

**5-NITRO-2-FURYL VINYLATION OF AROMATIC POLYCYCLIC COMPOUNDS\***

Ján HRABOVSKÝ, Miloslava DANDÁROVÁ and Jaroslav KOVÁČ

*Department of Organic Chemistry,  
Slovak Institute of Technology, 880 37 Bratislava*

Received December 22nd, 1980

1-Bromonaphthalene, acenaphthene, phenanthrene, pyrene, chrysene, fluorene, guaiazulene, dibenzofuran, indole, N-methylindole, N-butylindole and carbazole react with (*Z*)-2-bromo-1-(5-nitro-2-furyl)ethylene in the presence of aluminium chloride to afford 2-substituted 1-(5-nitro-2-furyl)ethylenes. The structure of the prepared derivatives was confirmed by electron impact mass and <sup>1</sup>H-NMR spectroscopies.

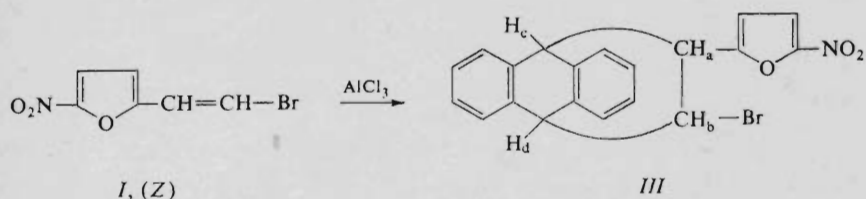
The 5-nitro-2-furylvinilation of condensed aromatic and heteroaromatic systems under conditions of Friedel-Crafts reaction<sup>1,2</sup> was further studied and a series of 2-substituted 1-(5-nitro-2-furyl)ethylenes *IIa–IIf* was synthesized. Products of vinylation are listed in Table I.

5-Nitro-2-furylvinilation of anthracene afforded, in contrast to other aromates, the addition product 1-(5-nitro-2-furyl)-2-bromo-1,2-*endo*-(9,10-dihydroanthranyl)-ethane (*III*, Scheme 1) and not the desired 2-(9-anthranyl)-1-(5-nitro-2-furyl)-ethylene. Probably, due to the catalyst (Lewis acid), a cycloaddition reaction was preferred before the substitution reaction<sup>3</sup>. The addition product also originated when reacting β-chlorovinyl ketones with anthracene<sup>4</sup>. The substitution product in position 2 was the only substance obtained when substituting naphthalene<sup>1</sup>. As known<sup>5</sup>, replacement of the solvent can lead to a change in the rate of isomers 1 and 2 resulting from acylation of naphthalene. A series of reactions under conditions of vinylation was carried out with various solvents (nitromethane, nitrobenzene, carbon disulfide) but the substitution product to position 1 was not isolated, even at –80°C. We have already reported<sup>2</sup> that neither N,N-dimethylaniline, nor N-acetanilide could be vinylated under the given reaction conditions. The nitrogen atom played here the role of a substituent. Now, we have ascertained that vinylation can well proceed were the nitrogen atom embodied in the aromatic ring (indole, N-methylindole, N-butylindole, carbazole).

Structures of the synthesized products were confirmed both by electron impact mass and <sup>1</sup>H-NMR spectrometries. The base peaks in the mass spectra of derivatives

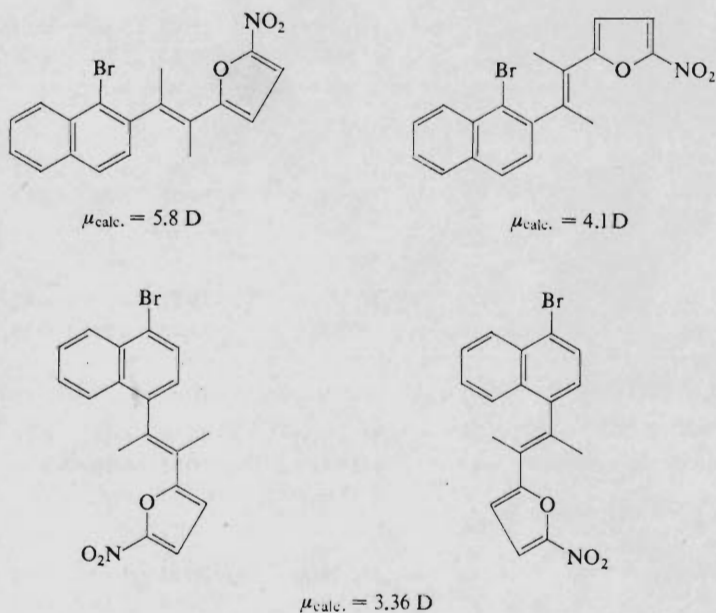
\* Part CLIX in the series Furan Derivatives; Part CLVIII: This Journal 46, 2564 (1981).

*Ila–IIIh* were represented by molecular ions. The principal fragmentation pattern was in accordance with that described in the preceding paper<sup>1</sup>. The chemical shift values and coupling constants are listed in Tables II and III. All prepared products were in the thermodynamically stable (*E*)-configuration and *s-trans* conformation<sup>2</sup> as evidenced by the coupling constant (15.6–16.5 Hz).



SCHEME 1

Splitting of protons of 1-bromonaphthalene ( $J = 8.1$  Hz) did not prove unambiguously the position of substitution (positions 2, or 4) and therefore, the dipole moment of derivative *IIa* (Table IV) was measured. The dipole moment of possible isomers was determined by a graphic addition of vectors<sup>6</sup>. Graphic solution of the derivative substituted in position 2 (Scheme 2) for both conformers which could not be unequi-



SCHEME 2

TABLE I  
5-Nitro-2-furylvinylation products of aromates

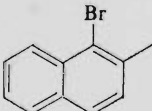
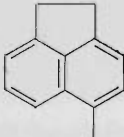
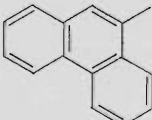
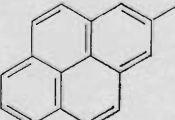
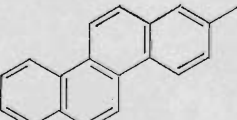
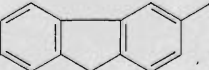
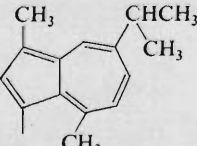
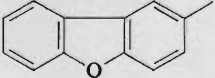
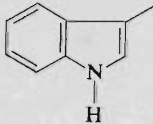
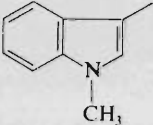
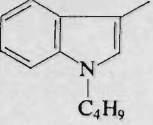
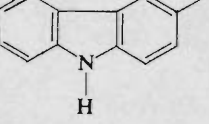
Compound	Ar	Formula (mol. weight)	Calculated/Found			M.p., °C yield, %
			% C	% H	% N	
<i>Ila</i>		$C_{16}H_{10}BrNO_3$ (344.2)	55.83 55.71	2.93 2.73	4.07 3.84	173–175 50.8
<i>Ilb</i>		$C_{18}H_{13}NO_3$ (291.3)	74.21 74.07	4.50 4.46	4.81 4.62	160–163 54.8
<i>Ilc</i>		$C_{20}H_{13}NO_3$ (315.3)	76.18 76.01	4.15 4.08	4.44 4.23	164–166 44.5
<i>Ild</i>		$C_{22}H_{13}NO_3$ (339.3)	77.87 77.75	3.86 3.82	4.13 3.96	199–200 29.1
<i>Ile</i>		$C_{24}H_{15}NO_3$ (365.4)	78.88 78.72	4.14 4.08	3.83 3.63	234–236 28.0
<i>Ilf</i>		$C_{19}H_{13}NO_3$ (303.3)	75.23 75.02	4.42 4.29	4.62 4.43	166–168 42.7
<i>Ilg</i>		$C_{21}H_{21}NO_3$ (335.4)	75.20 75.01	6.31 6.24	4.18 3.95	152–154 47.9
<i>Ilh</i>		$C_{18}H_{11}NO_4$ (305.3)	70.81 70.64	3.63 3.38	4.59 4.33	188–190 51.3

TABLE I  
(Continued)

Com- pound	Ar	Formula (mol. weight)	Calculated/Found			M.p., °C yield, %
			% C	% H	% N	
<i>Ili</i>		$C_{14}H_{10}N_2O_3$ (254.2)	66.14 65.93	3.96 3.86	11.02 11.26	199–201 37.4
<i>Ilj</i>		$C_{15}H_{12}N_2O_3$ (268.3)	67.14 67.03	4.51 4.46	10.44 10.46	143–145 45.2
<i>Iik</i>		$C_{18}H_{18}N_2O_3$ (310.3)	69.67 69.48	5.85 5.68	9.03 8.85	100–101 47.7
<i>Ill</i>		$C_{18}H_{12}N_2O_3$ (304.3)	71.04 70.87	3.97 3.78	9.21 9.33	242–244 46.3

vocally recognized from analysis of  $^1\text{H-NMR}$  spectra gave values  $\mu_{\text{calc.}} = 4.1 \text{ D}$  and  $\mu_{\text{calc.}} = 5.8 \text{ D}$ . The  $\mu_{\text{calc.}}$  value for derivatives substituted in position 4 was identical for both conformers, and amounted 3.36 D. Comparison of the calculated values with that obtained experimentally ( $\mu_{\text{exp.}} = 4.52 \text{ D}$ ) showed the point of attachment of the 5-nitro-2-furylvinyl group to be position 2 of the naphthalene ring. The difference between the found and calculated values might be associated with the mesomeric dipole moment, or with the mixture of conformers present<sup>7</sup>. The  $^1\text{H-NMR}$  spectra of derivatives *IId* and *IIE* could not be recorded due to a very little solubility in dimethyl sulfoxide even at 80°C. Basing only on the analogy with the naphthalene and 1-bromonaphthalene derivatives the structure corresponding to substitution to position 2 of the pyrene (*IId*) and chrysene (*IIE*) skeletons was assigned. Splitting of proton signals and the chemical shift values proved the attachment of the 5-nitro-2-furylvinyl group to position 9 of the phenanthrene ring (*IIC*). In contrast to anthracene, no addition product to positions 9,10 was observed. The doublet-doublet characterizing the proton  $\text{H}_7$  (derivative *IIB*) was assigned the value  $\delta = 7.53$ . The unsubsti-

TABLE II

<sup>1</sup>H-NMR spectra of derivatives II (CDCl<sub>3</sub>, tetramethylsilane, δ in ppm)

Compound	H <sub>3</sub> d	H <sub>4</sub> d	H <sub>A</sub> d	H <sub>B</sub> d	H <sub>3</sub> 'd	H <sub>4</sub> 'd	H <sub>6</sub> 'd	H <sub>7</sub> 'd	H <sub>8</sub> 'd
<i>Ila</i>	6.59	7.37	8.12	6.93	7.65	7.81	8.38–7.50 m, H <sub>5'</sub> – <sub>8'</sub>		
	$J_{3,4} = 3.9$		$J_{A,B} = 16.1$		$J_{3',4} = 8.1$				
<i>Ilb<sup>a</sup></i>	6.49	7.32	8.01	6.91	7.26	7.71	7.90	7.53	7.30
	$J_{3,4} = 3.9$		$J_{A,B} = 16.0$		$J_{3',4'} = 7.5$		$J_{6',7'} = 8.4$		$J_{7',8'} = 6.6$
<i>Ilc</i>	6.54	7.35	8.10	6.96	8.76–7.55 m, H <sub>1'</sub> – <sub>8'</sub> , 7.91 s, H <sub>10'</sub>				
	$J_{3,4} = 3.9$		$J_{A,B} = 16.0$						
<i>Ilg<sup>b</sup></i>	6.34	7.31	8.24	6.64	7.81 s, H <sub>2'</sub> , 7.29 dd, H <sub>6'</sub>			8.01	
	$J_{3,4} = 3.8$		$J_{A,B} = 15.6$		$J_{6',8'} = 2.1$ , $J_{5',6'} = 10.5$				

<sup>a</sup> *Ilb*: δ<sub>CH<sub>2</sub></sub> = 3.35 ppm (s),  $J_{6',8'} = 1.2$  Hz; <sup>b</sup> *Ilg*: δ<sub>H<sub>5'</sub></sub> = 6.96 ppm (d), δ<sub>CH<sub>3</sub>'</sub> = 2.98 ppm (s), δ<sub>iPr</sub> = 2.98 ppm (m), δ<sub>CH<sub>3</sub>'</sub> = 2.55 ppm (s), δ<sub>iPr</sub> = 1.31 ppm (d),  $J_{\text{CHCH}_3} = 7$  Hz.

TABLE III

<sup>1</sup>H-NMR spectra of derivatives II (CDCl<sub>3</sub>, tetramethylsilane, δ in ppm)

Compound	H <sub>3</sub> d	H <sub>4</sub> d	H <sub>A</sub> d	H <sub>B</sub> d	H <sub>1</sub> 'd	H <sub>2</sub> .dd	H <sub>4</sub> 'd	Other signals
<i>IIf<sup>a</sup></i>	6.48	7.32	7.43	6.85	7.75	7.61	8.61	7.88–7.27 m, H <sub>5'</sub> – <sub>8'</sub>
	$J_{3,4} = 3.8$		$J_{A,B} = 16.0$		$J_{2',4'} = 1.8$ , $J_{1',2'} = 7.9$			
<i>IIh</i>	6.48	7.32	7.50	6.87	7.92	7.52	8.03	8.00–7.30 m, H <sub>5'</sub> – <sub>8'</sub>
	$J_{3,4} = 3.9$		$J_{A,B} = 15.6$		$J_{2',4'} = 1.4$ , $J_{1',2'} = 8.4$			
<i>IIIi</i>	6.64	7.46	7.83	7.00	—	7.43 d	—	7.97–7.32 m, H <sub>4'</sub> – <sub>7'</sub>
	$J_{3,4} = 3.9$		$J_{A,B} = 16.2$		$J_{\text{H}_2'/\text{NH}} = 2.0$			
<i>IIj<sup>b</sup></i>	6.39	7.35	7.58	6.82	—	7.32 s	—	7.89–7.18 m, H <sub>4'</sub> – <sub>7'</sub>
	$J_{3,4} = 3.9$		$J_{A,B} = 16.3$					
<i>IIIk<sup>c</sup></i>	6.39	7.34	7.59	6.82	—	7.33 s	—	7.90–7.18 m, H <sub>4'</sub> – <sub>7'</sub>
	$J_{3,4} = 3.9$		$J_{A,B} = 16.5$					
<i>III<sup>d</sup></i>	6.88	7.63	7.70	7.23	8.23	7.82	8.51	8.35–7.20 m, H <sub>5'</sub> – <sub>8'</sub>
	$J_{3,4} = 3.9$		$J_{A,B} = 16.1$		$J_{2',4'} = 1.4$ , $J_{1',2'} = 8.3$			

<sup>a</sup> *IIf* δ<sub>CH<sub>2</sub></sub> = 3.88 ppm (s); <sup>b</sup> *IIj* δ<sub>CH<sub>3</sub></sub> = 4.15 ppm (s); <sup>c</sup> *IIIk* δ<sub>CH<sub>2</sub></sub> = 4.12 ppm (t), δ<sub>CH<sub>2</sub></sub> = 1.85 ppm (m), δ<sub>CH<sub>2</sub></sub> = 1.35 ppm (m), δ<sub>CH<sub>3</sub></sub> = 0.94 ppm (t); <sup>d</sup> in hexadeuterioacetone.

tuted acenaphthene protons  $H_5$  and  $H_6$  revealed the highest values (7.46, ref.<sup>8</sup>). The value  $\delta = 7.90$  was ascribed to proton  $H_6$ , on the basis of analogy and multiplicity  $\delta = 7.71$  to proton  $H_4$ , also on the base of multiplicity (doublet). Splitting pattern of proton  $H_8$ , was compared with the signal at  $\delta = 7.26$  to which proton  $H_3$ , was assigned; consequently, substitution to position 5 of acenaphthene was thereby evidenced. The known influence of the 5-nitro-2-furylvinyl residue on the chemical shift value of the proton in positions *ortho*, *meta*, or *para* on the aromatic ring<sup>2</sup>, and the character of splitting (doublet) and coupling constants (1.4–1.8 Hz) entitled to ascribe values  $\delta = 8.03$ –8.61 to proton  $H_4$ , 7.75–8.23 (doublet) to proton  $H_1$ , and 7.52–7.82 (doublet-doublet) to proton  $H_2$ , evidencing thus the substitution to position 3 of derivatives *IIf*, *Iih* and *III*. Values  $\delta = 7.32$ –7.43 (derivatives *IIIi*–*IIIk*) were assigned to proton  $H_2$ , on the basis of multiplicity, *i.e.* substitution to position 3 of the indole backbone. Proton  $H_2$ , of the azulene ring system of derivative *Iig* was shown to be a singlet what indicates the 3-substituted guaiazulene.

It could generally be concluded that comparison of structures proved by <sup>1</sup>H-NMR spectrometry, as well as molecular diagrams of the starting aromates<sup>9</sup> have shown that substitution of the aromatic, or heteroaromatic ring takes place in some cases at the point of the greatest concentration of electrons, whilst in other cases at the point of the minimum steric hindrance. This steric hindrance can be caused by formation of a bulky complex of the solvent with the reagent and catalyst.

## EXPERIMENTAL

Melting points were measured with a Kofler micro hot-stage. The <sup>1</sup>H-NMR spectra ( $\delta$ , ppm) were recorded with a Tesla BS 487 C instrument operating at 80 MHz. The electron impact mass spectra ( $m/z$ ) were taken with a MS 902 S (AEI, Manchester) apparatus at an ionizing ion energy 70 eV, and 100  $\mu$ A trap current. The UV spectra of methanolic solutions ( $\lambda_{max}$  in nm (log  $\epsilon$ )) were measured with a Specord UV-VIS (Zeiss, Jena) spectrophotometer. Dielectric constants of benzene solutions were recorded at  $25^\circ \pm 0.05^\circ\text{C}$  with a Dipolmeter WTW, type DMO1 (FRG). The total polarization  $P_2^\infty$  was obtained by the Halverstadt-Kumler method<sup>10</sup>

TABLE IV  
Polarization data and dipole moment of derivative *Ila*

Concentration range $100w_2$	$\alpha^a$	$\epsilon_{10}^b$	$\beta^c$	$v_{10}^d$	$P_{2^\infty}$ $\text{cm}^3$	$R_{D3}$ $\text{cm}^3$	$\mu$ D
0.00365–0.114	6.23	2.275	–0.03	1.4376	516.94	95.6	4.52

$$^a \alpha = d\epsilon_{12}/dw_2, \quad ^b \epsilon_{10} = \lim_{w_2 \rightarrow 0} \epsilon_{12}, \quad ^c \beta = dv_{12}/dw_2, \quad ^d v_{10} = \lim_{w_2 \rightarrow 0} v_{12}$$

by extrapolation. Polarization ( $P_e + P_a$ ) was determined as  $1.05R_D$  (ref.<sup>7</sup>),  $R_D$  (molar refraction) was calculated by addition of the additive increments<sup>11</sup>. Following moments of groups<sup>6</sup> were employed:  $\mu_{NO_2} = 4.00$  D,  $\mu_{Br} = 1.57$  D and  $0.67$  D for furan<sup>12</sup>.

(*E*)-2-Substituted 1-(5-Nitro-2-furyl)ethylenes *I*a–*I*l

The corresponding aromate (10 mmol) in 1,2-dichloroethane (20 ml) was added to a mixture of 2-bromo-1-(5-nitro-2-furyl) ethylene (2.18 g, 10 mmol) and aluminium chloride (2 g, 15 mmol) in 1,2-dichloroethane (20 ml) with stirring at 0°C. The mixture was poured into crushed ice (20 g), the organic layer was separated, washed with water, 5% sodium carbonate and dried over  $CaCl_2$ . The solvent was removed under diminished pressure and the residue chromatographed on a silica gel column with benzene, or chloroform as eluents according to the polarity of products. Following substances were synthesized according to this general procedure:

2-(1-Bromo-2-naphthyl)-1-(5-nitro-2-furyl)ethylene (*I*a) from 1-bromonaphthalene. UV spectrum: 212 (4.56), i242 (4.42), i261 (4.15), 320 (4.06), 403 (4.40).

2-(5-Acenaphthenyl)-1-(5-nitro-2-furyl)ethylene (*I*b), from acenaphthene. UV spectrum: 210 (4.65), 233 (4.57), i264 (4.28), 326 (4.16), 428 (4.43). Mass spectrum: 291 ( $M^+$ , 100), 274 (7), 259 (15), 258 (15), 245 (28), 244 (14), 227 (15), 217 (40), 216 (23), 215 (45), 203 (15), 202(53), 191 (8), 190 (18), 189 (38), 178 (14), 165 (6), 152 (23), 149 (14), 115 (4), 108 (4), 95 (5), 89 (6) 76 (7), 63 (13), 51 (7), 39 (16).

2-(9-Phenanthryl)-1-(5-nitro-2-furyl)ethylene (*I*c), from phenanthrene. UV spectrum: 210 (4.57), 251 (4.67), 311 (4.13), 403 (4.36). Mass spectrum: 315 ( $M^+$ , 81), 285 (4), 283 (12), 269 (40), 268 (16), 251 (23), 241 (81), 240 (54), 239 (100), 237 (14), 226 (19), 215 (21), 213 (16), 203 (19), 202 (33), 200 (16), 189 (8), 187 (7), 176 (11), 165 (5), 163 (5), 149 (16), 120 (14), 113 (7), 101 (17), 88 (8), 78 (8), 63 (8), 51 (7), 39 (13).

2-(2-Pyrenyl)-1-(5-nitro-2-furyl)ethylene (*I*d), from pyrene. UV spectrum: 205 (4.60), 234 (4.78), i267 (4.41), i293 (4.32), 352 (4.45), 443 (4.54). Mass spectrum: 339 ( $M^+$ , 100), 309 (5), 307 (31), 293 (23), 275 (19), 265 (62), 264 (42), 263 (69), 252 (8), 239 (19), 227 (31), 226 (46), 215 (4), 202 (8), 200 (9), 132 (14), 113 (27), 105 (6), 95 (6), 77 (10), 69 (11), 63 (5), 51 (7), 39 (8).

2-(2-Chryzenyl)-1-(5-nitro-2-furyl)ethylene (*I*e), from chryzene. UV spectrum: 201 (4.70), 222 (4.66), 267 (4.85), 420 (4.40). Mass spectrum: 365 ( $M^+$ , 100), 319 (10), 318 (10), 291 (40), 290 (38), 289 (73), 287 (16), 277 (32), 276 (35), 265 (7), 263 (13), 252 (17), 250 (11), 226 (8), 145 (11), 138 (6), 126 (11), 113 (7), 78 (7), 59 (14), 39 (7).

2-(3-Fluorenyl)-1-(5-nitro-2-furyl)ethylene (*I*f), from fluorene. UV spectrum: 209 (4.59), i224 (4.33), 318 (4.41), 421 (4.56). Mass spectrum: 303 ( $M^+$ , 100), 286 (7), 273 (9), 271 (14), 257 (25), 256 (15), 245 (10), 239 (18), 229 (64), 228 (59), 227 (32), 226 (32), 217 (11), 216 (10), 215 (16), 203 (18), 202 (39), 189 (27), 178 (5), 176 (6), 165 (18), 163 (13), 152 (8), 149 (13), 139(5), 114 (13), 101 (27), 95 (7), 88 (12), 76 (7), 63 (10), 51 (6), 39 (10).

2-(3-Guaiazulenylyl)-1-(5-nitro-2-furyl)ethylene (*I*g), from guaiazulene. UV spectrum: 211 (4.26), 251 (4.33), 316 (4.38), i368 (4.11), 524 (4.42). Mass spectrum: 335 ( $M^+$ , 100), 320 (8), 305 (6), 303 (10), 289 (16), 288 (16), 274 (8), 261 (13), 260 (16), 259 (14), 247 (25), 246 (25), 233 (14), 232 (44), 231 (28), 229 (16), 219 (28), 217 (19), 215 (25), 205 (16), 204 (28), 203 (44), 202 (38), 191 (19), 189 (28), 179 (16), 178( 19), 165 (25), 152 (14), 149 (9), 115 (9), 101 (12), 95 (7), 89 (10), 77 (12), 69 (11), 65 (6), 63 (6), 51 (10), 39 (17).

2-(3-Dibenzofurylyl)-1-(5-nitro-2-furyl)ethylene (*I*h), from dibenzofuran. UV spectrum: i207 (4.42), 225 (4.50), 248 (4.48), 256 (4.49), 288 (4.56), 407 (4.49). Mass spectrum: 305 ( $M^+$ , 100),

273 (30), 272 (26), 259 (27), 258 (17), 247 (11), 244 (12), 232 (20), 231 (86), 229 (20), 219 (14), 218 (11), 205 (36), 203 (21), 202 (53), 201 (12), 200 (14), 193 (9), 189 (9), 181 (8), 176 (12), 165 (24), 163 (18), 149 (26), 139 (17), 115 (6), 101 (10), 88(21), 75 (10), 63 (12), 51 (9), 39 (18).

2-(3-Indolyl)-1-(5-nitro-2-furyl)ethylene (IIi), from indole. UV spectrum: 217 (4.48), 287 (4.02), 307 (3.98), 455 (3.93).

2-(1-Methyl-3-indolyl)-1-(5-nitro-2-furyl)ethylene (IIj), from methylindole. UV spectrum: 205 (4.43), 232 (4.39), 280 (4.10), 314 (4.19), 467 (4.40).

2-(1-Butyl-3-indolyl)-1-(5-nitro-2-furyl)ethylene (IIk), from N-butylindole. UV spectrum: 206 (4.37), 234 (4.34), 289 (4.04), 312 (4.15), 470 (4.36).

2-(3-Carbazolyl)-1-(5-nitro-2-furyl)ethylene (III), from carbazole. UV spectrum: 202 (4.51), 210 (4.27), 238 (4.48), 295 (4.48), 318 (4.19), 440 (4.41).

1-(5-Nitro-2-furyl)-2-bromo-1,2-endo-(9,10-dihydroanthranlyl)ethane (III), from anthracene. Yield 3.2 g (80.8%), m.p. 123–126°C. For  $C_{20}H_{14}BrNO_3$  (396.2) calculated: 60.63% C, 3.56% H, 3.54% N, 20.17% Br; found: 60.46% C, 3.51% H, 3.76% N, 20.45% Br. UV spectrum: 208 (4.75), 322 (4.18).  $^1H$ -NMR spectrum ( $CDCl_3$ , tetramethylsilane): 5.74 (d,  $H_3$ ), 7.05 (d,  $J_{3,4} = 3.9$  Hz,  $H_4$ ), 3.56 (dd,  $J_{a,b} = 4.5$  Hz,  $H_a$ ), 4.20 (dd,  $H_b$ ), 4.49 (d,  $J_{a,c} = 2.3$  Hz,  $H_c$ ), 4.60 (d,  $J_{b,d} = 2.6$  Hz,  $H_d$ ), 7.49–7.09 (m,  $H_{1,2,4}$ ).

2-(2-Naphthyl)-1-(5-nitro-2-furyl)ethylene was prepared from naphthalene in various solvents: 1) In carbon disulfide at 15°C ( $CS_2$  does not form a complex with the catalyst); yield 0.2 g, (7.5%). 2) In nitrobenzene at 9°C; yield 0.3 g (11.3%). 3) In nitromethane at 0°C; yield 0.43 g (16.2%). 4) In dichloromethane at -80°C; yield 0.16 g (6.2%).

#### REFERENCES

1. Hrabovský J., Kováč J.: This Journal 44, 2096 (1979).
2. Hrabovský J., Kováč J.: This Journal, in press.
3. Kukushima M., Espinosa J., Valenta Z.: Can. J. Chem. 54, 3304 (1976).
4. Nesmeyanov A. N., Rybinskaya M. I., Kochetkov N. K.: Izv. Akad. Nauk SSSR, Otd. Khim. Nauk 1955, 817.
5. Baddeley G.: J. Chem. Soc. 1949, 99.
6. Exner O.: *Dipole Moments in Organic Chemistry*, p. 39. Thieme, Stuttgart 1975.
7. Gruttadauria S., Pappalardo G. C., Scarlata G., Torre M.: J. Chem. Soc., Perkin 2, 1974, 1580.
8. Pretsch E., Clerc T., Seibl J., Simon W.: *Tabellen zur Strukturklärung Organischer Verbindungen mit Spektroskopischen Methoden*. Springer, Berlin—Heidelberg 1976.
9. Dewar M. J. S., Uech D. S.: J. Chem. Soc. 1958, 3079.
10. Halverstadt I. F., Kumler W. D.: J. Amer. Chem. Soc. 64, 2998 (1942).
11. Vogel A. I., Cresswell W. T., Jeffery G. H., Leicester J.: J. Chem. Soc. 1952, 514.
12. McClellan A. L.: *Tables of Experimental Dipole Moments*. Freeman, San Francisco 1963.

Translated by Z. Votický.