity and modifying spectra of the structurally relative compounds [12–15]. The obtained data are reported in Table and in the Experimental.

The molecules of the study compounds include a 1N-substituted (**3, 8, 9**) or a non-substituted (**2, 6, 7**) naphthoimidazole fragment. The exploration of the structural features by NMR spectra of such a type of compounds presents some problems [13]. Owing to the versatility of the heterocyclic part, two tautomeric forms of the naphthoimidazole fragment are presumed [10, 11]. The suitable signals in compounds **2, 6** and **7** are poorly resolved, broadened or not observed at all. The introduction of the substituent CH_2CH_3 at N-1 position stabilizes the form of naphthoimidazole fragment [11, 13] of compounds **3, 8** and **9**. The resonances of naphthoimidazole moiety in NMR spectra become sharp.

The molecules of compounds **2** and **3** possess a pyrrolidinone ring which is sensitively affected by the 1,4 substituents. Interestingly, the ^{1}H NMR spectrum of compound **2** shows the equivalence of the protons as in

Table. **^{13}C NMR chemical shifts (125.758 MHz, δ, ppm) for compounds 1-(9-Ethyl-9*H*-carbazol-3-yl)-4-(1*H*-naphtho[2,3-d]imidazol-2-yl)-2-pyrrolidinone (2), 1-(9-Ethyl-9*H*-carbazol-3-yl)-4-(1-ethyl-1*H*-naphtho[2,3-d]imidazol-2-yl)-2-pyrrolidinone (3), substituted naphtho[2,3-d]imidazoles 6-9**

Carbon atom	**2**	**3***	**6**	**7**	**8***	**9**
C-1	108.97	108.63	109.60	122.58	107.36	121.19
C-2	119.65	118.91	126.44	128.71	126.47	129.07
C-3	131.28	130.55	120.53	110.99	122.96	110.14
C-4	112.65	113.64	122.24	149.10	122.08	149.32
$N(CH_2CH_3)_2$				43.67; 12.42		43.51; 12.38
C-4a	121.93	122.74	122.48		121.43	
C-1a	136.73	137.71	140.20		140.25	
C-5a	122.09	122.99	122.48		122.04	
C-5	120.49	120.66	120.48		120.32	
C-6	118.69	118.91	119.62		119.26	
C-7	125.14	125.99	125.03		125.51	
C-8	109.23	108.63	108.63		108.60	
C-8a	140.00	140.45	140.88		140.70	
CH_2CH_3 (R)	37.00; 13.70	38.29; 13.29	37.24; 13.79		37.55 13.74	
C-2’	171.41	171.12				
C-3’	37.19	37.63				
C-4’	31.26	30.61				
C-5’	53.07	53.55				
C=N	159.88	157.41	156.75	156.41	172.07	172.68
C-1”, C-4”	114.80 106.36	116.56; 105.17		115.65; 111.77	104.81	108.52
C-2”, C-3”	143.54	135.57		146.54	142.16	142.55
C-5, “C-8”	129.95; 129.44	128.57; 127.40	127.66	129.74; 127.44	127.56	129.10; 125.29
C-6”, C-7”	123.68; 122.94	124.60; 123.66	123.22	122.89	125.51	122.93
C-9”, C-10”	129.95; 129.44	130.45; 130.36	128.98	130.39; 128.90	134.35 129.11	135.46; 129.10
CH_2CH_3 (R^1)		38.74; 15.15			38.25; 14.77	38.18; 14.62

* ^{13}C NMR spectra for compounds **3**, **8** were recorded in $CDCl_3$.
^{13}C NMR spectra for compounds **2**, **6**, **7**, **9** were recorded in DMSO-d_6.

CH_2CO as in CH_2N fragments of pyrrolidinone ring, while the corresponding protons in compound **3** are easily differentiated.

The ^{13}C NMR spectrum of compound **2** shows the C=N group carbon resonance about 2.5 ppm at a lower field than in compound **3** due to the 1N substitution**.** The molecules of **6** and **8** contain naphthoimidazole fragment and carbazole moiety interconnected into an extended π-electron system. In compound **8,** 1N substitution significantly affects C=N carbon. It has been found to exert a strong deshielding influence on the C=N carbons up to 15 ppm. An analogous observation has been made when comparing the spectra of **7** and **9:** the deshielding of the C=N group carbon reaches 16 ppm.

The NMR spectra of compounds **10a, 10b** were assigned by comparison with the model compounds [10, 14]. The disappearance of the proton signal of the NH group in ^1H NMR spectrum confirmed existence of the above-mentioned compounds.

Compounds **3, 8, 9** and **10a, b** have been tested as hole-transporting substances in electrophotographic material. Samples for the measurement of the ionisation potential were prepared by coating solutions of the compounds in THF on polyester substrates with an Al conductive layer. The ionisation potential was measured by the method of electron photoemission in air, similar to that described in Ref. [5].The ionisation potentials of these transporting molecules were 5.49, 5.56, 5.7 eV, respectively (Figure) and 5.7, 5.45 eV.

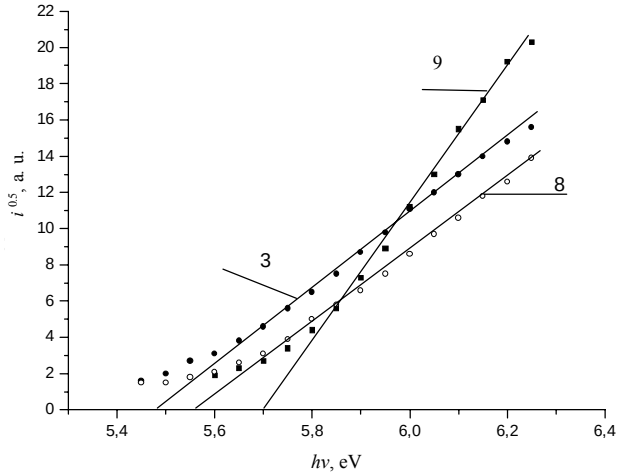


Figure. Photoemission spectra of compounds 1-(9-ethyl-9*H*-carbazol-3-yl)-4-(1-ethyl-1*H*-naphtho[2,3-d]imidazol-2-yl)-2-pyrrolidinone **(3),** 2-(9-ethyl-9*H*-carbazol-3-yl)-1-ethyl-1*H*-naphtho[2,3-d]imidazole **(8)** and N,N-diethyl-4-(1-ethyl-1*H*-naphtho[2,3-d]imidazol-2-yl)aniline **(9)**

A comparison of the ionization potential values of compounds **3**, **8** and **9** has shown that the lowest energy needed to release electrons is characteristic of compound **3** and the highest one to compound **9** containing 4-diethylaminophenyl chromophores. Analysis of the potential values of the latter compounds has enabled to assume that the pyrrolidinone ring has an influence on the decrease of the ionization potential value. However, the degree of molecule conjugation does not necessarily ordain the ionization potential value [16].

A comparison of electron photoemission in air spectra for compounds **10a** and **10b** which differ by the substituent at the 9-possition of carbazole ring only has revealed that the difference of the ionization potential values is 0.25 eV. The ionization potential decreases with a longer aliphatic chain.

The investigation has revealed that the compounds studied possess hole-transporting properties. The values of their ionisation potential are characteristic of hole-transporting materials used for producing of electrophotographic layers.

EXPERIMENTAL

Melting points were determined on an automatic APA1 melting point apparatus and are uncorrected. NMR spectra were recorded on a Joel FX 100 (100 MHz) and a Bruker DRX 500 (500 MHz) spectrometers using tetramethylsilane as the internal standard. TLC was performed with Silufol UV-254 plates, visualization was done under UV light or using iodine.

1-(9-Ethyl-9*H*-carbazol-3-yl)-4-(1*H*-naphtho[2,3-d]imidazol-2-yl)-2-pyrrolidinone (2). A mixture of 1-(9-ethyl-9H-carbazol-3-yl)-4-carboxy-2-pyrrolidinone **(1a)** (6.44 g, 0.02 mol) and 2,3-diaminonaphthalene (3.16 g, 0.02 mol) was melted at 170-230 ˚C for 2 h and additionally at 230 ˚C for 30 min. The reaction mixture was dissolved in dimethylformamide (30 ml) and while still hot was poured to 5% solution of sodium carbonate (100 ml) with stirring. The solution obtained was cooled down, and the resin deposited. It was washed with water and crystallised. Yield 6.83 g (77%). M.p. 264-266 ºC (dioxane). ^1H NMR (500 MHz, DMSO-d_6, δ, ppm): 1.29 (t, J = 7.0 Hz, 3H, NCH_2CH_3), 3.11 (d, J = 8.2 Hz, 2H, CH_2CO), 4.15 (quin, 1H, CH), 4.43 (overlapping t, q, 4H, NCH_2, NCH_2CH_3), 7.15–8.35 (m, 13H, H_{ar}), 12.59 (s, 1H, NH). Elemental analysis data: found, %: C, 78.56; H, 5.78; N, 12.52; formula $C_{29}H_{24}N_4O$ (444.536): calculated, %: C, 78.36; H, 5.44; N, 12.60.

1-(9-Ethyl-9*H*-carbazol-3-yl)-4-(1-ethyl-1*H*-naphtho[2,3-d]imidazol-2-yl)-2-pyrrolidinone (3). A mixture of compound **2** (1.33 g, 0.003 mol)**,** potassium hydroxide powder (0.4 g, 0.006 mol), sodium carbonate (1 g) and ethyl iodide (30 ml) was refluxed for 4 h. Then acetone (30 ml) was added, the hot solution was filtered and volatile fractions were evaporated on a rotary evaporator. Diethyl ether was added to the residue, the crystals formed were filtered, washed with diethyl ether, dried, and purified by column chromatography (Silufol L 40/100, acetone) to give 0.78 g (55%) of compound **3**. M.p. 214-215 ˚C. ^1H NMR (500 MHz, $CDCl_3$, δ, ppm): 1.42 (t, J = 7.2 Hz, 3H, NCH_2CH_3), 1.55 (t, J = 7.2 Hz, 3H, NCH_2CH_3), 3.12-3.29 (m, 2H, CH_2CO), 4.10 (quin, 1H, CH), 4.29-4.38 (m, 5H, $(NCH_2CH_3)_2$, NCH), 4.89 (t, $^2J_{HCH}$ = 9,1 Hz, $^3J_{HCHCH}$ = 9,1 Hz, 1H, NCH), 7.22-8.26 (m, 13H, H_{ar}). Elemental analysis data: found, %: C, 78.43; H, 5.7; N,

11.58; formula $C_{31}H_{28}N_4O$ (472.589): calculated, %: C, 78.79; H, 5.98; N, 11.85.

2-(9-Ethyl-9*H*-carbazol-3-yl)-1*H*-naphtho[2,3-d]imidazole (6). A mixture of 9-ethyl-3-formylcarbazole **4** (5.58 g, 0.025 mol), 2,3-diaminonaphthalene (3.95 g, 0.025 mol) and nitrobenzene (20 ml) was refluxed for 2 h and cooled down. The solution was diluted with benzene; the precipitate formed was filtered and washed with diethyl ether. Yield 3.1 g (34.33%). M.p. 172-174 ˚C (ethanol). ^{1}H NMR (500 MHz, DMSO-d_6, δ, ppm): 1,35 (t, J = 7.2 Hz, 3H, NCH_2CH_3), 4.50 (q, J = 7.2 Hz, 2H, NCH_2CH_3), 7.28–9.11 (m, 13H, H_{ar}), 12.93 (s, 1H, NH). Elemental analysis data: found, %: C, 87.95; H, 5.15; N, 11.32; formula $C_{25}H_{19}N_3$ (361.446): calculated, %: C, 83.08; H, 5.30; N, 11.62.

N,N-Diethyl-4-(1*H*-naphtho[2,3-d]imidazol-2-yl)aniline (7) was obtained from 4-diethylaminobenzaldehyde **5** (3.5 g, 0.02 mol) and 2,3-diaminonaphthalene (3.16 g, 0.02 mol) according to the synthesis method of compound **6**. Yield 3.69 g (59.4%). M.p. 182 ˚C (decomposes; dioxane or chloroform). ^{1}H NMR (500 MHz, DMSO-d_6, δ, ppm): 1.13 (t, 6H, $(NCH_2CH_3)_2$), 3.42 (m, 4H, $(NCH_2CH_3)_2$), 6.46-8.13 (m, 10H, H_{ar}), 12.49 (s, 1H, NH). Elemental analysis data: found, %: C, 80.02; H, 6.54; N, 13.40; formula $C_{21}H_{21}N_3$ (315.412): calculated, %: C, 79.97; H, 6.71; N, 13.32.

2-(9-Ethyl-9*H*-carbazol-3-yl)-1-ethyl-1*H*-naphtho[2,3-d]imidazole (8). A mixture of compound **6** (1.45 g, 0.004 mol), potassium hydroxide powder (0.448 g, 0.008 mol), anhydrous sodium carbonate (0.8 g) and ethyl iodide (30 ml) was refluxed for 3 h. Then acetone (30 ml) was added, hot solution was filtered, volatile fractions were evaporated on a rotary evaporator. Diethyl ether was added to the residue, the crystals formed were filtered, washed with diethyl ether and dried. Yield 1 g (64%). M.p. 198-199 ˚C (chloroform). ^{1}H NMR (500 MHz, $CDCl_3$, δ, ppm): 1.31 (t, J = 6.9 Hz, 3H, NCH_2CH_3), 1.43 (t, J = 7.2 Hz, 3H, NCH_2CH_3), 4.19 (q, J = 6.9 Hz, 2H, NCH_2CH_3), 4.34 (q, J = 7.2 Hz, 2H, NCH_2CH_3), 6.90-8.30 (m, 13H, H_{ar}). Elemental analysis data: found, %: C, 83.47; H, 5.69; N, 10.68; formula $C_{27}H_{23}N_3$ (389.490): calculated, %: C, 83.27; H, 5.95; N, 10.78.

N,N-Diethyl-4-(1-ethyl-1*H*-naphtho[2,3-d]imidazol-2-yl)aniline (9) was obtained from compound **7** (1.57 g, 0.005 mol) according to the synthesis method of compound **3**. Crystals were purified by column chromatography (Silufol L 40/100, acetone : hexane, 1 : 1). Yield 0.68 g (40%). M.p. 146–147.5 ˚C. ^{1}H NMR (500 MHz, DMSO-d_6, δ, ppm): 1.04 (t, 6H, $N(CH_2CH_3)_2$), 1.31 (t, J = 6.9 Hz, 3H, NCH_2CH_3), 3.42 (m, 4H, $(NCH_2CH_3)_2$), 4.19 (q, J = 6.9 Hz, 2H, NCH_2CH_3), 6.23-8.40 (m, 10H, H_{ar}). Elemental analysis data: found, %: C, 80.20; H, 7.15; N, 12.07; formula $C_{23}H_{25}N_3$ (343.465): calculated, %: C, 80.43; H, 7.34; N, 12.23.

4-(1,3-Benzoxazol-2-yl)-1-(9-ethyl-9*H*-carbazol-3-yl)-2-pyrrolidinone (10a). A mixture of pyrrolidinone **1a** (1.93 g, 0.006 mol) and 2-aminophenol (1.3 g, 0.012 mol) was melted at 190 ºC for 2 h and additionally at 220 ºC for 30 min. 5% solution of Na_2CO_3 (100 ml) was added and mixture was refluxed for 5 min. Afterwards it was cooled down and the resin formed was washed with water. It was crystallised from acetone. Yield 0.59 g (25%). M.p. 172-173 ˚C. ^{1}H NMR (100 MHz, $CDCl_3$, δ, ppm): 1.32 (t, J = 7.1 Hz, 3H, NCH_2CH_3), 2.98-3.28 (m, 2H, $COCH_2$), 3.76-4.63 (m, 5H, NCH_2, NCH_2CH_3, CH), 6.91-8.34 (m, 11H, H_{ar}). Elemental analysis data: found, %: C, 75.82; H, 5.46; N, 10.85; formula $C_{25}H_{21}N_3O_2$ (395.460): calculated, %: C, 75.93; H, 5.35; N, 10.63.

4-(1,3-Benzoxazol-2-yl)-1-(9-propyl-9*H*-carbazol-3-yl)-2-pyrrolidinone (10b) was obtained from pyrrolidinone **1b** (2 g, 0.006 mol) according to the synthesis method of compound **10a**. The product was purified by column chromatography (Silufol L 40/100, acetone : hexane, 1 : 1). Yield 0.87g (36%). M.p. 144-145 ˚C. ^{1}H NMR (100 MHz, $CDCl_3$, δ, ppm): 0.84 (t, J = 7.4 Hz, 3H, $NCH_2CH_2CH_3$), 1.78 (quin, J = 7.4 Hz, 2H, $NCH_2CH_2CH_3$), 2.94-3.26 (m, 2H, $COCH_2$), 3.74-4.56 (m, 5H, NCH_2, $NCH_2CH_2CH_3$, CH), 6.86-8.33 (m, 11H, H_{ar}). Elemental analysis data: found, %: C, 75.98; H, 5.66; N, 10.50; formula $C_{26}H_{23}N_3O_2$ (409.487): calculated, % C, 76.27; H, 5.66; N, 10.26.

CONCLUSIONS

Reactions of 1-(9-alkyl-9*H*-carbazol-3-yl)-4-carboxy-2-pyrrolidinones with 2,3-diaminonaphthalene and *o*-aminophenol were investigated, and it has been determined that melting of reagent mixtures at 170–230 ˚C provides the corresponding 2-substituted derivatives of naphthoimidazole and benzoxazole. The structure of the synthesized compounds was determined.

The positive charge transporting properties of some of the synthesized compounds have been investigated by electrographic methods. The results have shown that the synthesized carbazole-containing compounds exhibit hole-transporting properties. Their ionization potentials are characteristic of hole-transporting substances used in electrography.

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2-PAKEISTŲ NAFT[2,3-d]IMIDAZOLO IR BENZOKSAZOLO DARINIŲ SINTEZĖ IR SAVYBĖS

S a n t r a u k a

1-(9-Alkil-9*H*-karbazol-3-il)-4-karboksi-2-pirolidinonų **1a,b** kondensacija su 2,3-diaminonaftalenu ar 2-aminofenoliu buvo vykdyta lydant reakcijos mišinį 170–230 ˚C temperatūroje. Junginiai **6**, **7** gauti virinant atitinkamus aldehidus **4, 5** su 2,3-diaminonaftalenu nitrobenzene. 1-(9-etil-9*H*-karbazol-3-il)-4-(1-etil-1*H*-naft[2,3-d]imidazol-2-il)-2-pirolidinonas **3** ir naft[2,3-d]imidazolo dariniai **8, 9** susintetinti alkilinant junginius **2, 6, 7** jodetanu be tirpiklio, esant kalio šarmo. Susintetintų junginių struktūra nustatyta ^{1}H ir ^{13}C BMR spektroskopijos metodais. Dalies susintetintų junginių teigiamųjų krūvių pernašos ypatybės ištirtos elektrografiniais metodais. Jų jonizacijos potencialai yra būdingi skylučių pernašos medžiagoms, naudojamoms elektrofotografijoje.