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BENZINDOLES. 21.\* NITRATION OF 3-FORMYL[4,5]-AND 3-FORMYL[6,7] BENZINDOLES

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It was established that products of monosubstitution of 4-nitro- and 7-nitro-3-formyl[4,5] benzindoles and products of disubstitution of 4,8-dinitro- and 7,8-dinitro-3-formyl[4,5] benzindoles are formed in low yields in the nitration of 3-formyl[4,5]benzindole with sodium nitrate in concentrated sulfuric acid. Similar nitration of 3-formyl[6,7]benzindole leads to 9-nitro- and 5,6-dinitro-3-formyl[6,7]benzindoles. The 3-formylnitrobenzindoles obtained were converted to nitrovinyl derivatives by condensation with nitromethane.

The regularities in the behavior of indole in electrophilic substitution reactions, particularly in nitration, have been studied extensively and correlated [2, 3], and the nitration of 3-formylindoles has also been studied [4-6]. However, the introduction of an additional benzene ring in the indole molecule has a substantial effect on the course of the reaction [7-9]. Not enough information is available in the literature to enable one to draw an unambiguous conclusion regarding the behavior of condensed indole structures in electrophilic substitution reactions, the orientation of the substituents, and the possibilities for their preparative utilization.

The aim of the present research was to study the behavior of 3-formyl[4,5]- (I) and 3-formyl[6,7]benzindole (II), which were obtained by modified methods [10, 11], in nitration and the possibilities of the preparation of 3-formylbenzindoles with a nitro group in the benzene ring. Considering the fact that the nitration of indole derivatives in weak acids leads to side reactions (addition to the  $C_2-C_3$  multiple bond, replacement of an acyl group by a nitro group, etc.), we carried out the nitration in concentrated sulfuric acid. Sodium nitrate was used as the nitrating agent.

Complex mixtures of compounds are formed as a result of nitration both in the case of 3-formyl[4,5] benzindole (I) and in the case of 3-formyl[6,7]benzindole (II). We were able to isolate four compounds, two of which are monosubstituted compounds, viz., 4-nitro- (III) and 7-nitro-3-formyl[4,5]benzindole (IV), and two disubstituted compounds, viz., 4,8-dinitro- (V) and 7,8-dinitro-3-formyl[4,5]benzindole (VI), from the products of the nitration of benzindole I.



We were able to isolate only two stable compounds, viz., 9-nitro- (VII) and 5,6-dinitro-3-formyl[6,7] benzindole (VIII), from the products of the nitration of benzindole II.

<sup>\*</sup>See [1] for Communication [1].

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Thus, in the case of nitration of the aldehydes indicated above, the substituent (the nitro group) in the monosubstitution products is incorporated in the second benzene ring. The orientation of the substituent is the same as in the nitration of naphthalene, i.e., in the  $\alpha$  position. The formation of two products of nitration with a substituent in the  $\alpha$  position is possible for an unsymmetrically substituted naphthalene, and this is actually realized in the case of 3-formyl[4,5]benzindole. In addition, the possibility of electrophilic attack on the second benzene ring to give disubstitution products is realized. An analogy with naphthalene is also observed here. As in the nitration of 2,3-dimethyl[6,7]benzindole [1], the formation of only one mononitro derivative was noted in the case of 3-formyl[6,7]benzindole (II).

The low yields make it impossible to draw an unambiguous conclusion as to whether only substitution products are formed under the nitration conditions. However, the other components of the reaction mixture were unstable and decomposed during isolation.

The nitro-3-formylbenzindoles (III and V-VIII) obtained were converted to nitrovinyl derivatives IX-XIII in 44-64% yields by condensation with nitromethane.



The structures of the compounds were established on the basis of the results of elementary analysis and data from IR, PMR, and mass spectroscopy (Tables 1-3).\* The UV spectra were not recorded because of the low solubilities of the compounds obtained.

The chemical shifts and spin-spin coupling constants (SSCC) in the PMR spectra of the products of the nitration of benzindoles I and II are presented in Table 2. The assignment of the signals of the protons in all of the investigated compounds was made by comparison with the spectra of starting benzindoles I and II [12] and from a prior evaluation of the multiplicity and chemical shifts of the signals of each proton for all possible cases of replacement of the protons by a nitro group [1]. The integral intensities of the signals of the protons in the PMR spectra of III and IV correspond to eight protons and monosubstitution. From a prior

\* The mass spectra are not discussed in this paper.

Compound		IR spectru	R <sub>t</sub> a	M+		
	NH	сон	N	iO <sub>2</sub>	,	
III IV VI VII VII	3230 3115 3340 3340 3420 3435 <b>c</b> 3400	1650 1658 1670 1650 1675 1680 1660	1525 1520 1520 1520 1545—1520 1545 1545 1500	1380—1340 1360—1310 1350—1320 1350—1320 1370—1330 1370—1330 1370—1330 1390—1335	0,24 0,35 0,30 0,26 0,81 0,71	240 240 - b - b 240 285

TABLE 1. Characteristics of Nitro-3-formylbenzindoles III-VIII

<sup>a</sup> Elution with benzene-acetone (4:1). <sup>b</sup> The mass spectrum does not contain a molecular-ion peak. <sup>c</sup> IR spectrum of the compound in CHCl<sub>3</sub>.

Com-	I-Н	2-H	сно	4-H	5-H	6•H	7-H	8-H	9-H	Δδ <sup>8,9</sup> , Hz	Δδ <sup>4,5</sup> , Hz	J, Hz
I	12,51	8,35	9,96	9,56	7,46	7,46	7,91	7,63	7,70	6,6		$J_{45} = 7.5;  J_{46} = 1.9;  J_{56} = 6.9;$
111	12,60	8,29	9,72	-	8,17	7,52	8,28	7,82	7,90	7,9		$J_{56} = 7,5;  J_{57} = 1,2;  J_{67} = 8,3;$
IV	12,82	8,52	9,96	10,00	7,71	8,12		8,06	7,83	14,3		$J_{45} = 8,3;  J_{46} = 1,2;  J_{48} = 0,5;$
V	13,08	8,60	9,75		8,34	7,74	8,60		8,56			$J_{56} = 7,5; J_{89} = 9,0$ $J_{66} = 7,5; J_{57} = 1,2; J_{67} = 8,3$
II	12,89	8,30	9,98	8,16	7,85 7,64	8,26 7,95	7,51	7,51	8,74 8,38	-	52,3	$J_{45} = 8,5; J_{46} = 1,4; J_{56} = 7,5$ $J_{45} = 8,5; J_{67} = 8,1; J_{68} = 1,3;$
												$J_{78} = 6.9; J_{79} = 1.3; J_{89} = 8.1; J_{14} = 0.5$
VII	11,64	8,25	10,04	8,40	7,86	8,35	7,58	8,21			53,4	$J_{14} = 0,4;  J_{45} = 8,5;  J_{67} = 8,7;$
VIII	12,14	8,40	10,06	9,02		—	8,32	7,79	<b>8</b> ,66			$J_{14} = 0.5;  J_{78} = 7.7;  J_{79} = 1.2;$
							i					$J_{89} = 8,7$

TABLE 2. PMR Spectra of Nitro Derivatives of 3-Formyl[6,7] benzindoles (I-VIII)

TABLE 3. Characteristics of Nitrovinyl Derivatives IX-XIII

Compound		IR sp	R /a			
	NH	C = C	N	O <sub>2</sub>		1484
IX X XI XII XIII	3410 3425 3380 3400 3410	1620 1640 1625 1630 1630	1530—1490 1540—1510 1540—1510 1540—1500 1550—1500	1340—1300 13301300 13601280 13501300 13701320	0,3 0,34 0,27 0,93 0,71	283 328 b 283 c

a Elution with benzene-acetone (4:1). <sup>b</sup> The mass spectrum does not contain a molecular-ion peak. <sup>c</sup> The results of elementary analysis are presented in the Experimental Section.

evaluation of the spectra of these compounds they can be assigned to monosubstituted (in the 4 or 7 position, respectively, of benzene ring B) compounds. A comparison of the chemical shifts and multiplicities of the protons of the B ring in the spectra of these compounds with those of the starting 3-formyl[4,5]benzindole shows that the multiplet signal with a chemical shift of 10.0 ppm in the spectrum of IV belongs to the 4-H proton. This signal has constants of spin-spin coupling with the protons of benzene ring B of  $J_{45} = 8.3$  and  $J_{46} = 1.2$  Hz and a small constant of 0.5 Hz of coupling with one of the protons of benzene ring A, which evidently form an AB system with the 8-H proton, as in substituted naphthalenes [13-15]. The signal with a chemical shift of 8.12 ppm has a smaller ortho constant than the signal of the 4-H proton. Noting that  $J_{56}$  is smaller for 3-formyl [4,5] benzindole than  $J_{45}$ , we assign the signal with a chemical shift of 8.12 ppm to the 6-H proton. The 0.43 ppm and 0.13 ppm changes in the chemical shifts of the 8-H and 9-H protons, respectively, as compared with the chemical shifts of 3-formyl[4,5]benzindole confirms substitution in the 7 position in the case of IV. The lowest-field multiplet signal with a chemical shift of 8.28 ppm,  $J_{ortho} = 8.3 \text{ ppm}$ , and  $J_{meta} = 1.2$  Hz in the spectrum of III cannot, as in the case of IV, be assigned to the 4-H proton on the basis of a comparison with the chemical shift of the 4-H proton in the spectrum of 3-formyl[4,5]benzindole (9.56 ppm). We therefore assign this signal to the 7-H proton and the other multiplet signal at 8.17 ppm with  $J_{ortho} = 7.5$  Hz to the 5-H proton. An uncertainty remains in the assignment of the signals of the 8-H and 9-H signals, which form an AB system, because of the small difference in their chemical shifts. However, the assignment made seems correct to us if it is assumed that the nitro group in the B ring changes the chemical shift of the closest proton of the adjacent ring, i.e., the 8-H proton, to the greatest extent.

The integral intensities of the signals of the protons in the spectra of V and VI correspond to seven protons and disubstitution. The signals of the protons of the B ring of these compounds have identical multiplicities as compared with the signals of the analogous protons in the spectra of III and IV. The chemical shift of the proton of the formyl group undergoes the most pronounced change in the case of V, as in the case of III. This provides a basis for the assumption that the nitro group in V is located in the 4 position. The simultaneous presence of a multiplet signal at 10.06 ppm and the slight change in the chemical shift of the formyl proton in the spectrum of VI make it possible, as in the case of IV, to assign substitution of the proton in the 7 position by a nitro group. In addition, the spectra of V and VI contain a singlet with an intensity of one proton unit. It hence follows that replacement of a proton by a nitro groups takes place not only in the B ring but also in the A ring in either the 8 or 9 position. The absence in the spectrum of VI of the constant of spin-spin coupling of the 4-H proton with the 8-H proton that is characteristic for substituted benzindoles indicates substitution in the 8 position. The chemical shifts of the 1-H proton in the spectra of V and VI virtually coincide, on the basis of which it may be assumed that the substitution in the indole part of these compounds is identical.

From the integral intensities of the signals of the protons, we assign the nitro derivatives (VII and VIII) of 3-formyl[6,7]benzindole to mono- and disubstituted compounds, respectively. A preliminary evaluation of the multiplicities and chemical shifts for VII suggests substitution in the 9 position. The change of more than 1 ppm in the chemical shift of the 1-H proton as compared with 3-formyl[6,7]benzindole constitutes evidence in favor of the proposed structure. The assignment of the multiplet signals [at 8.35 ppm ( $J_{ortho} = 8.7 \text{ Hz}$ ) and 8.21 ppm ( $J_{ortho} = 7.7 \text{ Hz}$ ) to the 6-H and 8-H protons, respectively, does not contradict this.

The signals of the indole protons in VII, which form an AB system, were assigned on the basis of the  $J_{14}$  constant that is characteristic for indoles [16] and benzindoles [12]. The distribution of the multiplet signals of the B ring in VIII indicates replacement of the proton in the 6 or 9 position by a nitro group. However, the change in the chemical shift of the 1-H proton as compared with the chemical shifts of 3-formyl[6,7] benzindole and VII makes it possible to assume substitution in the 6 position. The spectrum of VIII does not contain signals of an AB quartet of indole 4-H and 5-H protons but does contain a signal with a chemical shift of 9.02 ppm and  $J \approx 0.5$  Hz from coupling with the 1-H proton. This signal is evidently from the 4-H proton.

It should be noted that the proton in the para position relative to the nitro group has the greatest  $J_{ortho}$  constant in all of the investigated compounds. The signal of the 2-H proton in the investigated compounds is broadened (the half width is ~1 Hz at 100-Hz scanning). The rate of exchange of the pyrrole proton with the protons of the water that is present in the  $d_6$ -DMSO solvent is evidently such that this constant averages out to values less than 1 Hz. The assignment of the signal of the 2-H proton was made by means of an experiment involving double resonance in the case of superimposition of the resonance field on the 1-H proton.

The reaction mixtures were separated by thin-layer chromatography (TLC) on silica gel. In an attempt at separation on aluminum oxide with acetone-benzene as the eluent in the case of 3-formyl[6,7]benzindole we isolated a compound with a PMR spectrum in which we observed signals of vinyl protons at 6-8 ppm  $[\Delta\delta_{(AB)} = 116.8$  Hz; the  $\alpha$ -H and  $\beta$ -H signals are observed at 6.80 and 7.97 ppm] with a spin-spin coupling constant of 17 Hz, which is characteristic for their trans orientation. An absorption band at 1600 cm<sup>-1</sup>, which is related to the stretching vibrations of an exocyclic double bond, and the band of a carbonyl group at 1682 cm<sup>-1</sup>, in contrast to the band at 1660 cm<sup>-1</sup> that is present in the spectrum of the starting aldehyde, appear in the IR spectrum of this compound. All of these data, including the results of elementary analysis, indicate that crotonic condensation with acetone to give 5,6-dinitrobenzoskatylideneacetone (XIV) occurred.



## EXPERIMENTAL

The PMR spectra of solutions of the compounds in  $d_6$ -DMSO were recorded at 31.5°C with a Varian HA-100D high-resolution spectrometer with hexamethyldisiloxane as the internal standard. The IR spectra were recorded with a UR-10 spectrometer. The compounds were separated by thin-layer chromatography (TLC) on plates (12 by 18) in a loose layer of silica gel (L 40/100  $\mu$ , Chemapol, Czechoslovakia); the thickness of the adsorbent layer was 1.5 mm. An MKh-1303 mass spectrometer with direct introduction of the samples into the ion source at ionizing-electron energies of 16 to 50 eV was used for the mass-spectroscopic studies.

<u>3-Formyl[4,5]benzindole (I).</u> 1) A 3.0-ml (0.033-mole) sample of phosphorus oxychloride (d 1.675) was added dropwise with vigorous stirring at  $-5^{\circ}$ C to 2.5 ml (0.033 mole) of dimethylformamide (DMF) ( $d_4^{20}$  0.95), and 3 g (0.018 mole) of [4,5]benzindole, with mp 35-36°C, in 9 ml of DMF was added at  $-5^{\circ}$ C to the resulting complex. The mixture was then stirred at this temperature for 1 h and at room temperature for 1.5 h. The resulting precipitate was removed by filtration, washed with 4 ml of DMF, and dissolved in 500 ml of water. The mixture was decomposed with a 1 N solution of potassium hydroxide and air dried to give 1.79 g (51%) of

I with mp 195-196°C [mp 185-186°C (from alcohol) [10, 11] and 192°C (from aqueous methanol [17]). The DMF mother liquor was poured into 500 ml of water and was also decomposed with alkali. The precipitate was worked up as in the case described above to give 1.6 g (46%) of I with mp 183-185°C.

2) The reaction mixture was poured into 500 ml of water and decomposed with alkali as described above. The product was recrystallized from 50% aqueous ethanol to give 1.92 g (83.5%) of I with mp 190-191°C.

<u>3-Formyl[6,7]benzindole (II).</u> A 4,6-ml (0.05 mole) sample of phosphorus oxychloride ( $d_4^{20}$  0.675) was added dropwise with vigorous stirring at  $-5^{\circ}$ C to 3.85 ml (0.05 mole) of DMF ( $d_4^{20}$  0.95), and a solution of 4.56 g (0.027 mole) of [6,7]benzindole, with mp 179°C, was added to 27 ml of DMR was added to the resulting complex at  $-5^{\circ}$ C at such a rate that the temperature did not rise above 10°C. The mixture was stirred at room temperature for 1 h, and the precipitate was removed by filtration, washed with 6 ml of DMF, and dissolved in 1 liter of water. The aqueous mixture was decomposed with a 1 N solution of potassium hydroxide, and the solid was removed by filtration, washed with water until the wash waters were neutral, and air dried to give 4.88 g (92%) of formylbenzindole II with mp 172-173°C.

<u>Nitration of 3-Formyl[4,5]benzindole</u>. A 1.64-g (8.4 mmole) sample of I was dissolved in 15 ml of cold concentrated  $H_2SO_4$ , and a solution of 0.723 g (8.5 mmole) of sodium nitrate in 10 ml of concentrated  $H_2SO_4$  was added dropwise to the cooled (to  $-10^{\circ}$ C) solution at such a rate that the temperature did not rise above  $-8^{\circ}$ C. The mixture was stirred at this temperature for 20 min, after which it was poured into ice water, and the solid material was removed by filtration, washed with water until the wash waters were neutral, and air dried to give 1.87 g of a mixture of substances. A 1.07-g sample of this mixture was decomposed on plates with a loose layer of silica gel; for this, a solution of 10 mg of the substance in 0.2 ml of acetone was mixed with silica gel, and the mixture was air dried and applied in a 3-mm-widegroove at a distance of 1.5 cm from the lower edge of the plate (elution with diethyl ether). The colored bands of silica gel were removed, the identical fractions were combined, and the silica gel was washed with acetone. The acetone solution was evaporated, and the residue was dried over  $H_2SO_4$ . The fractions containing several spots were reseparated on plates by the method described above and recrystallized to give 28 mg of III, with mp 220-223°C (twice from absolute alcohol), 17 mg of IV, which decomposed at 270°C (from acetone), 20 mg of V, with mp 240-243°C (twice from absolute alcohol), and 60 mg of VI, which decomposed at 285°C (twice from absolute alcohol).

<u>Nitration of 3-Formyl[6,7]benzindole.</u> A 3-g (15.4 mmole) sample of II was dissolved in 50 ml of cold concentrated  $H_2SO_4$ , and a solution of 1.43 g (16.9 mmole) sodium nitrate in 17 ml of concentrated  $H_2SO_4$  was added to the solution at  $-14^{\circ}C$  at such a rate that the temperature did not rise above  $-10^{\circ}C$ . The mixture was stirred at  $-5^{\circ}C$ , after which it was poured into a mixture of ice and water, and the precipitate was removed by filtration, washed with water until the wash waters were neutral, and air dried to give 2.95 g of yellow crystals, which were identified as a mixture of reaction products. The mixture was separated by TLC on plates with a loose layer of silica gel. For this, 30-40 mg of the mixture was applied to a plate by the method described above. Elution with benzene-acetone (4:1) as in the preceding experiment gave 170 mg of nitro derivative VII, with mp 188-189°C (from acetone), and 160 mg of VIII, which decomposed at 280°C (from acetone).

Similar separation of a 1.36-g sample of a mixture of products obtained by nitration of II on plates with aluminum oxide [Brockmann neutral grade (Hungary)] in a benzene—acetone system (4:1) gave 0.24 g of 5,6-dinitro[6,7]benzoskatylideneacetone (XI), which decomposed at 275-276°C (from acetone) and had Rf 0.59. IR spectrum (KBr): 3290 (NH); 1682 (C=O); 1600 (C=C); 1520-1540, 1310-1360 cm<sup>-1</sup> (NO<sub>2</sub>). PMR spectrum (d<sub>6</sub>-DMSO): 11.93 (s, NH); 2.33 (s, COCH<sub>3</sub>); 6.80 and 7.97 ppm (AB q, J = 17 Hz,  $\Delta \delta$  = 116.8 Hz,  $\alpha$ -H and  $\beta$ -H); 8.15 (br s, 2-H); 9.10 (br s, 4-H); 8.28 (dd, 7-H); 7.75 (dd, 8-H); 8.61 ppm (dd, 9-H); J<sub>12</sub> = 0.3, J<sub>89</sub> = 8.4, J<sub>78</sub> = 7.6, and J<sub>79</sub> = 1.2 Hz. Found: C 59.2; H 3.6; N 13.0%. C<sub>16</sub>H<sub>11</sub>N<sub>3</sub>O<sub>5</sub>. Calculated: C 59.1; H 3.4; N 12.9%.

<u>4-Nitro-3-nitrovinyl[4,5]benzindole (IX)</u>. A mixture of 0.07 g (0.29 mmole) of III, 0.4 g (5.2 mmole) of ammonium acetate, and 7 ml of nitromethane was refluxed for 45 min, after which the solution was evaporated and the residue was washed with water and recrystallized from methanol to give 0.053 g (64%) of IX, which decomposed at 187°C. Chromatographic monitoring of the course of the reaction was realized here and subsequently.

4,8-Dinitro-3-nitrovinyl[4,5]benzindole (X). A mixture of 0.048 g (0.17 mmole) of V, 0.3 g (3.9 mmole) of ammonium acetate, and 10 ml of nitromethane was refluxed for 50 min, after which it was cooled, and the

crystals were removed by filtration, washed with water and nitromethane, air dried, and recrystallized from nitromethane to give 0.029 g (53%) of X, which decomposed at 200°C.

<u>7,8-Dinitro-3-nitrovinyl[4,5]benzindole (XI)</u>. A mixture of 0.1 g (0.35 mmole) of VI, 0.15 g (2.2 mmole) of ammonium acetate, and 10 ml of nitromethane was refluxed for 3.5 h, after which it was cooled, and the precipitate was removed by filtration, washed with water and nitromethane, air dried, and recrystallized from acetone to give 0.056 g (49%) of XI, which melted with decomposition at  $280-285^{\circ}$ C.

<u>9-Nitro-3-nitrovinyl[6,7]benzindole (XII)</u>. A mixture of 0.08 g (0.33 mmole) of VII, 0.1 g (1.3 mmole) of ammonium acetate, and 15 ml of nitromethane was refluxed for 2 h, after which it was evaporated in vacuo, and the residue was washed with water, air dried, and recrystallized from nitromethane to give 0.047 g (50%) of XII with mp 223-225°C.

<u>5,6-Dinitro-3-nitrovinyl[6,7]benzindole (XIII)</u>. A mixture of 0.07 g (0.24 mmole) of VIII, 0.053 g (0.7 mmole) of ammonium acetate, and 15 ml of nitromethane was refluxed for 35 min, after which it was cooled, and the crystals were removed by filtration, washed with water and nitromethane, air dried, and recrystal-lized from nitromethane to give 0.035 g (44%) of XIII, which melted with decomposition at 295°C. Found: C 51.0; H 2.6; N 17.2%.  $C_{14}H_8N_4O_6$ . Calculated: C 51.2; H 2.5; N 17.1%.

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