duces a detectable amount of a volatile material giving the xanthate test for carbon disulfide. Mere oxygen concentration does not seem to govern the amount produced, for the test with air was very faint. The results also strongly suggest that ethylene alone is without effect.

The poisoning effect of carbon disulfide on nickel catalysts is well known and may account for the erratic results obtained using Fieser's solution.

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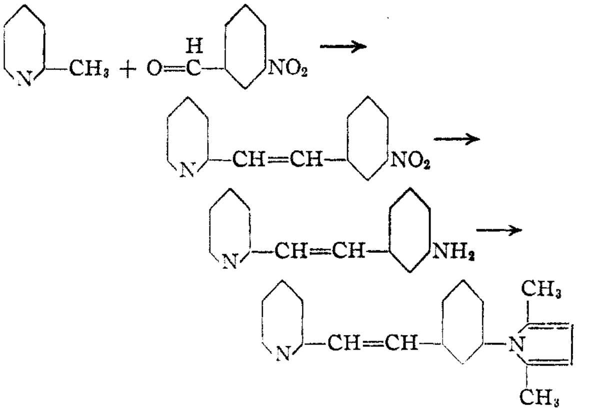
Some Styrylpyridines and -Quinolines

BY H. GILMAN AND GEORGE KARMAS

Incidental to a study of anils, several substituted styrylpyridines and -quinolines have been prepared. Some of the nitrostyryl types were reduced to the corresponding aminostyryls, and these in turn were condensed with acetonylacetone to give the corresponding 2,5-dimethylpyrrylstyryl compounds. The following is an illustrative sequence of transformations:



Experimental

The condensations of aldehydes with the picolines, quinaldine, and lepidine were effected by conventional procedures.[1,2] In a few comparative preparations it was observed that the method using acetic anhydride was better than the one in which anhydrous zinc chloride was used. The general results are given in Table I. Tests on the following compounds will also be reported later: α-(*p*-dimethylaminostyryl)-quinoline[3]; α-(*m*-aminostyryl)-quinoline[4]; α,α′-(di-*m*-nitrostyryl)-pyridine[5]; α- and γ-(*m*-nitrostyryl)-pyridine[2,5]; α-(*m*-nitrostyryl)-quinoline[6]; α-(*p*-nitrostyryl)-quinoline[7]; α-(*m*-2,5-dimethylpyrrylstyryl)-quinoline; α- and γ-(*p*-nitrostryl)-pyridine[2,5]; and α- and γ-(*m*-aminostyryl)-pyridines.[8]

The authors are grateful to Parke, Davis and Company for arranging the tests.

TABLE I

STYRYL DERIVATIVES

Product	M. p. (cor.), °C.	Anal., % N Calcd.	Anal., % N Found
α,γ-(C_5H_3N)($CH{=}CHC_6H_4NO_2$-*m*)$_2$[a]	242–243	10.80	11.02
γ-(C_5H_4N)$CH{=}CHC_6H_4N(CH_3)_2$-*p*[b]	240–241	12.50	12.37
γ-(C_9H_6N)$CH{=}CHC_6H_4N(CH_3)_2$-*p*[c]	141–142	10.23	10.36
γ-(C_5H_4N)$CH{=}CHC_4H_3O$-α (picrate)[d]	212–214	14.00	14.25
α-(C_9H_6N)$CH{=}CHC_6H_4CF_3$-*m*[e]	104–105	4.68	4.18
α-(C_5H_4N)$CH{=}CHC_6H_4NH_2$-*m*[f]	103–104	14.30	14.32
γ-(C_5H_4N)$CH{=}CHC_6H_4NH_2$-*m*[g]	189–190	14.30	14.45
α-(C_5H_4N)$CH{=}CHC_6H_4NC_6H_8$-*m*[h]	87–88	10.20	10.40
α-(C_9H_6N)$CH{=}CHC_6H_4NC_6H_8$-*m*[i]	132–133	8.63	8.70

[a] This condensation product of α,γ-lutidine and *m*-nitrobenzaldehyde was crystallized from tetrachloroethane. The yield was 34%. [b] Crystallization of the condensation product from *p*-dimethylaminobenzaldehyde was from methanol. [c] Crystallization was from petroleum ether (b. p. 80–110°). [d] The condensation product with furfural distilled at about 200° (18 mm.), and because of its instability in air was converted to the picrate which was crystallized from ethanol. [e] This compound was synthesized in 65% yield by condensation with *m*-trifluoromethylbenzaldehyde (prepared by F. Yeoman), and was crystallized from dilute ethanol. [f] The nitrostyrenes were reduced to the corresponding amines by refluxing an ethanol solution with a saturated aqueous solution of sodium sulfide. The hemi-hydrate of this *m*-aminostyryl derivative of α-picoline was reported to melt at 85° [Schuftan, *Ber.*, **23**, 2717 (1890)]. Our product, obtained in 49% yield, was crystallized from dilute ethanol. [g] No m. p. was given for this compound. See Deutsche Hydrierwerke Akt.-Ges., French Patent 775,101 (June 27, 1934) [*Chem. Zentr.*, **106**, I, 3853 (1935)]. Our product, obtained in 47% yield, was crystallized from ethanol. [h] The *m*-2,5-dimethylpyrrylstyryl compound, obtained by condensation of the *m*-aminostyryl product with acetonylacetone, distilled as a viscous light yellow oil at 180–190° (0.001 mm.). This solidified after a short time, and crystallization from dilute methanol gave small, colorless flakes. [i] The reduction of α-(*m*-nitrostyryl)-quinoline by sodium sulfide gave the corresponding amine in a 57% yield and melting at 160–161°. The m. p. reported by Wartanian, *Ber.*, **23**, 3648 (1890), is 158–159°. In addition to the amine we isolated a small quantity of an orange colored compound which was insoluble in hot ethanol, but crystallized from dioxane as fine, orange needles melting at 226–227°. This may be the azo compound, $C_9H_6NCH{=}CHC_6H_4N{=}NC_6H_4CH{=}CHC_9H_6N$. *Anal.* Calcd. for $C_{34}H_{24}N_4$: N, 11.46. Found: N, 11.21. The *m*-(3,5-dimethylpyrrylstyryl) product, obtained in 72% yield by condensation of the amine with acetonylacetone, distilled at 200–210° (0.001 mm.). Crystallization from ethanol gave fine, buff platelets.

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[1] Kaplan and Lindwall, THIS JOURNAL, **65**, 927 (1943).

[2] Shaw and Wagstaff, *J. Chem. Soc.*, 77 (1933).

[3] Noelting and Witte, *Ber.*, **39**, 2750 (1906). Our m. p. for this compound was 181–182°, and they reported 177°.

[4] Wartanian, *Ber.*, **23**, 3648 (1890). Our m. p. for this compound was 160–161°, and he reported 158–159°.

[5] Wagstaff, *J. Chem. Soc.*, 277 (1934).

[6] Taylor and Woodhouse, *ibid.*, 2971 (1926).

[7] Bulach, *Ber.*, **20**, 2047 (1887).

[8] See footnotes *f* and *g* in Table I.

Fluorescence Chromatography of the Methyl Glucoses[1]

BY ETHELDA J. NORBERG, IRVING AUERBACH[2] AND R. M. HIXON

Several investigators have attempted, by chromatographic techniques, the quantitative separation of the ultimate hydrolysis products of methylated starch: *i. e.*, 2,3-dimethyl glucose,

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