may be attributed to predominant competitive reactions of the fluorine atoms alpha to the nitrogen. The great reactivity of such fluorine atoms has been previously reported.[10]

Acknowledgment. We are indebted to the Research Corporation for financial aid in support of this work.

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[10] (*a*) D. Barr and R. Haszeldine, *J. Chem. Soc.*, 2532 (1955); (*b*) R. L. Dannley and M. Lukin, *J. Org. Chem.*, 21,1036 (1956); (*c*) R. L. Dannley, R. G. Taborsky, and M. Lukin, *J. Org. Chem.*, 21, 1318 (1956).

[CONTRIBUTION NO. 998 FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF PITTSBURGH]

Pyridylethylation of Active Hydrogen Compounds. VI. Reactions of Ketones, Alkylpyridines, and Alkylquinolines with 2- and 4-Vinylpyridine[1,2,3]

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A study has been made of the conjugate addition reactions of a series of ketones with 4-vinylpyridine and of four alkylpyridines and two alkylquinolines with 2- and 4-vinylpyridine.

In 1947 Doering and Weil published a classical paper[4] in which they demonstrated elegantly that 2- and 4-vinylpyridine undergo conjugate addition reactions with a representative group of nucleophilic reagents. We have extended their observations and in previous papers from this laboratory the reactions of 2-vinylpyridine with ketones[5,6] and of both 2- and 4-vinylpyridine with ammonia,[2] amines,[2,7,8] amides,[2] and nitriles[2] were discussed.

The present paper is concerned with the reactions of 4-vinylpyridine with a series of ketones and of both 2- and 4-vinylpyridine with several alkylated tar bases. Apparently the only previously reported direct pyridylethylation of a ketone with 4-vinylpyridine appears in a paper by Levine and Wilt,[5] who obtained a 21.5% yield of phenyl γ-(4-pyridyl)-propyl ketone by the interaction of a 2:1:0.2 molar ratio of acetophenone, 4-vinylpyridine and sodium for a reaction time of 4 hr. This compound also has been prepared indirectly by the ketonic cleavage of ethyl α-[β-(4-pyridyl)ethyl]benzoylacetate,[9] which was obtained by the reaction of 4-vinylpyridine with ethyl benzoylacetate.

In the present study the reaction between acetophenone, 4-vinylpyridine and sodium has been reinvestigated. The interaction of a 2:1 molar ratio of ketone to 4-vinylpyridine for periods of 2, 4, and 6 hr. (Table I) gave mixtures of the monopyridylethylated product, phenyl γ-(4-pyridyl)-propyl ketone (16 to 18%) and the dipyridylethylated product, 3-benzoyl-1,5-di-(4-pyridyl)pentane, (65 to 73%). Even when a molar ratio of ketone to vinylpyridine of 4:1 was used in an attempt to greatly increase the amount of mono- and decrease the dipyridylethylated product, these derivatives were obtained in 21.6% and 36% yields, respectively. These results appear to indicate that 4-vinylpyridine reacts more readily with the initially formed monopyridylethylated compound to give the dipyridylethylated derivative than it does with acetophenone to give the monopyridylethylated compound.

The reaction of a 2:1 molar ratio of *p*-methylacetophenone to 4-vinylpyridine gave a low yield of the mono- (18.2%) and a higher yield of the dipyridylethylated (46.8%) product. Although under similar conditions the reaction of propiophenone with 4-vinylpyridine gave a high yield (88.5%) of the monopyridylethylated product, when this reaction was repeated using a 1:2 ratio of ketone to 4-vinylpyridine, essentially equal amounts of the mono- (43.4%) and the dipyridylethylated (41.0%) products were obtained.

Five symmetrical ketones have been condensed with 4-vinylpyridine (Table I). It may be seen that with the exception of acetone, each of these ketones was monopyridylethylated in fair to good yield. In the cyclohexanone reaction a mixture of a considerable amount of self-condensed ketone, 2-(1-cyclohexenyl)cyclohexanone, 40.8% of the mono-

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[1] This paper is based on part of a thesis presented by George Magnus to the graduate faculty of the University of Pittsburgh in partial fulfillment of the requirements of the Ph.D. degree.

[2] For the previous paper in this series, see G. Magnus and R. Levine, *J. Am. Chem. Soc.*, 78, 4127 (1956).

[3] This work was performed under Contract No. AT(301)-670 between the U. S. Atomic Energy Commission and the University of Pittsburgh.

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[5] R. Levine and M. H. Wilt, *J. Am. Chem. Soc.*, 74, 342 (1952).

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[7] H. E. Reich and R. Levine, *J. Am. Chem. Soc.*, 77, 4913 (1955).

[8] H. E. Reich and R. Levine, *J. Am. Chem. Soc.*, 77, 5434 (1955).

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TABLE I

REACTIONS OF 4-VINYLPYRIDINE WITH KETONES

Ketone	Yield, %	B.p., °C.	Mm.	Products Formula	Products % Nitrogen Calcd.	Products % Nitrogen Found	Monopicrates Formula	Monopicrates % Nitrogen Calcd.	Monopicrates % Nitrogen Found	M.p., °C.
Acetophenone[a,b]	18.4	78–79 (m.p.)[c]								
	66.0[d]	232–235	1	$C_{22}H_{22}NO$	8.48	8.16	$C_{34}H_{28}N_8O_{15}$[e,f]	14.21	13.88	172.8–173.8
p-Methylacetophenone[a]	18.2	72–73 (m.p.)		$C_{16}H_{17}NO$	5.86	6.06	$C_{22}H_{20}N_4O_8$	11.96	12.12	127–128
	46.8[d]	247–250	1	$C_{23}H_{24}N_2O$	[g]		$C_{35}H_{30}N_8O_{15}$[e]	[h]		173.5–174.5
Propiophenone	88.5[a]	198–200	4	$C_{16}H_{17}NO$	5.86	5.98	$C_{22}H_{20}N_4O_8$	11.96	11.84	137–138
	43.4[i]	198–200	4							
	41.0[d,i]	245–247	1.5	$C_{23}H_{24}N_2O$	8.13	8.16	$C_{35}H_{30}N_8O_{15}$[e]	13.96	13.55	191–192
Cyclohexanone[a]	40.8	157–160	1.5	$C_{13}H_{17}NO$	6.89	7.33	$C_{19}H_{20}N_4O_8$	12.95	12.67	129.5–131
	36.4[d,j]	245–255	2	$C_{20}H_{24}N_2O$	[k]		$C_{32}H_{30}N_8O_{15}$[e]	[l]		199–200
Acetone[a]	5.0	109–111	2	$C_{10}H_{13}NO$	8.59	8.93	$C_{16}H_{16}N_4O_8$	14.29	14.17	113–114
Diethyl[a]	45.0	125–128	1.5	$C_{12}H_{17}NO$	7.32	7.57	$C_{18}H_{20}N_4O_8$	13.33	13.27	102–103
Diisopropyl[a]	64.0[m]	125–127	1	$C_{14}H_{21}NO$	6.39	6.93	$C_{20}H_{24}N_4O_8$	12.50	12.81	110.2–111.2
Diisobutyl[a]	48.6	147–150	1.5	$C_{16}H_{25}NO$	5.66	5.48	$C_{22}H_{28}N_4O_8$	11.76	11.65	113.5–114.5
Methyl ethyl[a]	26.0	136–138	4	$C_{11}H_{15}NO$	7.91	8.18	$C_{17}H_{18}N_4O_8$	13.78	13.76	109–110
Methyl isopropyl[a]	64.0	121–124	1.5	$C_{12}H_{17}NO$	7.32	7.67	$C_{18}H_{20}N_4O_8$	13.33	13.05	125.5–126.5
Methyl benzyl	7.5[a]	194–195	5.0	$C_{16}H_{17}NO$	[n]		$C_{22}H_{20}N_4O_8$	11.96	11.54	133.5–134.5
	79.5[o]	194–195	5.0							

[a] Molar ratio of ketone:4-vinylpyridine:sodium was 2:1:0.2 and reaction time was 2 hr. [b] Reaction time of 4 hr. gave 17.6% of mono- and 73.0% of dipyridylethylated product and a 6-hr. reaction time gave 16.4% mono and 65.3% di. Reaction of a 1:0.25:0.05 molar ratio of ketone:4-vinylpyridine:sodium gave 21.6% mono and 36% di. [c] See refs. 5 and 9; b.p. 190–195° at 1.5 mm. [d] This is a dipyridylethylated product. All the products not so marked are monopyridylethylated ketones. [e] This is a dipicrate. [f] *Anal.* Calcd.: C, 51.77; H, 3.57. Found: C, 51.96; H, 3.43. [g] *Anal.* Calcd.: C, 80.23; H, 6.97. Found: C, 80.31; H, 6.63. [h] *Anal.* Calcd.: C, 52.36; H, 3.74. Found: C, 52.54; H, 3.50. [i] The molar ratio of ketone:4-vinylpyridine:sodium was 1:2:0.2 and the reaction time was 2 hr. [j] There was also obtained 23.5 g. of 2-(1-cyclohexenyl)cyclohexanone (b.p. 136–138° at 10 mm.; semicarbazone, m.p. 175–177° [J. Reese, *Ber.*, **75B**, 384 (1942)]. [k] *Anal.* Calcd.: C, 77.92; H, 7.79. Found: C, 77.48; H, 7.70. [l] *Anal.* Calcd.: C, 50.13; H, 3.91. Found: C, 49.86; H, 3.77. [m] There was obtained 3.5 g. (6.6%) of 1,4-di-(4-pyridyl)butane, m.p. 117.5–118.5° (see refs. 2 and 17). [n] *Anal.* Calcd.: C, 80.33; H, 7.11. Found: C, 79.90; H, 6.99. [o] A mixture of 5 ml. of Triton B and 0.5 mole each of ketone and 4-vinylpyridine was kept at 70–75° for 2 hr. and then processed.

pyridylethylated product, and 36.4% of the dipyridylethylated product were isolated. Although the structure of the last compound was not elucidated, it is suggested that it is 2,2-bis-[2-(4-pyridyl)-ethyl]cyclohexanone by analogy with the reaction of cyclohexanone with acrylonitrile, which apparently gives 2,2-bis-(β-cyanoethyl)cyclohexanone.[10] As a by-product in the reaction of diisopropyl ketone with 4-vinylpyridine, there was obtained a small amount (6.6%) of the bimolecular reduction product of 4-vinylpyridine, 1,4-di(4-pyridyl)butane. This compound was previously obtained in the reaction between acetamide and 4-vinylpyridine.[2]

Although acetone has been pyridylethylated by 4-vinylpyridine to give only extremely low yields (4 to 5%) of 5-(4-pyridyl)-2-pentanone, this ketone was prepared in an over-all yield of 56% by condensing 4-vinylpyridine with ethyl acetoacetate to give ethyl α-[β-(4-pyridyl)ethyl]acetoacetate (67%), which was then cleaved to the desired ketone in 84% yield by refluxing with concentrated hydrochloric acid.

While the structures of the first eight products listed in Table I are unambiguous, it is entirely possible that the reactions of 4-vinylpyridine with the unsymmetrical ketones, methyl ethyl, methyl isopropyl and methyl benzyl ketones, could give rise to isomeric condensation products by pyridylethylation at either or both of the α-carbon atoms of these ketones. It was definitely established that the product derived from methyl ethyl ketone and 4-vinylpyridine is 3-methyl-5-(4-pyridyl)-2-pentanone, II, *i.e.*, condensation had occurred at the methylene carbon atom of the ketone. This was done by showing that the product was identical with an authentic sample of II, which was prepared by treating ethyl α-methylacetoacetate with 4-vinylpyridine to give ethyl α-methyl,α-[β-(4-pyridyl)-ethyl]acetoacetate, III, which was then subjected to ketonic cleavage.

$$CH_3COCH_2CH_3 + \underset{\text{I}}{4\text{-}C_5H_4NCH{=}CH_2} \xrightarrow{Na} \underset{\text{II}}{CH_3COCH(CH_3)CH_2CH_2C_5H_4N\text{-}(4)}$$

$$CH_3COCH(CH_3)CO_2C_2H_5 + \text{I} \xrightarrow{Na} \underset{\text{III}}{CH_3COC(CH_3)(CO_2C_2H_5)CH_2CH_2C_5H_4N\text{-}(4)}$$

$$\text{III} \xrightarrow{HCl/H_2O} \text{II}$$

It was also shown that the compound derived from methyl isopropyl ketone and 4-vinylpyridine is 3,3-dimethyl-5-(4-pyridyl)-2-pentanone (*i.e.*, reaction had occurred at the methinyl carbon atom of the ketone) by subjecting the product to the haloform reaction and obtaining 2,2-dimethyl-4-(4-

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(10) H. A. Bruson and T. W. Riener, *J. Am. Chem. Soc.*, 64, 2850 (1942).

pyridyl)butanoic acid. An authentic sample of this acid was prepared by saponifying ethyl 2,2-dimethyl-4-(4-pyridyl)butanoate, which was synthesized in 28.5% yield by condensing 4-vinylpyridine with ethyl isobutyrate. There is also little doubt that the compound derived from methyl benzyl ketone is 3-phenyl-5-(4-pyridyl)-2-pentanone since it also undergoes a haloform reaction.

It is well known that the hydrogen atoms of the methyl groups in 2- and 4-picoline are quite acidic. The reactivity of these compounds has often been compared with that of methyl ketones and therefore it is not surprising that they may be effectively alkylated[11,12] and acylated[13-15] at their side chains. It was therefore of interest to determine whether these and related compounds could be condensed with 2- and 4-vinylpyridine. The results obtained are listed in Table II. Prior to our study, Leonard and Boyer[16] had condensed 2-vinylpyridine with 2-picoline using sodium as the condensing agent and obtained a 33% yield of 1,3-di-(2-pyridyl)propane. In the present study this reaction was repeated and a 37% yield of the same product was obtained. Jampolsky *et al.*[17] have studied the reaction between 4-vinylpyridine and 4-picoline and claim that while sodium was an ineffective catalyst for this condensation, the use of potassium gave a 41% yield of 1,3-di(4-pyridyl)-propane. Contrary to these findings we have been able to use sodium successfully and have obtained a 41% yield of the expected product. We have also prepared the mixed dipyridylpropane, 1-(2-pyridyl)-3-(4-pyridyl)propane by the reaction of 2-picoline with 4-vinylpyridine and 4-picoline with 2-vinylpyridine. It is interesting to note that the former route gave a 28% yield and the latter route a 10% yield of product. It has also been possible to pyridylethylate 2- and 4-ethylpyridine with both 2- and 4-vinylpyridine in fair to good yields. However, both 4-*n*-propyl- and 4-isopropylpyridine failed to condense with 4-vinylpyridine. In the quinoline series, although quinaldine and lepidine have been pyridylethylated with 2-vinylpyridine in low yield, both of these tar bases failed to condense with 4-vinylpyridine.

TABLE II

REACTIONS OF 2- AND 4-VINYLPYRIDINE WITH TAR BASES

Tar Base	Vinylpyridine	Yield, %	Products					Derivative			M.P., °C.
			B.P.			% Nitrogen			% Nitrogen		
			°C.	Mm.	Formula	Calcd.	Found	Formula	Calcd.	Found	
2-Picoline[a]	2	37.4	130–135	1.5							208–209[b,c]
	4	28.2	128–130	1.0	$C_{13}H_{14}N_2$	14.14	14.51	$C_{25}H_{20}N_8O_{16}$[d]	16.27	16.57	211–212
4-Picoline	2	10.1	140–142	2.0		14.14	14.45	$C_{25}H_{20}N_8O_{14}$[b]	17.04	16.76	178.5–179.5
	4	40.6	158–162	2.0		14.14	13.77	[b]	17.04	17.20	185–186
			62–65 (m.p.)[e]								
2-Ethylpyridine	2	62.5	134–136	2.0	$C_{14}H_{16}N_2$	13.21	12.84	$C_{26}H_{22}N_8O_{16}$[d]	15.95	15.92	175.5–176.5
	4	34.4	160–162	6.0		13.21	12.99	[d]	15.95	16.25	188–189
4-Ethylpyridine	2	37.4	140–142	1.5		13.21	13.56	[d]	15.95	16.20	185.5–186.0
	4	50.8	160–161	2.0		13.21	12.96	[d]	15.95	15.81	192–192.5
Quinaldine	2	21.0	182–184	1.0	$C_{17}H_{16}N_2$	11.29	11.45	$C_{29}H_{22}N_8O_{14}$[b]	15.87	16.28	210–211
	4	0.0									
Lepidine	2	17.0	185–187	1.0	$C_{17}H_{16}N_2$	11.29	11.35	$C_{29}H_{22}N_8O_{14}$[b]	15.87	16.13	214–215
	4	0.0									

[a] In all reactions a 2:1:0.1 molar ratio of tar base:vinylpyridine:sodium and a 2-hr. reaction time were employed. [b] This derivative is a dipicrate. [c] See ref. 16. [d] This derivative is a distyphnate. [e] A melting point of 57–60° is reported in ref. 17.

EXPERIMENTAL[18]

General procedure for reactions of ketones with 4-vinylpyridine using sodium as the catalyst and acetophenone as an example. Acetophenone (120.0 g., 1.0 mole), 4-vinylpyridine (52.5 g., 0.50 mole), and 2.3 g. (0.10 mole) of sodium were placed in the previously described reactor.[5] After stirring the mixture for a few minutes, a sufficiently vigorous exothermic reaction occurred so that the mixture refluxed. After about 15 min., the exothermic reaction began to subside. Heat was then applied and the mixture was refluxed for an additional 2 hr. After cooling to room temperature the contents of the flask was poured onto a mixture of ice and 100 ml. of concentrated hydrochloric acid, and the aqueous solution was extracted with three 100-ml. portions of chloroform. The combined chloroform extracts (Extract I) were dried over anhydrous sodium sulfate. The aqueous phase was made strongly basic by the addition of saturated

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[11] For leading references, see "The Chemistry of the Alkali Amides. III," R. Levine and W. C. Fernelius, *Chem. Revs.*, **54**, 540 (1954).

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[13] N. N. Goldberg, L. B. Barkley, and R. Levine, *J. Am. Chem. Soc.*, **73**, 4301 (1951).

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[15] N. N. Goldberg and R. Levine, *J. Am. Chem. Soc.*, **77**, 3647 (1955).

[16] N. J. Leonard and J. H. Boyer, *J. Am. Chem. Soc.*, **72**, 4818 (1950).

[17] L. M. Jampolsky, M. Baum, S. Kaiser, L. H. Sternbach, and M. W. Goldberg, *J. Am. Chem. Soc.*, **74**, 5222 (1952).

[18] The 2- and 4-vinylpyridine, 2- and 4-picoline, quinaldine and lepidine were kindly supplied by Dr. F. E. Cislak, Reilly Tar and Chemical Corp.

aqueous sodium carbonate solution, extracted with chloroform and dried over anhydrous sodium sulfate (Extract II). The solvents from Extracts I and II were removed by atmospheric distillation and the residues were distilled in vacuum. From Extract I there was obtained 20.0 g. of recovered acetophenone, b.p. 114–116° at 52 mm. and 20.0 g. neutral material, b.p. 190–225° at 0.5 mm. (probably self-condensation products of acetophenone). Extract II yielded 20.5 g. (18.4%) of phenyl γ-(4-pyridyl)propyl ketone b.p. 190–195° at 1.5 mm., m.p. 78–79°; 55.6 g. (66.0%) of 3-benzoyl-1,5-di-(4-pyridyl)pentane (b.p. 232–235° at 1 mm.) and 12.0 of a tarry, nitrogenous residue.

When Triton B was employed as the catalyst, the exothermic reaction mixtures were not allowed to reflux but were maintained at 70–75° by using an ice water bath when necessary.

Preparation of an authentic sample of 5-(4-pyridyl)-2-pentanone. A mixture of 52.0 g. (0.4 mole) of ethyl acetoacetate, 21.0 g. (0.2 mole) of 4-vinylpyridine and 0.6 g. (0.026 mole) of sodium was refluxed for 6 hr. and processed to give 31.5 g. (67.0%) of ethyl α-[β-(-4-pyridyl)-ethyl]-acetoacetate, b.p. 162–164° at 2.0 mm. This product, 90 ml. of concentrated hydrochloric acid, and 90 ml. of water were refluxed for 4 hr. and processed to give 18.3 g. (84%) of 5-(4-pyridyl)-2-pentanone, b.p. 102–105° at 1.0 mm. This compound formed a yellow picrate, m.p. 113–114° alone and when mixed with a sample prepared from the material obtained by the direct reaction of acetone with 4-vinylpyridine.

Preparation of authentic sample of 3-methyl-5-(4-pyridyl)-2-pentanone. A mixture of 72.0 g. (0.5 mole) of ethyl α-methylacetoacetate, 63.2 g. (0.6 mole) of 4-vinylpyridine and 2.0 g. (0.085 mole) of sodium was refluxed for 24 hr. and processed to give 37.0 g. (29.7%) of ethyl α-methyl-α-[β-(4-pyridyl)ethyl]acetoacetate, b.p. 154–157° at 1.5 mm. This product, 100 ml. of concentrated hydrochloric acid, and 100 ml. of water were refluxed for 16 hr. and processed to give 19.0 g. (61.7%) of 3-methyl-5-(4-pyridyl)-2-pentanone, b.p. 120–122° at 2 mm. This compound formed a yellow picrate, m.p. 109–110° alone and when mixed with a sample prepared from the material obtained by the direct reaction of methyl ethyl ketone with 4-vinylpyridine.

Proof of structure of adduct obtained from methyl isopropyl ketone and 4-vinylpyridine. (*a*) *Oxidation with potassium hypochlorite.* The adduct from methyl isopropyl ketone and 4-vinylpyridine (21.0 g., 0.11 mole) was oxidized with potassium hypochlorite using the procedure described previously[6] for the oxidation of 3,3-dimethyl-5-(2-pyridyl)-2-pentanone to give 2.5 g. (11.8%) of 2,2-dimethyl-4-(4-pyridyl)butanoic acid, m.p. 149.5–150.5° (from 95% ethanol).

Anal. Calcd. for $C_{11}H_{15}NO_2$: N, 7.25. Found: N, 7.48.

(*b*) *Preparation of an authentic sample of 2,2-dimethyl-4-(4-pyridyl)butanoic acid.* A mixture of 57.5 g. (0.50 mole) of ethyl isobutyrate, 52.6 g. (0.50 mole) of 4-vinylpyridine and 1.5 g. (0.065 mole) of sodium was refluxed for 3 hr. and processed to give 31.5 g. (28.5%) of ethyl 2,2-dimethyl-4-(4-pyridyl)butanoate, b.p. 118–120° at 1.5 mm. This ester formed a yellow picrate, m.p. 85.5–86.5° (from 95% ethanol).

Anal. Calcd. for $C_{19}H_{22}N_4O_9$: N, 12.44. Found: N, 12.61.

A mixture of 10.0 g. (0.05 mole) of the ester, 10.0 g. of sodium hydroxide and 100 ml. of water was refluxed until a homogeneous solution was obtained. The mixture was then cooled and acidified with glacial acetic acid to give 7.5 g. (77.5%) of 2,2-dimethyl-4-(4-pyridyl)butanoic acid, m.p. 149.5–150.5° alone and when mixed with a sample of the acid obtained in part (*a*).

General procedure for reactions of tar bases with 2- and 4-vinylpyridine using reaction between 2-ethylpyridine and 4-vinylpyridine as an example. A mixture of 2-ethylpyridine (52.5 g., 0.50 mole), 4-vinylpyridine (26.3 g., 0.25 mole) and 1.15 g. (0.05 mole) of sodium was placed in the reactor. Since no apparent reaction occurred, the mixture was heated cautiously to 96°. At this temperature a vigorous exothermic reaction took place and it was necessary to use an ice bath to control the reaction. After the exothermic reaction had subsided, the mixture was refluxed for 2 hr. and allowed to cool to room temperature. Absolute ethanol (5 ml.) was cautiously added and then the contents of the flask were poured onto a mixture of ice and water. The mixture was extracted with chloroform and processed in the regular manner to give 18.0 g. (34.4%) of 1-(4-pyridyl)-3-(2-pyridyl)butane, b.p. 160–162° at 6.0 mm. In addition, 34.0 g. of a mixture of recovered 2-ethylpyridine and 4-vinylpyridine, b.p. 60–62° at 35 mm., and 26.0 g. of a tarry nitrogenous residue were obtained.

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