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REACTION OF 2- AND 4-VINYLPYRIDINES WITH PHENACYLPYRIDINIUM YLIDS

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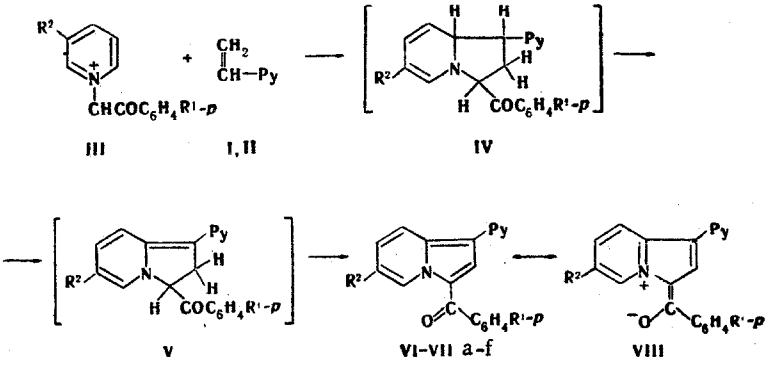
The reaction of 2- and 4-vinylpyridines with 3-substituted phenacylpyridinium ylids takes place regioselectively with the formation of only 6-substituted 1-pyridyl-3-aroylindolizines. The reaction of the same ylids with dimethyl acetylenedicarboxylate gives a mixture of isomeric 6- and 8-substituted 1,2-dicarbomethoxy-3-aroylindolizines. In the analogous reaction of 2-bromo-1-phenacylpyridinium ylid, in addition to the corresponding 5-bromoindolizine, the product of its spontaneous cyclization, viz., 4,5-dicarbomethoxy-6-oxo-(6H)-10c-azaacephenanthrylene, was isolated.

We have previously shown that the double bond in vinylpyridine is capable of undergoing 1,3-dipolar cycloaddition with dipolar compounds such as diazomethane; the cycloaddition proceeds regioselectively and is controlled primarily by electronic factors [1].

In order to study the regioselectivity of the cycloaddition of other dipolarophiles to vinylpyridines we studied the reaction of 2- and 4-vinylpyridines (I, II) with phenacylpyridinium ylids III.



III I, II IV V VI-VII a-f VIII

I, VIa-f Py=2-pyridyl; II, VIIa-f Py=4-pyridyl;
I—VII a $R^1=R^2=H$; b $R^1=NO_2$, $R^2=H$; c $R^1=H$, $R^2=COOC_2H_5$; d $R^1=NO_2$, $R^2=COOC_2H_5$; e $R^1=H$, $R^2=Br$; f $R^1=H$, $R^2=CH_3$

The fundamental possibility of the use of phenacylpyridinium ylids in 1,3-dipolar cycloaddition reactions was demonstrated in the case of their reaction with acrylonitrile [2]; Fröhlich and Kröhnke were able to isolate the primary products of cycloaddition, viz., the tetra- and dihydro derivatives (of the IV and V type), which are readily converted to 1-cyano-3-aroylindolizines. However, the effect of a substituent in the pyridine ring on

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TABLE 1. 3-Aroylindolizines VI and VII

Compound	mp, °C	UV spectrum ($CHCl_3$), λ max (log ε)	IR spectrum, ν, cm^{-1}	PMR spectrum (CF_3COOH)		Found, %			Empirical formula	Calc., %			Yield, % (synthetic method)
				2-H	5-H (J_{56}, Hz)	C	H	N		C	H	N	
VIa	156—157[b]	241 (4,25), 274 (4,23), 302 (4,16), 354 (4,08), 391 (4,18)	1600	7,90	9,93 d	80,5	4,5	—	$C_{20}H_{14}N_2O$	80,5	4,7	—	10 (A), 9 (B)
VIb	234—235[c]	258 (4,45), 274 (4,46), 243 (4,14), 412 (4,12)	1618	7,70	9,97 d	70,1	4,3	12,1	$C_{20}H_{13}N_3O_3$	70,0	3,8	12,2	18 (A), 19 (B)
VIc	149—149.5[d]	258 (4,55), 320 (4,38), 405 (4,25)	1725 1630	7,84	10.50s	74.7	4,8	7,9	$C_{23}H_{18}N_2O_3$	74,6	4,9	7,6	14(B)
VId	238—239[c]	255 (4,56), 310 (4,38), 414 (4,20)	1735 1640	—	10,98s	66,5	4,5	10,1	$C_{23}H_{17}N_3O_5$	66,5	4,1	10,1	31 (B)
VIe	174—175[d]	250 (4,49), 281 (4,39), 310 (4,36), 388 (4,22)	1605	7,80	10,10s	63,9	3,3	7,8	$C_{20}H_{13}BrNO_2$	63,7	3,5	7,4	6 (B)
VIIa	162—163[b]	236 (4,40), 268 (4,26), 309 (4,20), 353 (4,18), 384 (4,26)	1600	7,84	9,94 d	81,0	5,0	9,0	$C_{20}H_{14}N_2O$	80,5	4,7	9,4	10 (B)
VIIb	209—210[c]	255 (4,29), 275 (4,29), 298 (4,26), 340 (3,98), 400 (4,07)	1610	7,77	10,10 d (7)	70,2	4,2	12,2	$C_{20}H_{13}N_3O_3$	70,0	3,8	12,2	18 (B)
VIIc	160—161,5[b]	257 (4,56), 315 (4,34), 398 (4,25)	1720 1630	7,95	10,65s	74,5	4,9	—	$C_{23}H_{18}N_2O_3$	74,6	4,9	—	14 (B)
VIId	213—214[c]	260 (4,59), 305 (4,38), 407 (4,19)	1740 1640	—	11,02s	66,4	4,2	—	$C_{23}H_{17}N_3O_5$	66,5	4,1	—	29 (B)
VIIe	224—225[d]	250 (4,41), 278 (4,39), 302 (4,29), 383 (4,22)	1620	—	—	63,6	4,0	—	$C_{20}H_{13}BrNO_2$	63,7	3,5	—	5 (B)

[a]The most intense bands at 1600-1750 cm^{-1} are presented. [b]From methanol. [c]From ethyl acetate. [d]From ethanol.

the regioselectivity of the process was not studied in [2], whereas, according to the data in [3], substituents in the 3 or 4 position of the pyridine ring have an appreciable effect on the position of the signals of the carbon atoms of the CH_2 and CO groups of the phenacyl residue in the ^{13}C NMR spectra of substituted phenacylpyridinium ions. One therefore should have expected increased reactivities for those systems that contain electron-acceptor substituents in both the phenacylium residue and in the pyridine ring.

It was found that the reaction of ylids III with vinylpyridines I and II gives the products in relatively low yields, which, however, are increased when electron-acceptor substituents are present in the ylid structure (see Table 1, VIb-d and VIIb-d), whereas the yields decrease markedly when substituents that display +I or +M effects are present (VIe and VIIe; VIf and VIIf are detected only by chromatography). Let us note that the primary products of addition (IV and V) cannot be detected in the reaction mixture, and the only reaction products are indolizines VI and VII. The dehydrogenation of intermediates IV and V occurs at least partially due to the excess corresponding vinylpyridine that is present in the reaction mixture, since we identified ethylpyridine among the reaction products by gas–liquid chromatography.

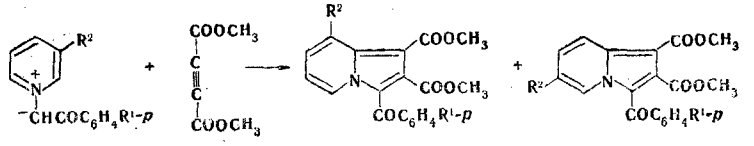
Only one compound containing a pyridine residue in the 1 position and substituent R^2 in the 6 position was isolated in all cases. However, indolizines VIa and VIIa were isolated from the reaction mixture along with VIe and, respectively, VIIIe in the reaction of 2- and 4-vinylpyridines with ylid IIIe. The formation of indolizines VIa and VIIa is probably associated with partial reduction of the ylid (or the reaction product) due to the hydrogen liberated during aromatization of structures IV and V.

Compounds VI and VII had similar UV spectra with several absorption maxima that are characteristic for the indolizine ring [4] (Table 1). A singlet of the 2-H proton at 7.7-7.9 ppm was observed in the PMR spectra of all of the compounds, and a singlet of a 5-H proton (VI and VIIc-e) was observed at 10.1-11.0 ppm. This anomalously weak-field signal of the 5-H proton is characteristic for 3-benzoylindolizines [2, 5].

A more rigorous confirmation of the presence of a pyridyl substituent in the 1 position was obtained on the basis of the results of x-ray diffraction analysis of VIe [6].

It is interesting to note that the IR spectra of VI and VII do not contain an absorption band characteristic for the carbonyl group; only an absorption band at 1600-1630 cm^{-1} (Table 1), which we assigned to vibrations of the conjugated $\rangle\overset{+}{N}—C=C—O^-$ system, is present. In fact, according to the results of x-ray diffraction analysis [6], the C–O bond (1.32 Å) in VIe is considerably longer than the C=O bond and coincides with the length of the C–O bond in the carbonate anion (1.30 Å) [7]. At the same time, the length of the bond between the carbon atom in the 3 position of indolizine and the carbonyl carbon atom (1.38 Å) is considerably shorter than the aliphatic C–C bond (1.54 Å) and approaches the length of the C=C bond (1.34 Å). These data make it possible to assume that the structures of VI and VII are closer to the structure of dipolar ion VIII.

It is known that the reaction of ethyl propiolate with 3-substituted nitrogen ylids leads to both 6- and 8-substituted indolizines with predominance of the latter in most cases [8]. The formation in our case of only one isomer that contains a substituent in the 6 position is evidently explained by steric hindrance created by the bulky pyridyl substituent in the 1 position. To verify this assumption we studied the reaction of ylids IIIc-e with dimethyl acetylenedicarboxylate (IX); both possible isomers X and XI, with predominance of indolizine X, were identified in the reaction mixture in all cases.

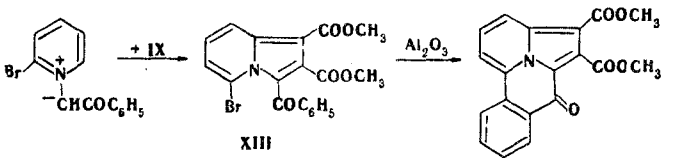


IIIc-e IX Xc-e XIc-e

As in the case of the reaction with vinylpyridine, 1,2-dicarbomethoxy-3-benzoylindolizine (XII) was found along with bromine-containing products in the reaction mixture in the reaction of ester IX with ylid IIIe.

The reaction of ester IX with 2-bromo-1-phenacylpyridinium ylid proceeded anomalously: thus XIV, which does not contain bromine but has a molecular mass that is two units lower

than that of indolizine XII, was detected in the reaction mixture along with 5-bromo-1,2-dicarbomethoxy-3-benzoylindolizine (XIII). An absorption band at 1635 cm^{-1}, which is characteristic for the stretching vibrations of the ketone carbonyl group of polycondensed heterocycles [9], was observed in its IR spectrum along with two characteristic frequencies of the vibrations of ester carbonyl groups (1740 and 1705 cm^{-1}). The UV spectrum of XIV had a fine structure and differed markedly from the spectra of indolizines X-XIII. In the PMR spectrum of this compound signals of 3-H and 1-H protons (each in the form of a doublet of doublets) with chemical shifts of, respectively, 8.60 and 9.17 ppm, and spin–spin coupling constants of 8 and 1.5 Hz can be isolated from the overall multiplet of seven aromatic protons; this confirmed the XIV structure:



Compound XIV is probably a secondary reaction product, since indolizine XIII is converted to it quantitatively on contact with aluminum oxide.

EXPERIMENTAL

The UV spectra of the compounds were recorded with a Cary-15 spectrophotometer. The IR spectra of suspensions of the compounds in mineral oil were recorded with an IKS-22 spectrometer. The PMR spectra of solutions of the compounds in trifluoroacetic acid were obtained with T-60 and XL-100 spectrometers with hexamethyldisiloxane as the external standard. The mass spectra of 3-aroylindolizines VI and VII were described in [10]. The degree of purity of all of the compounds obtained was monitored by thin-layer chromatography (TLC), on aluminum oxide [neutral and activities II-III (Brockmann classification)] or on Silufol UV-254 with development in UV light or with iodine vapors. The isolation of the substances by means of preparative TLC was carried out on plates (18×24 cm) with an aluminum oxide layer thickness of 1 mm or with a column filled with aluminum oxide (the column diameter was 30 mm, and the layer height was 25 cm) in systems selected for each specific case.

Phenacylpyridinium ylids IIIa-f were obtained by the general method in [11] or in situ from the corresponding phenacylpyridinium bromides described in [11-13]. However, 3-bromo-1-phenacylpyridinium bromide, which was obtained in 63% yield, had mp 211-212°C, which is close to the melting point presented in [13] but does not agree with the data in [12]; the results of elementary analysis were in agreement with the structure.

3-Carbethoxy-1-phenacylpyridinium Bromide. This compound was obtained in 71% yield and had mp 124-125°C. Found: C 54.7; H 4.7; Br 22.6%. $C_{16}H_{16}BrNO_3$. Calculated: C 54.9; H 4.6; Br 22.8%.

3-Carbethoxy-1-(p-nitrophenacyl)pyridinium Bromide. This compound was obtained in 46% yield and had mp 203-204°C (dec.). Found: C 48.5; H 3.6; Br 20.0%. $C_{16}H_{15}BrN_2O_5$. Calculated: C 48.6; H 3.8; Br 20.2%.

1-[2(4)-Pyridyl]-3-aroylindolizines (VIa-d and VIIa-d, Table 1). A) A mixture of 0.005 mole of phenacylpyridinium ylid IIIa,b and 1 g (0.01 mole) of vinylpyridine I or II in 50 ml of acetonitrile was maintained at room temperature for 2 days, after which the acetonitrile and excess pyridine bases were evaporated in vacuo, and the residue was chromatographed with a column filled with aluminum oxide; the yellow band was eluted with benzene or benzene–chloroform (1:1).

B) A 4-ml (0.04 mole) sample of vinylpyridine I or II was added to a suspension (0.02 mole) of phenacylpyridinium bromide in 60 ml of DMF, and a solution of 2.8 ml (0.02 mole) of triethylamine in 10 ml of DMF was added with stirring and cooling with ice water. The mixture was allowed to stand with cooling for 2 h then at room temperature overnight. It was then poured into 400 ml of water, and the precipitate was removed by filtration and washed with ether, and the aqueous solution was extracted with ether. The ether extract was dried with anhydrous sodium sulfate, the ether and pyridine bases were evaporated in vacuo, and the residue was chromatographed with a column filled with aluminum oxide as in method A. The constants and yields of compounds VIa-d and VIIa-d are presented in Table 1.

Reaction of Ylid IIIe with Vinylpyridine I. A solution of 2.8 ml (0.02 mole) of triethylamine in 10 ml of DMF was added with stirring and cooling (0°C) to a mixture of 7.14 g (0.02 mole) of 3-bromo-1-phenacylpyridinium bromide and 4 ml (0.04 mole) of 2-vinylpyridine in 60 ml of DMF, after which the mixture was allowed to stand at room temperature for 2 days. It was then poured into ice water, and the resinous precipitate was removed by filtration and washed with water. After drying in vacuo, the precipitate was chromatographed with a column filled with aluminum oxide using a system of solvents with a gradual increase in eluting ability [petroleum ether, benzene–petroleum ether (2:1), benzene]. Separation into two bands was observed during the chromatography. Evaporation of the solvent from the first fraction gave 480 mg (6.4%) of indolizine VIe (Table 1) with R_f 0.8 (Al_2O_3, chloroform). Workup of the second fraction gave 240 mg (4%) of a substance that gave a negative test for halogen and had a chromatographic mobility (R_f 0.5, Al_2O_3, chloroform) that was similar to that of the previously obtained indolizine VIa. No melting-point depression was observed for a mixture of samples of the two compounds, and their IR and mass spectra were identical.

Reaction of Ylid IIIe with Vinylpyridine II. This compound was obtained by a method similar to the method in the preceding experiment. The reaction of 7.14 g (0.02 mole) of 3-bromo-1-phenacylpyridinium bromide, 4 ml (0.04 mole) of 4-vinylpyridine, and 2.8 ml (0.02 mole) of triethylamine in 60 ml of DMF yielded, after chromatography with a column filled with aluminum oxide (as in the preceding experiment), 370 mg (4.9%) of indolizine VIIe (Table 1) and 130 mg (2.6%) of indolizine VIIa. No melting-point depressions were observed for mixtures with previously obtained samples, and their IR and mass spectra were identical.

Reaction of Ester IX with Ylid IIIc. A solution of 2 ml (0.14 mole) of triethylamine in 5 ml of DMF was added at −10°C to a mixture of 3.5 g (0.01 mole) of 3-carbethoxy-1-phenacylpyridinium bromide and 2.3 g (0.03 mole) of ester IX in 50 ml of DMF, and the mixture was allowed to stand with cooling for 3 h then at room temperature for 3 days. It was then poured into 300 ml of water, and the aqueous mixture was extracted with chloroform. The extract was dried with sodium sulfate, the chloroform was removed by evaporation, and the residue was chromatographed with a column filled with aluminum oxide with a gradual increase in the eluting ability of the system of solvents [petroleum ether, petroleum ether–benzene (1:1), petroleum ether–benzene (1:2), benzene]. Workup gave 0.5 g (12%) of XIc with mp 109-110°C (ethanol) [UV spectrum, λ_{max} (log ε): 248 (4.69), 293 (4.21), 358 (4.11), and 374 nm (4.18). IR spectrum: 1735, 1720, 1705, and 1638 cm^{-1} (C=O). PMR spectrum: 1.43 (3H, t, J = 8 Hz, CH_3), 3.48 (3H, s, CH_3), 3.92 (3H, s, CH_3), 4.45 (2H, q, J = 8 Hz, CH_2), 8.00 (1H, q, J = 9 Hz, 1 Hz), 8.33 (1H, d, J = 9 Hz), 10.22 (1H, d, J = 1 Hz). Found: C 64.4; H 4.8%. $C_{22}H_{19}NO_7$. Calculated: C 64.5; H 4.7%] and 1.4 g (35%) of indolizine Xc with mp 134-135°C [IR spectrum: 1720, 1635 cm^{-1} (C=O). UV spectrum (in chloroform), λ_{max} (log ε): 248 (4.52), 365 nm (4.18). PMR spectrum: 1.34 (3H, t, J = 8 Hz, CH_3), 3.32 (3H, s, CH_3), 3.88 (3H, s, CH_3), 4.43 (2H, q, J = 8 Hz, CH_2), 7.16 (1H, dd, J = 7 Hz, 8 Hz), 8.00 (1H, d, J = 8 Hz), and 9.67 (1H, d, J = 7 Hz). Found: C 64.4; H 4.9%. $C_{22}H_{18}NO_7$. Calculated C 64.5; H 4.7%].

Reaction of Ester IX with Ylid IIId. This reaction was carried out in the same way as in the preceding experiment. The reaction of 6.0 g (0.015 mole) of 3-carbethoxy-1-(4-nitrophenacyl)pyrinium bromide, 4.3 g (0.03 mole) of ester IX, and 4 ml (0.03 mole) of triethylamine in 100 ml of DMF gave, after chromatography with a column filled with aluminum oxide with successive elution with petroleum ether, benzene, benzene–chloroform (2:1), and benzene–chloroform (1:2), 0.9 g (13%) of indolizine XId with mp 210-211°C [IR spectrum: 1730, 1720, 1705, 1638 cm^{-1} (C=O). UV spectrum (in chloroform), λ_{max} (log ε): 252 (4.64) and 381 nm (4.17). PMR spectrum: 1.33 (3H, t, J = 8 Hz, CH_3), 3.33 (3H, s, CH_3), 3.88 (3H, s, CH_3), 4.35 (2H, q, J = 8 Hz, CH_2), 7.60 (2H, d, J = 8 Hz), 8.15 (2H, d, J = 8 Hz), 7.97 (1H, d, J = 8 Hz), 8.30 (1H, d, J = 8 Hz), and 10.30 (1H, s). Found: C 57.9; H 4.2; N 6.1%. $C_{22}H_{18}N_2O_7$. Calculated: C 58.2; H 4.2; N 6.2%] and 2.0 g (30%) of indolizine Xd with mp 189-190°C [IR spectrum: 1725, 1710, and 1618 cm^{-1}. UV spectrum (in chloroform), λ_{max} (log ε): 265 (4.54) and 373 nm (4.12). PMR spectrum: 1.26 (3H, t, J = 8 Hz, CH_3), 3.30 (3H, s, CH_3), 3.83 (3H, s, CH_3), 4.40 (2H, q, J = 8 Hz, CH_2), 7.20 (1H, dd, J = 7 and 8 Hz), 7.75 (2H, d, J = 9 Hz), 7.90 (1H, d, J = 8 Hz), 8.25 (2H, d, J = 9 Hz), and 9.75 (1H, d, J = 7 Hz). Found: C 57.9; H 4.2; N 6.5%. $C_{22}H_{18}N_2O_7$. Calculated: C 58.2; H 4.0; N 6.2%].

Reaction of Ester IX with 3-Bromo-1-phenacylpyridinium Bromide. This reaction was carried out in the usual way (see the method used to prepare indolizines VI and VII, method B). The reaction of 7.14 g (0.02 mole) of 3-bromo-1-phenacylpyridinium bromide, 5.8 g (0.04 mole) of ester IX, and 4 ml (0.03 mole) of triethylamine in 100 ml of DMF gave, after chromatography with a column filled with aluminum oxide (elution with benzene), 5 g of an oily substance. Crystallization from methanol gave 4.2 g of a yellow crystalline substance with mp 120-140°C and R_f 0.6 [Al_2O_3, elution with benzene–chloroform (1:4)]. Preparative separation of 200 mg of this mixture on plates in the case of three-stage chromatography [Al_2O_3, benzene–heptane (10:1)] yielded 50 mg of bromoindolizine XIe with mp 159-160°C [PMR spectrum: 3.24 (3H, s, CH_3), 3.80 (3H, s, CH_3), 7.0-7.6 (6H, m), 8.06 (1H, d, J = 9 Hz), and 9.63 (1H, s). Found: C 55.2; H 3.4%. $C_{19}H_{14}BrNO_5$. Calculated: C 54.8; H 3.4%], 100 mg of a mixture of XIe and, possibly, Xe, and 1,2-dicarbomethoxy-3-benzoylindolizine (XII), with mp 120-130°C, and 30 mg of indolizine XII, which, according to its IR spectrum and the results of a mixed-melting-point determination, was identical to a specially prepared sample [14].

Reaction of Ester IX with 2-Bromo-1-phenacylpyridinium Bromide. Similarly, the reaction of 7.14 g (0.02 mole) of 2-bromo-1-phenacylpyridinium bromide, 5.7 g (0.04 mole) of ester IX, and 3.5 ml (0.025 mole) of triethylamine in 60 ml of DMF gave, after chromatography with a column filled with aluminum oxide [elution with benzene–chloroform (2:1)] 750 mg of 4,5-dicarbomethoxy-6-oxo(6H)-10c-azaacephenanthrylene (XIV) with mp 265-266°C [IR spectrum: 1740, 1705, and 1630 cm^{-1} (C=O). UV spectrum (in chloroform), λ_{max} (log ε): 263 (4.73), 307 (3.78), 319 (3.78), 337 (3.71), 353 (3.87), 397 (4.28), and 418 nm (4.38)] and 1.7 g of a mixture of substances with R_f 0.5 and 0.2 (Silufol, elution with chloroform), which gives a positive Beilstein test for halogen. A 500-mg sample of the mixture was dissolved in 30 ml of chloroform, the solution was filtered through a layer (5 cm) of silica gel, and the filtrate was immediately evaporated to dryness in vacuo. The residue was crystallized from ethanol several times with monitoring of the degree of purity by TLC on Silufol, as a result of which 100 mg of chromatographically pure 1,2-dicarbomethoxy-3-benzoyl-5-bromoindolizine (XIII), with mp 162-163°C, was obtained. IR spectrum: 1745, 1710, and 1640 cm^{-1} (C=O). UV spectrum (in chloroform), λ_{max} (log ε): 240 (4.58), 295 (4.20) sh, 350-355 (3.99), and 412 nm (3.45). M^+ 415 (^{79}Br isotope).

Conversion of 5-Bromoindolizine XIII to XIV. A 1-g sample of aluminum oxide was added to a solution of 50 mg of indolizine XIII in 80 ml of chloroform, and the mixture was allowed to stand overnight at room temperature. The aluminum oxide was removed by filtration, and the chloroform was removed from the filtrate to give 40 mg (99%) of XIV.

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