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BENZINDOLES.

17.* VILSMEIER REACTION IN THE ANGULAR TETRAHYDROBENZINDOLE SERIES

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The behavior of tetrahydro [4,5]- and tetrahydro [6,7] benzinodoles in the Vilsmeier reaction was investigated. It was established that the steric hindrance created by the cyclohexane ring condensed in the 4 and 5 positions has a substantial effect on the character of the reaction of tetrahydrobenzindoles with various N,N-disubstituted amines. A case of N-acylation in the investigated reactions is described. 3-Formy1-4,5,6,7-tetrahydro [4,5]- and 6,7,8,9-tetrahydro [6,7]- benzindoles were converted to the corresponding tryptamines.

The study of the Vilsmeier reaction in the case of angular benzindoles and their tetrahydro derivatives has made it possible to ascertain the effect of a condensed benzene ring on the reactivity of the 3 position. It has been previously established [2] that the yields of 3-acyl[4,5]-and 3-acyl[6,7]-benzindoles in the Vilsmeier reaction with various dimethylacylamines depend on both the electrophilicity of the attacking complex and on the distribution of electron density in the benzindole molecule. The electron density in the 3 position of [6,7]benzindole is somewhat higher than in the case of the [4,5] isomer [3]. It follows from Table 1 that the yields of 3-acyl[6,7]benzindoles are somewhat higher than the yields of 3-acyl[4,5] isomers: this was manifested particularly clearly in the reaction with the least reactive compound, N,N-dimethylacetamide: in the case of [4,5]benzindole no reaction at all occurred. On the basis of the different reaction times for the isomeric benzindoles with dimethylbenzamide (Table 1), it was concluded that in the case of attack by a bulky complex the benzene ring condensed in the 4 and 5 positions evidently creates steric hindrance.

This conclusion is confirmed by the results of a study of the Vilsmeier reaction with tetrahydro [4,5]and tetrahydro [6,7]-benzindoles, which differ little with respect to the magnitude of the electron density in the 3 position. For example, 6,7,8,9-tetrahydro [6,7] benzindole therefore reacts with N,N-diethylchloroacetamide under conditions similar to those for indole itself, and the yield of the corresponding 3-chloroacetyl derivatives is 60% in both cases. However, the yield of 3-chloroacetyl-4,5,6,7-tetrahydro [4,5] benzindole (V) falls to 42%, and this constitutes evidence for steric hindrance created by the hydrogenated benzene ring condensed in the 4 and 5 positions, as in the [4,5] benzindole series.

We have established that the reaction of tetrahydro [4,5] benzindole with N,N-dimethylbenzamide leads to two substances. Treatment of the reaction mixture with water precipitates IX, the IR spectrum of which does not contain absorption bands in the region of NH vibrations. When it was hydrolyzed with alcoholic alkali, it gave the starting tetrahydro [4,5] benzindole and benzoic acid. This enabled us to assign the N-benzoyl-4,5, 6,7-tetrahydro [4,5] benzindole structure to IX. The PMR spectrum does not contain the signal of protons of an NH group but does contain signals of 2-H (7.26 ppm) and 3-H (6.64 ppm) protons with characteristic spinspin coupling constant (SSCC) $J_{2,3} = 4.1$ Hz, as well as signals of 8-H and 9-H protons, which form an AB system with 7.00 ppm for 6-H and 7.90 ppm for 7-H, $J_{3,9} = 0.6$ Hz, $J_{8,9} = 8.00$ Hz, similar to what is observed for unsubstituted benzindole and its derivatives [3]. The considerable deshielding of the 7-H proton of the N-

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^{*}See [1] for communication 16.

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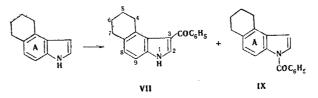
L.B	ę		Reaction conditions		mp, °C (crystall-	Found, %			Empirical	Calc.,%			d, %
Com-	Fusic	R	temp., °C	time	ization solvent)	с	H	N	formula	с	н	N	Yield,
I	4,5	н	35—37 (35—37)	20 min (30 min)	130—132 (aqueous alcohol)	78,3	6,6	7,1	C13H13NO	78,1	6,5	7,0	98 (86)
11	6,7	н	35—37 (35—37)	30 min (30 min)	160—162 (aqueous alcohol)	78,1	6,8	6,9	C ₁₃ H ₁₃ NO	78,1	6,5	7,0	93 (91)
III	4,5	CH3	50—60	13 h	231-232 (aqueous alcohol)	78,9	7,0	6,3	C14H10NO	78,9	7,1	6,6	36 (0)
IV	6,7	CH₃	35—40 (20)	6 h (24 h)	210-211 (aqeuous alcohol)	79,3	7,2	6,4	C ₁₄ H ₁₀ NO	78,9	7,1	6,6	89 (45)
v	4,5	CH₂Cl	60 (60)	1 h (30 min)	204206 (CHCl ₃)	67,4	5,4	5,6	C14H14CINO	67,9	5,7	5,7	42 (52)
VI	6,7	CH₂CI	60 (60)	1 h (30 min)	205—206 (CHCl ₃)	67,8	5,5	5,5	C14H14CINO	67,9	5,7	5,7	60 (74)
VII	4,5	C ₆ H ₅	70 (75)	2 h (30 min)	188—190 (alcohol)	82,7	6,4	4,9	C19H17NO	82,9	6,2	5,0	18 (63)
VIII	6,7	C ₆ H ₅	70 (75)	1 h (30 min)	257—258 (alcohol)	83,4	6, 3	4,9	C19H17NO	82,9	6,2	5,0	48 (78)
IX	4,5	C ₆ H ₅	70	2 h	145—146	83,1	6,5	4,7	C ₁₉ H ₁₇ NO	82,9	6,2	5,0	16

TABLE 1. Acyl Derivatives of Tetrahydrobenzindoles*

*The corresponding data for benzindoles are presented in parentheses.

benzoyl group (as compared with 6.69 ppm for unsubstituted tetrahydro [4,5]benzindole [5]) can be compared in magnitude with the change in the chemical shift of the 7-H proton under the influence of the N-acyl group in 1-benzoylindole [6] ($\Delta \delta = 1.2$ ppm) and in 1-acety1-2,3-dimethyl[6,7]benzindole ($\Delta \delta = 0.8$ ppm) as compared with the starting nitrogen-unsubstituted indoles. This constitutes evidence that the carbonyl group in all cases is oriented toward benzene ring A.

The C-alkylation product was isolated in 18% yield after alkalization of the aqueous solution. Essentially no instances of N-acylation under the conditions of the Vilsmeier reaction have been previously described for indoles [4].



The frequencies of the absorption of the carbonyl group (Table 2) are virtually in the same position as the band of the C=O group in the spectra of the analogous indole derivatives and benzindoles, for which this band is independent of the character of the fusion of the second benzene ring with the indole molecule. The difference between the spectra in the solid phase and in solution can be explained, as demonstrated for 3-acylindoles, by the presence of strong intermolecular bonds.

The signals of the 4-H protons in the [4,5] isomers in the PMR spectra of 3-acyltetrahydrobenzindoles (Table 3) are shifted to weaker field (to 3.20 ppm) as compared with the position of the signals of the 6-H protons in the analogous derivatives of the [6,7] isomer and with unsubstituted tetrahydro [4,5] benzindole, and this leads to the appearance of two broad triplets for the 4-H and 7-H protons. However, this shift to weak field of the signals of the 4-H protons is considerably smaller than the change in the chemical shift of the 4-H proton in the spectrum of 3-formyl [4,5] benzindole (as compared with unsubstituted benzindole: 9.56 and 8.15 ppm, respectively), for which a trans orientation of the pyrrole $C_2=C_3$ and aldehyde C=O bonds is proposed.

The deshielding of the 4-H protons observed in the spectra of 3-acyl-tetrahydro [4,5] benzindoles is therefore difficult to assign unambiguously to a transoid orientation of the double bonds and the deshielding effect of the oxygen atom of the keto group. In the case of a benzoyl group in the 3 position one common multiplet is recorded for the 4-H and 7-H protons, i.e., the certain degree of directed effect of the carbonyl

TABLE 2. IR Spectra of Acyl Derivatives of Tetrahydrobenzindoles

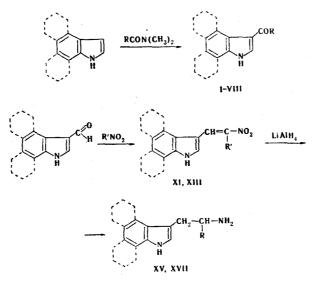
		J	v, cm ⁻¹									
Compound			in KBr pe	ellets	in solutions							
	Fusion	R	C=0	N-H		CHC13	tetrahy- drofuran		dioxane			
					C=0	N-H	C≔0	N—H	C=0	N-H		
VIII	4,5 6,7 4,5 6,7 4,5 6,7		1630 1640 1645 1615 1670 1665 1600—1615 1600 1675	3180-3240 3230 3150-3250 3250 3325 3160 3170-3210 3170-3270 -	1660 1660 1650 1670 	3270, 3460 3280, 3462 3465 3250, 3280 		3270 3270 		3300		

group is absent. Somewhat less deshielding of the 4-H protons is observed for 3-vinyl derivatives X-XIII. It is possible that in this case, as in the case of [4,5]benzoskatolideneacetones [7], a cisoid orientation of the double bonds is realized.

Considerable shielding is observed in the PMR spectra of 3-acyltetrahydro [6,7]benzindoles, whereas less shielding of the 4-H proton as compared with unsubstituted [5] and 3-aminoalkyl derivatives of this isomer is observed for 3-vinyl derivatives. These changes in the chemical shifts are of the same order of magnitude as the changes in the position of the signals from the 4-H proton on passing from unsubstituted [6,7]-benzindole to the 3-formyl derivative ($\Delta \delta = 0.55$ ppm) and to [6,7]benzoskatolideneacetones ($\Delta \delta 0.3-0.4$ ppm). In other words, a trans orientation of the double bonds is evidently characteristic for both types of substituents.

The existence of spin-spin coupling between the 4-H and 8-H and 7-H and 9-H protons of the tetramethylene chain and the benzene ring must be noted for [4,5]tetrahydrobenzindoles. This sort of coupling was not observed in the spectra of alkylindoles [6,8]. The existence of $J_{4,8}$ and $J_{7,9}$ constants was established for tetrahydro [4,5]benzindole derivatives. These constants are an order of magnitude wider than the lines for the 8-H and 9-H protons. Since the 6-H and 9-H protons for tetrahydro [6,7]benzindole derivatives give one complex multiplet in their spectra, one can only point to their joint coupling with both protons of the benzene ring.

3-Formyltetrahydro [4,5] and 3-formyltetrahydro [6,7] benzindoles I and II, which were synthesized by the Vilsmeier reaction, were similarly [9] condensed with nitroalkanes to give nitrovinyl derivatives of tetrahydrobenzindoles (X-XIII). The corresponding benzindolylalkylamines (XIV-XVII) were obtained by reduction of the latter.



ent)	Notes	$I_{1,2} \sim 0,5 \text{ Hz}, I_{1,5} = 8,2 \text{ Hz}$ $I_{4,5} = 8,0 \text{ Hz}, I_{1,4} \sim 0,5 \text{ Hz}, I_{1,2} = 2,8 \text{ Hz}$ $I_{1,2} = 2,4 \text{ Hz}, I_{4,5} = 8,0 \text{ Hz}$ $I_{1,2} = 2,4 \text{ Hz}, I_{4,5} = 8,0 \text{ Hz}$	$I_{1,2} < I,0$ Hz, $I_{4,5} = 8,2$ Hz	$I_{\beta+H-CH_3} = 0,7 \text{ Hz}, J_{1,2} < 0,5 \text{ Hz}, J_{2,H-CH_3} = 0,6 \text{ Hz}, J_{4,5} = 8,1 \text{ Hz}.$	$J_{1,2} = 2.4 \mathrm{Hz}$, $J_{4,5} = 8,0 \mathrm{Hz}$	$J_{1,2}=2,4$ HZ $J_{4,5}=8,2$ HZ,	Notes	$J_{8,9} = 8,0$ Hz $J_{1,2} = 2,8$ Hz, $J_{6,9} = 8,0$ Hz $J_{1,2} = 2,0$ Hz, $J_{8,9} = 8,0$ Hz $J_{1,2} = 3,2$ Hz, $J_{6,9} = 8,2$ Hz	/ _{1,2} <0,5 Hz	$J_{1,2} \sim 0.5 \mathrm{Hz}, J_{2.H-B-H} = 0.6 \mathrm{Hz}, J_{B-H-CH_3} = 0.6 \mathrm{Hz}$	$J_{1,2} = 2,4$ Hz, $J_{4,5} = 8,0$ Hz	$I_{\rm CHCH_{\circ}} = 6.4 {\rm Hz}_{\rm s} I_{1,2} = 2.4 {\rm Hz}_{\rm s}$ $I_{4.5} = 8.2 {\rm Hz}$
O as the Sol	H-8 H-2	1,76 1,75 1,77 1,76	1,75	1,76	1,74	1,75	5-H 6-H	1,71 1,67 1,69 1,68	1,76	1,75	ł	1,72
(with DMSC	H-6 H-8	2,78 2,77 2,80 2,81, 2,75	2,77	2,79	2,73	2,75	4-H 7-H	3,18, 2,74 3,14, 2,73 2,95 3,09, 2,74	3,04, 2,72	3,07, 2,75	1	3,02, 2,71
spunod	5-H	6,86 6,81 6,88 6,88 6,88	6,88	6,84	6,67	6,67	H-6	6,87 6,83 6,89 6,86	6,85	6,85	6,68	6,68
Com	4-H	7,74 7,82 7,89 7,80	7,56	7,44	7,21	7,24	8-H	7,15 7,11 7,11 7,12	7,15	7,17	7,0	7,02
Synthesized	ðR	$^{9,84}_{7,76-7,39}$	$\beta-H 8,30$ $\alpha-H 7,76$ $J_{\alpha,\beta} = 13,2 Hz$	β-H 8,34 α-CH ₃ 2,41	$\dot{\rm NH}_{\rm 3}$ ~ 8,0	${ m VH_{3}}_{{ m CH_{3}}}{ m 8,1}$ CH ₂ 2,97 CH 6,20	ôR	9,86 2,40 4,83 4,83	β -H 8,53 α -H 7,95 $J_{\alpha,\beta} = 13,0$ Hz	α-Η 2,38 β-Η 8,69	$^{+}_{\rm NH_{\rm s}}$ 8,0	ТН ₃ 8,0 СН ₃ 1,15 СН ₂ 3,12
f the	2-H	8,09 8,08 7,67 8,25	8,08	7,77	7,05	7,04	2-H	8,07 8,11 7,50 8,24	8,28	7,75	7,02	7,04
٥ (٥)	HN	11,84 11,56 11,75 11,78	11,92	11,89	10,64	1	ΗN	11,92 11,62 11,65 11,83	12,01	11,86	10,68	10,79
TABLE 3. Chemical Shifts (0) of the Synthesized Compounds (with DMSO as the Solvent)	3-R	CHO COCH3 COCGH5 COCGH2CI	CH=CHNO2	CH=GN02	CH2CH2NH2 · HCI	сн _з снин ₂ . нсі сн _з	3-R	CHO COCH ₃ COC ₆ H ₅ COCH ₂ CI	cH=CHNO ₂	$CH = C - NO_2$	CH2CH2NH2·HCI	CH ₂ CHNH ₂ ·HCI CH ₃
TABLE	6,7		×	XII	XIV	XVI	4,5		IX	IIIX	XV	XVII

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EXPERIMENTAL

The IR spectra of the compounds were recorded with a UR-10 spectrometer. The PMR spectra of solutions of the compound in d_6 -DMSO were recorded with a Varian HA-100D spectrometer with hexamethyldisiloxane as the internal standard.

3-Acyl-4,5,6,7-tetrahydro [4,5]- and 6,7,8,9-Tetrahydro [6,7] benzindoles (I-VI). These compounds were obtained by the method in [2]. The reaction conditions, the yields, and the results of elementary analysis are presented in Table 1.

<u>3-Benzoyl-4,5,6,7-tetrahydro [4,5]benzindole (VII) and 1-Benzoyl-4,5,6,7-tetrahydro [4,5]benzindole</u> (IX). A 0.5-g (2.9 mmole) sample of 4,5,6,7-tetrahydro [4,5]benzindole was added to the complex obtained from 0.68 g (4.5 mmole) of N,N-dimethylbenzamide and 0.46 g (4.0 mmole) of POCl₃, and the reaction mixture was heated at 75 deg C for 1 h. It was then treated with water and worked up to give 0.13 g (16%) of IX with mp 142-144 deg C (from alcohol). Found: C 83.1; H 6.5; N 4.7%. $C_{19}H_{17}NO$. Calculated: C 82.1; H 6.2; N 5.1%. Alkalization of the aqueous filtrate gave 0.14 g (18%) of VII with mp 188-190 deg C (from alcohol) (see Table 1).

Saponification of 1-Benzoyl-4,5,6,7-tetrahydro[4,5]benzindole (IX). A 0.2-g (1.34 mmole) sample of IX was refluxed for 1 h in 15 ml of a 5% alcohol solution of KOH, after which the alcohol was removed by distillation, and the residue was dissolved in water. The aqueous solution was extracted with ether, and the ether was removed from the extract by distillation to give 0.06 g (50%) of starting tetrahydro [4,5]benzindole with mp 93-94 deg C. The aqueous solution was acidified and extracted with ether, and the ether extract was worked up to give 0.05 g (50%) of benzoic acid with mp 120 deg C.

<u>3-(2-Nitroviny1)-6,7,8,9-tetrahydro[6,7]</u> benzindole (X). A mixture of 0.5 g (25 mmole) of acylbenzindole II, 0.04 g (5.0 mmole) of anhydrous ammonium acetate, and 0.72 ml [0.78 g (13 mmole)] of nitromethane was refluxed with stirring for 15 min, after which it was cooled to room temperature and treated with 0.5 ml of ethanol and 0.1 ml of water. The mixture was then stirred at 0-5 deg C for 30 min, and the precipitated X was removed by filtration, washed with aqueous ethenol, and dried to give 0.52 g (85%) of a product with mp 217-218 deg C (from absolute alcohol). IR spectrum (KBr): 1280-1310 (NO₂), 1480-1530 (NO₂), 1615 (CH= CH); 3110, 3440 cm⁻¹ (indole NH). Found: C 69.4; H 5.7; N 11.5%. C₁₄H₁₄N₂O₂. Calculated: C 69.4; H 5.8; N 11.6%.

<u>3-(2-Nitrovinyl)-4,5,6,7-tetrahydro[4,5]benzindole (XI).</u> Similarly, 0.46 g (75.5%) of XI, with mp 215-216 deg C, was obtained from 0.5 g (2 mmole) of acylbenzindole I, 0.04 g of ammonium acetate, and 0.72 ml of nitromethane after refluxing for 25 min. IR spectrum (KBr): 1275-1315 (NO₂); 1490, 1525 (NO₂); 1620 (CH=CH); 3110, 3300 cm⁻¹ (indole NH). Found: C 60.1; H 6.1; N 11.1%. $C_{14}H_{14}N_2O_2$. Calculated: C 69.4; H 5.8; N 11.6%.

<u>3-(2-Nitro-1-methylvinyl)-6,7,8,9-tetrahydro[6,7]benzindole (XII).</u> A mixture of 1.79 g (14 mmole) of acylbenzindole II, 7 ml of nitroethane, and 0.28 g of anhydrous ammonium acetate was refluxed at 125-130 deg C, after which it was cooled to room temperature and treated with 2.8 ml of ethanol and 0.7 ml of water. The mixture was cooled to 0-5 deg C, and the precipitated XII was separated and washed with aqueous ethanol (1:1) to give 3.1 g (83.3%) of a product with mp 200-202 deg C. IR spectrum (KBr): 1640 (CH=CH), 1480-1540 (NO₂), and 3290 cm⁻¹ (indole NH). Found: C 70.1; H 6.3; N 10.9%. C₁₅H₁₆N₂O₂. Calculated: C 70.3; H 6.3; N 10.9%.

 $\frac{3-(2-\text{Nitro-2-methylvinyl})-4,5,6,7-\text{tetrahydro}[4,5]\text{benzindole} (XIII).}{(11.6 \text{ mmole}) \text{ of acylbenzindole I, 5.4 ml of nitroethane, and 0.2 g of ammonium acetate. The yield was 2.35 g (79%), and the product had mp 209-210 deg C. IR spectrum (KBr): 1620 (CH=C) and 1500-1520 cm⁻¹ (NO₂). Found: N 10.8%. C₁₆H₁₆N₂O₂. Calculated: N 10.9%.$

<u>3-(2-Aminoethyl)-6,7,8,9-tetrahydro[6,7]benzindole (XIV).</u> A 1.82-g (7.5 mmole) sample of X in 20 ml of tetrahydrofuran (THF) was reduced with 1.82 g (4.8 mmole) of LiAlH₄ in 60 ml of THF. The amine was identified in the form of the hydrochloride. The yield of product with mp 277-279 deg C was 69.71%. IR spectrum (KBr): 2800-3500 (alkyl NH₂); 3285, 3425 cm⁻¹ (indole NH). Found: C 66.9; H 7.7; N 11.2%. C₁₄H₁₈N₂ · HCl. Calculated: C 67.1; H 7.6; N 11.2%.

<u>3-(2-Aminoethyl)-4,5,6,7-tetrahydro [4,5]benzindole (XV).</u> This compound was similarly obtained. The amine was identified in the form of the hydrochloride. The yield of product with mp 269-270 deg C was 60%. IR spectrum (KBr): 2600-3500 (alkyl NH₂); 3275, 3410 cm⁻¹ (indole NH). Found: C 67.1; H 6.8; N 10.9%. $C_{14}H_{18}N_2 \cdot HCl$. Calculated: C 67.1; H 7.7; N 11.2%.

<u>3-(2-Amino-2-methylethyl)-6,7,8,9-tetrahydro [6,7]benzindole (XVI).</u> This compound was obtained from 2.5 g (9.8 mmole) of XII and 2.5 g (66 mmole) of LiAlH₄ in 165 ml of THF. The yield of product with mp 140-144 deg C was 1 g (43.5%). IR spectrum (KBr): 3150-3200 (alkyl NH₂) and 3550 cm⁻¹ (indole NH). Found: N 12.5%. $C_{15}H_{20}N_2$. Calculated: N 12.3%. The amine was identified in the form of the hydrochloride. The yield of product with mp 259-261 deg C was 0.21 g (91.3%). IR spectrum (KBr): 3150-3200 (alkyl NH₂) and 3350 cm⁻¹ (indole NH). Found: C 69.1; H 8.0; N 10.3%. $C_{15}H_{20}N_2$ ·HCl. Calculated: C 69.7; H 7.9; N 10.6%.

<u>3-(2-Amino-2-methylethyl)-4,5,6,7-tetrahydro[4,5]benzindole (XVII).</u> This compound was obtained from 1.75 g (6.8 mmole) of XIII, 1.75 g (46 mmole) of LiAlH₄, and 115 ml of THF. The yield of product with mp 137-138 deg C was 0.62 g (39%). IR spectrum (KBr): 3020-3220 (alkyl NH₂) and 3350 cm⁻¹ (indole NH). Found: C 69.1; H 8.0; N 10.3%. $C_{15}H_{20}N_2 \cdot HCl$. Calculated: C 69.7; H 7.9, N 10.6%.

The amine was identified in the form of the hydrochloride. The yield of product with mp 272-273 deg C was 0.2 g (87%). IR spectrum (KBr): 3280 (alkyl NH₂) and 3310-3320 cm⁻¹ (indole NH). Found: N 10.2%. $C_{15}H_{20}N_2 \cdot HC1$. Calculated: N 10.6%.

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