in Table 2, were similarly obtained in quantitative yield.

2-Alkoxy-4-aziridino-5,5-dimethyldihydrofurans XII and XIII. A 1.13-g (7 mmole) sample of IVa was dissolved in 10 ml of absolute methanol or ethanol, and the solution was allowed to stand at room temperature for 12 h. The alcohol was removed by evaporation, and the residue was distilled in vacuo to give the products. A total of 1.16 g (98%) of 2-methoxy-4-aziridino-5,5-dimethyldihydrofuran (XII), with bp 28-30 deg C (0.01 mm), was obtained. Found: C 63.9; H 8.7; N 8.4%. $C_9H_{15}NO_2$. Calculated: C 63.9; H 8.9; N 8.3%. IR spectrum: 1655 cm^{-1} (C=C). PMR spectrum, δ: 5.45 (s, 1H, 2-H), 4.69 (s, 1H, 3-H), 3.31 (s, 3H, OCH_3), 1.81 [s, 4H, $N(CH_2)_2$], 1.38 (s, 3H, CH_3), and 1.30 ppm (s, 3H, CH_3). A total of 1.22 g (95%) of 2-ethoxy-4-aziridino-5,5-dimethyldihydrofuran (XIII), with bp 34-35 deg C (0.01 mm), was obtained. Found: C 65.3; H 9.3; N 7.3%. $C_{10}H_{17}NO_2$. Calculated: C 65.67; H 9.3; N 7.6%. IR spectrum: 1665 cm^{-1} (C=C). PMR spectrum, δ: 5.46 (s, 1H, 2-H), 4.64 (s, 1H, 3-H), 3.51 (m, 2H, OCH_2), 1.81 [s, 4H, $N(CH_2)_2$], 1.39 (s, 3H, 5-CH_3), 1.29 (s, 3H, 5-CH_3), and 1.14 ppm (t, 3H, CH_3).

Reaction of 2-Hydroxy-4-aziridino-5,5-dimethyldihydrofuran (VIa) with Alcohols. A 1.6-g (0.01 mole) sample of VIa was refluxed for 8 h in a solution (30 ml) of methanol or ethanol, after which the solvent was removed by evaporation, and the residue was identified by PMR spectroscopy as the corresponding 2-methoxy-4-aziridino-5,5-dimethyldihydrofuran (XII) [1.6 g (94%)] or, respectively, 2-ethoxy derivative XIII [1.7 g (91%)].

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SYNTHESIS AND REACTIONS OF BENZOFORMYLINDOLES

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A method was developed for the synthesis of 5- or 6-formylindoles by reaction of 5- or 6-aminomethylindoles with hexamethylenetetramine in acidic media. 1-Methyl-7-formylindole was obtained by recyclization of nicotyrine methiodide under the influence of alkaline agents. The corresponding vinylindoles were obtained by condensation of formylindoles with nitromethane, nitroethane, and malonic acid. The structures of the products were proved by alternative synthesis, the results of elementary analysis, and the UV, IR, PMR, and mass spectra.

In [1] it is shown that aldehydes of the indole series with a formyl group attached to the benzene ring may be convenient starting substances, for example, for the synthesis of aminoethylindoles. This is also true of aldehydes of the tetrahydrocarbazole series, although it has been reported that the carbonyl group in these compounds has extremely low reactivity [2]. Troxler synthesized such aldehydes by reduction of the corresponding nitriles with sodium hyposulfite in the presence of Raney nickel in acetic acid containing pyridine [1]. However, it is often necessary to use circuitous pathways to obtain the nitriles themselves, since the corresponding acids are difficult to convert to even the chlorides [2, 3]. The direct introduction of a formyl group in the benzene ring of the indole molecule is difficult. Only 5-methoxy-6-formylindoles have been obtained by this method [4].

Having samples of 5- and 6-aminomethylindoles [5, 6] at our disposal, we investigated the possibility of

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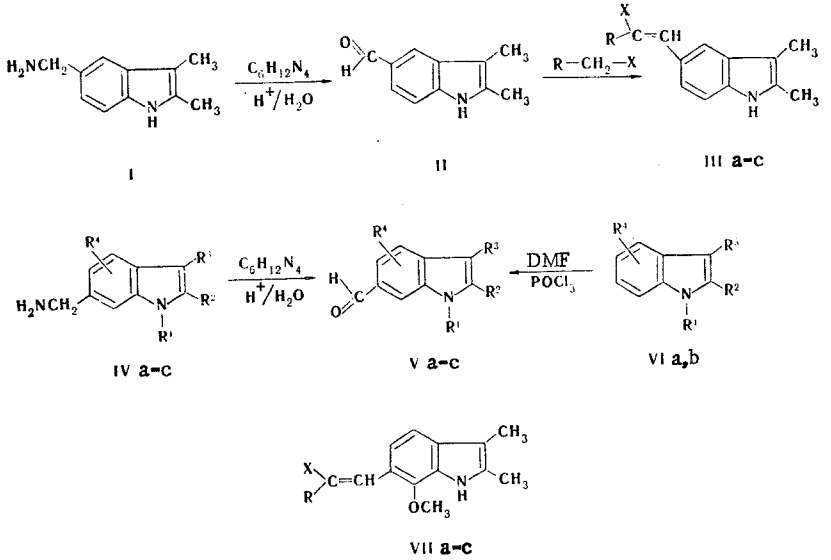
their conversion to aldehydes by means of the Sommelet reaction.* The applicability of this method was in doubt, since furfural could not be obtained from 2-aminomethylfuran [8], although the analogous synthesis of α-formylthiophene has been carried out [9].

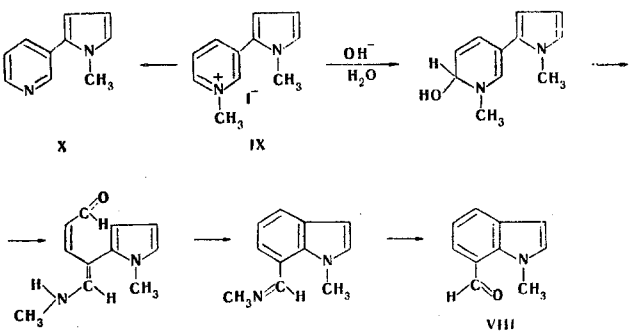


III a R=H, X=NO_2; b R=CH_3, X=NO_2; c R=H, X=CO_2H; IV, V, VI a R=H, R^2=R^3=CH_3, R^4=5-OCH_3; b R^1=H, R^2=R^3=CH_3, R^4=7-OCH_3; c R^1=R^2=CH_3, R^3=$COOC_2H_5$, R^4=5-OCH_3; VII a R=H, X=NO_2; b R=CH_3, X=NO_2; c R=H, X=CO_2H

Correspondingly, 4-formyl-2-phenylthiazole was synthesized in satisfactory yield, but the isomeric 2-formyl-4-phenylthiazole was not. The attempted synthesis of 2- and 4-formylpyridines was unsuccessful, but 3-formylpyridine was obtained in good yield [8]. The possibility of the synthesis of 2-carbethoxy-3-formylindole by means of the Sommelet reaction in the indole series has been reported [10], but 2-carbethoxy-4-formyl-5-hydroxyindole could not be obtained from 2-carbethoxy-4-dimethylaminomethyl-5-hydroxyindole [11].

Our experiments have shown that in neutral media aminomethylindoles (I or IV) virtually do not react with hexamethylenetetramine, that in glacial acetic acid the process gives the products in very low yields, and that the addition of acetic acid up to pH 5 does not have a substantial effect on the course of the reaction, but preparative yields of the aldehydes (up to 96%) are obtained in 50% acetic acid.

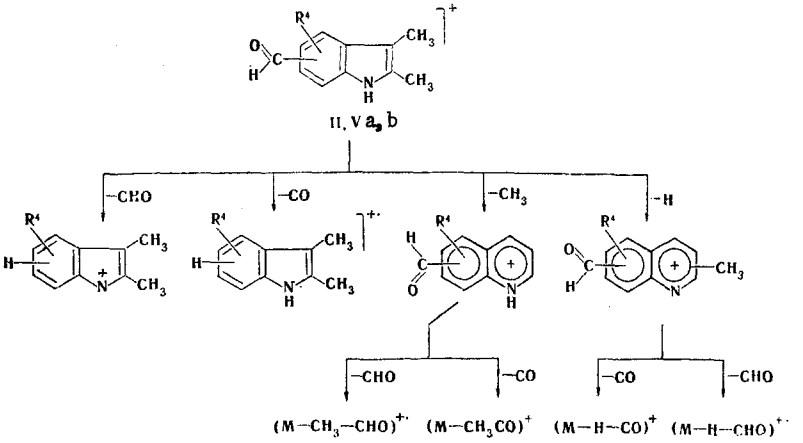
The compositions and structures of aldehydes II and V are confirmed by the results of elementary analysis and the spectroscopic characteristics. In addition, Va and Vb were synthesized independently (by Vilsmeier formylation), and aldehydes II and Vc were previously described. Let us note that the 3-carbethoxy group, which is usually easily eliminated during acid hydrolysis, was retained in the synthesis of Vc. We obtained 1-methyl-7-formylindole (VIII) by rearrangement of nicotyrine methiodide (IX) under the influence of alkaline agents [12].



The initially formed pseudo base readily undergoes ring opening, after which the formyl group attacks the electron-surplus pyrrole ring with the formation of the energically favorable benzene ring. The methylamine grouping is split out during the solvolytic reaction (it is possible that this occurs partially in the open form). Side attack by the nucleophile on the CH_3–N bond leads to demethylation to give nicotyrine X.

*A preliminary communication has been published [7].

The peak of the molecular ion, which subsequently undergoes fragmentation with elimination of a hydrogen atom, CH_3, CO, or CHO, is the most intense peak in the mass spectra of II and Va,c. The loss of hydrogen is a more intensive process in the case of II ($I_m = I_{m-1}$), whereas its peak does not exceed 0.2 of the molecular ion peak in the spectrum of Va. The M−H and M−CH_3 ions subsequently lose CO or CHO; this was confirmed by comparison with the mass spectra of compounds that contain ^{18}O in the formyl group. The loss of R^4 occurs only in the case of more profound stages of the fragmentation. These data make it possible to assume that the positive charge in the molecular ions of II and Va,b is localized primarily in the pyrrole part of the molecule, since the indicated character of the fragmentation differs markedly from the dissociative ionization of both 2- or 3-formylindoles [13] and 6- or 7-methoxyindoles [14].



The mass spectrum of aldehyde VIII differs from the mass spectra of aldehydes II and Va,b with respect to the virtual absence of M−CH_3 and M−CO ions and the intense M−H and M−H−CO ions. These processes are confirmed by metastable ions. In addition, an appreciable peak of an M−OH ion, the formation of which from the molecular ion is also confirmed by a metastable ion, is observed. This process is possibly associated with transfer of a hydrogen atom from the CH_3 group in the 1 position to the oxygen atom of the carbonyl group; this was observed in the spectra of 1-methyl-7-nitroindoles [14].

The condensation of aldehydes II and Vb with nitromethane, nitroethane, or malonic acid proceeds smoothly under the influence of alkaline agents. Two doublets of olefin protons (J = 14-15 Hz), which correspond to a trans configuration, appear in the PMR spectra of the resulting III. Nitro vinyl derivatives III and VII are stable during storage and are brightly colored. The absorption maximum (in alcohol) lies at 233, 285, and 435 nm; this is typical for ω-nitrostyrenes [15]. If there is a methoxy group in the benzene ring (VIIa, b), a bathochromic shift of the absorption band, as also noted in the case of 3-(2-nitrovinyl)-5-methoxyindole [16], is noted in the spectrum.

Intense peaks of molecular ions and the absence of M−H and M−CH_3 ions are characteristic for the mass spectra of IIIa,b and VIIa,b. This indicates primary localization of the charge in the benzene rather than the pyrrole part of the molecule, especially since the fragmentation of the molecular ions is similar to that observed for 5- and 6-nitroindoles [14] and is characterized by elimination of the NO_2, NO, or OH group. The loss of a methyl group was observed only in the case of (M−NO_2) or (M−OH−NO) ions.

EXPERIMENTAL

The UV spectra of methanol solutions of the compounds were recorded with a Specord spectrophotometer. The IR spectra of mineral oil suspensions of the compounds were recorded with IKS-22 or UR-20 spectrometers. The PMR spectra of DMSO (aldehydes II and Va), acetonitrile (Vb), and CCl_4 (Vc, VIII) solutions of the compounds were obtained with Varian T-60 and XL-100 PMR spectrometers with hexamethyldisiloxane as the external standard.

The mass spectra were recorded with an MKh-1303 spectrometer with introduction of the compounds directly into the ionization region at an ionizing-electron energy of 50 eV and at a temperature close to the melting point of the compound. Aldehydes II and Va,b, which contain ^{18}O, were obtained by shaking the corresponding aldehydes with $H_2^{18}O$ and alcohol in the presence of $ZnCl_2$.

2,3-Dimethyl-5-formylindole (II). A 0.5-g (3.5 mmole) sample of hexamethylenetetramine was added with stirring to a solution of 0.5 g (2.9 mmole) of 2,3-dimethyl-5-aminomethylindole (I) in 2.5 ml of 50% ace-

tic acid, and the mixture was refluxed with stirring for 2 h. It was then diluted with cold water to a volume of 10 ml, and the resulting precipitate was extracted with ether [three 10-ml portions; the process was monitored by TLC on aluminum oxide or Silufol, benzene–methanol (9 : 1)]. The extract was dried with magnesium sulfate, and the ether was removed by evaporation to give 250 mg (51%) of aldehyde II, which was purified by chromatography on silica gel [40/100 μ, elution with benzene–ethyl acetate (5 : 2)] to give a product with mp 138–139 deg C [mp 137–139 deg C (from benzene) [3]]. IR spectrum: 1680 (C=O) and 3200 cm^{-1} (N–H). PMR spectrum: 7.25 (d, 7H, $J_{6,7}$ = 8 Hz), 7.53 (d, 6H, $J_{6,7}$ = 8 Hz), 7.90 (s, 4H), 9.35 (broad s, 1-NH), and 9.90 ppm (s, 5-CHO). Mass spectrum:* 173 (100), 172 (82), 158 (22), 144 (22), 143 (15.5), 115 (10.5), 77 (11.8), 72 (11.8), 51 (23.8). Found: C 76.2; H 6.2%. $C_{11}H_{11}NO$. Calculated: C 76.3; H 6.4%.

2,3-Dimethyl-5-methoxy-6-formylindole (Va). Similarly, 110 mg (44%) of aldehyde Va, with mp 195–196 deg C [purified by chromatography in a thin layer of aluminum oxide with elution by benzene–methanol (9 : 1)], was obtained from 0.25 g (1.2 mmole) of 2,3-dimethyl-5-methoxy-6-aminomethylindole (IVa), 1.5 ml of 50% acetic acid, and 0.25 g (1.78 mmole) of hexamethylenetetramine after refluxing for 1.5 h. IR spectrum: 1665 (C=O) and 3300 cm^{-1} (N–H). PMR spectrum: 7.04 (s, 4-H), 7.67 (s, 7-H), 9.73 (s, 6-CHO), and 10.33 ppm (broad s, 1-NH). Mass spectrum: 203 (100), 202 (24), 188 (11), 186 (11), 174 (4), 172 (4), 159 (6), 158 (6), 157 (9), 156 (27), 144 (4), 143 (6), 132 (11), 131 (10), 97 (6), 83 (7). Found: C 70.9; H 6.4%. $C_{12}H_{13}NO_2$. Calculated: C 70.9; H 6.4%.

2,3-Dimethyl-6-formyl-7-methoxyindole (Vb). Similarly, a light-yellow precipitate, which was removed by filtration and washed with water, was obtained from 0.3 g (1.45 mmole) of 2,3-dimethyl-6-aminomethyl-7-methoxyindole (IVb) after refluxing for 3 h and dilution with water. The filtrate was extracted with ether (two 10-ml portions), and the extract was dried with anhydrous magnesium sulfate. The ether was removed by evaporation to give an additional amount of aldehyde Vb. The overall yield was 0.28 g (96%). After purification by preparative chromatography in a thin layer of aluminum oxide [elution with benzene–methanol (9 : 1)], the product had mp 183–184 deg C. IR spectrum: 1670 (C=O) and 3220 cm^{-1} (N–H). PMR spectrum: 6.90 (d, 4-H, $J_{4,5}$ = 8 Hz), 7.79 (d, 5-H, $J_{5,4}$ = 8 Hz), 10.49 (s, 6-CHO), and 11.49 ppm (broad s, 1-NH). Mass spectrum: 203 (100), 202 (50), 188 (23), 174 (5), 160 (5), 159 (10), 132 (13), 131 (7), 130 (6), 117 (6), 85 (11), 83 (17). Found: C 70.7; H 6.5%. $C_{12}H_{13}NO_2$. Calculated: C 70.9; H 6.4%.

1,2-Dimethyl-3-carbethoxy-5-methoxy-6-formylindole (Vc). Similarly, the precipitate obtained from 0.15 g (0.54 mmole) of 1,2-dimethyl-3-carbethoxy-5-methoxy-6-aminomethylindole (IVc) after refluxing for 3 h and dilution with water was separated, and the filtrate was extracted with ether (the degree of extraction was monitored by TLC) to give 70 mg (47%) of aldehyde Vc with mp 135.5–136.5 deg C (from alcohol) [4].

Formylation of 2,3-Dimethyl-7-methoxyindole. A 0.76-g (5 mmole) sample of freshly distilled phosphorus oxychloride was added dropwise with stirring at 0 deg C to a solution of 0.7 g (4 mmole) of 2,3-dimethyl-7-methoxyindole in 7.3 g (0.1 mole) of DMF, and the mixture was stirred at room temperature for 30 min and at 95 deg C for 3 h. It was then cooled to room temperature and poured over ice. The aqueous mixture was neutralized with sodium hydroxide solution to pH 6, and the resulting precipitate was extracted with ether. The ether extract was dried with sodium sulfate, the ether was removed by evaporation, and the residue was purified by preparative chromatography in a thin layer of aluminum oxide [benzene–methanol (9 : 1)]. The yield of aldehyde Vb, with mp 182–183.5 deg C, was 0.121 g (15%). The product was identical to that described above.

Similarly, 25 mg (6%) of Va, with mp 194–195 deg C, which was purified by TLC on Al_2O_3 [benzene–methanol (9 : 1)], was obtained from 350 mg (2 mmole) of 2,3-dimethyl-5-methoxyindole. The product was identical to that described above with respect to its melting point and IR spectrum.

1-Methyl-7-formylindole (VIII). A) A mixture of 150 mg (0.5 mmole) of nicotyrine methiodide (IX), with mp 211–213 deg C [17], 1 g of NaOH, 6 ml of water, and 20 ml of ethanol was heated in a stream of nitrogen on a water bath for 1 h, after which it was cooled, and the excess alcohol was removed by evaporation. Cold water (4 ml) was added to the residue, and the mixture was extracted with two 10-ml portions of benzene. The benzene extract was dried with calcined $MgSO_4$, and the benzene was removed by evaporation. The residue was purified by preparative chromatography on silica gel (100/160μ) [elution with benzene–ethyl acetate (10 : 4)] to give 10 mg (12%) of aldehyde VIII with mp 82–84 deg C. IR spectrum: 1690 cm^{-1} (C=O). UV spectrum (in methanol), λ_{max} (log ε): 226 (4.38), 247 (4.34), 340 nm (3.92). PMR spectrum: 3.30 (s, 1-CH_3), 5.65 (d, 3H, $J_{3,2}$ = 4 Hz), 6.15 (d, 2H, $J_{2,3}$ = 4 Hz), 6.32 (t, 5H, $J_{5,4}$ = $J_{5,6}$ = 8 Hz), 6.75 (q, 4H, $J_{4,5}$ = 8 Hz, $J_{4,6}$ = 2

*Here and subsequently, the most intense ion peaks are presented. The relative intensities in percent of the intensity of the molecular-ion peak are given in parentheses.

TABLE 1. Characteristics of the Nitrovinylindoles

Compound	Method	Solvent	Time	mp, deg C	IR spectrum, cm^{-1}				UV spectrum		Found, %		Empirical formula	Calc., %		Mass spectrum, m/e	Yield, %
					NO_2 sym	NO_2 asym	C=C trans	N–H	λ_{max}	lg ε	C	H		C	H		
IIIa	A	CH_3OH	7 days	153—154*	1340	1560	1630	3370	380	4,36	66,5	5,6	$C_{12}H_{12}N_2O_2$	66,6	5,5	216 (100), 171 (21), 168 (31), 159 (25), 154	33
	B	CH_3OH	5 days				980		285	4,20						(31), 145 (16), 144 (18), 143 (21), 129	58
	C	CH_3NO_2	1 h 30 min						237	4,44						(22), 115 (24), 77 (29)	85
IIIb	A	C_2H_5OH abs.	5 days	156—157 (Nitroethane)	1380	1510	1645 990	3340	370 287	4,11 4,14	68,2	6,2	$C_{13}H_{14}N_2O_2$	68,4	6,1	230 (100), 213 (8), 200 (6), 199 (6), 198 (5), 197 (5), 185 (12), 183 (24), 182 (20), 169	60
	C	$CH_3CH_2NO_2$	3 h						237	4,46						(45), 168 (50), 160 (20), 145 (25), 144 (12), 115 (24), 81 (23)	87
VIIa	B	C_2H_5OH abs.	20 days	227—228 (ethanol)	1335	1555	1670 975	3320	457 275	4,20 4,14	63,5	5,8	$C_{13}H_{14}N_2O_3$	63,4	5,7	246 (100), 229 (4), 214 (4), 200 (10), 199 (52), 198 (18), 185 (9), 184 (33), 183 (8),	31
	C	CH_3NO_2	5 h						233	4,46						170 (9), 168 (9), 156 (15), 154 (10), 115 (15), 89 (16)	90
VIIb	A	CH_3OH	17 days	183—184*	1355	1550	1670	3335	435	3,93	64,8	6,2	$C_{14}H_{16}N_2O_3$	64,6	6,3	260 (100), 243 (2), 230 (2), 215 (5), 214	30
	C	$CH_3CH_2NO_2$	6 h				950		272 231	4,11 4,55						(10), 213 (50), 199 (12), 198 (40), 185 (13), 184 (22), 175 (12), 170 (10), 168 (10), 115 (8), 77 (7)	86

*Compounds IIIa and VIIb were purified by TLC on silica gel (5/40 μ, elution with benzene–methanol 9 : 1).

Hz), 6.95 (q, 6H, $J_{6,5}$ = 8 Hz, $J_{6,4}$ = 2 Hz), and 9.22 ppm (s, CHO). Found: C 75.2; H 5.9%. $C_{10}H_9NO$. Calculated: C 75.5; H 5.7%. Mass spectrum: 159 (100), 158 (94), 130 (90), 102 (15), 89 (22), 77 (60), 76 (25), 75 (22), 63 (25), 51 (25).

B) A mixture of 150 mg (0.5 mmole) of methiodide IX, 3 ml of methylammonium sulfate, and 6 ml of a 30% aqueous solution of methylamine was heated in a sealed ampule (in an autoclave) at 150 deg C for 70 h, after which it was extracted with two 7-ml portions of benzene. The extract was dried with $MgSO_4$, and the benzene was removed by evaporation. The residue was purified as in method A to give 20 mg (25%) of aldehyde VIII and 34 mg (43%) of nicotyrine methiodide with mp 211-213 deg C.

Synthesis of IIIa,b and VIIa,b. A) Equivalent amounts of nitromethane or nitroethane and a catalytic amount of piperidine were added to an alcohol solution of II or Vb, and the mixture was maintained at room temperature.

B) An alcohol solution of II, Vb, nitromethane, and a catalytic amount of n-butylamine was maintained at room temperature.

C) A mixture of II or Vb, nitromethane or nitroethane, and ammonium acetate was heated at 100 deg C. The yields, physical constants, and spectral characteristics of the products are presented in Table 1.

2,3-Dimethyl-5-(2-carboxyvinyl)indole (IIIc). A 173-mg (1 mmole) sample of aldehyde II and 124 mg (1.2 mmole) of malonic acid were dissolved in 3 ml of pyridine containing traces of piperidine, and the mixture was refluxed for 6 h. It was then poured over ice, and the aqueous mixture was acidified to pH 4-5 with hydrochloric acid. The resulting precipitate was removed by distillation, washed with water, and dried to give 195 mg (90%) of IIIc with mp 207-210 deg C (in a sealed capillary after vacuum distillation). IR spectrum: 1680 (C=O); 3340 (OH, NH); 1615 and 985 cm^{-1} (trans C=C). Found: C 72.6; H 6.1%. $C_{13}H_{13}NO_2$. Calculated: C 72.5; H 6.0%.

2,3-Dimethyl-6-(2-carboxyvinyl)-7-methoxyindole (VIIc). Similarly VIc was obtained in 98% yield from 100 ml of Vb. The product was purified preparatively in a thin layer of 5/40μ silica gel [benzene–methanol–ethyl acetate (9 : 1 : 2)] to give a product with mp 256-257 deg C. IR spectrum: 1675 (C=O); 1610 and 980 (trans C=C); 3390 cm^{-1} (N–H, OH). Found: C 68.6; H 6.2%. $C_{14}H_{15}NO_3$. Calculated: C 68.5; H 6.1%.

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