from benzene or from a benzene–petroleum ether solution yielded platelets whose "melting" point depended on the rate of heating as described above. *Anal.* Calcd. for 3-hydroxymethylindole: C, 73.4; H, 6.16. Found: C, 73.2; H, 6.03. The infrared spectrum showed the N–H bond stretching peak at 2.97 mμ, and a shoulder at about 3.05 mμ ascribed to hydrogen-bonded hydroxyl.

The reduction was also carried out in an aqueous dioxane solution, and the same product was obtained.

Sodium Borohydride Reduction of 2-Pyrrolealdehyde.—A solution of 2.0 g. of sodium borohydride in 10 ml. of water was added over a period of ten minutes to a stirred solution of 1.8 g. of 2-pyrrolealdehyde in 30 ml. of water. The temperature rose to 42°. After one hour, the solution was saturated with potassium carbonate and extracted with ether. The ether solution was dried and evaporated, and the residue was distilled at 81–83° at 2 mm. The distillate was a colorless, moderately viscous oil; yield 1.3 g. (71%) *Anal.* Calcd. for 2-hydroxymethylpyrrole: C, 61.8; H, 7.27. Found: C, 61.9; H, 7.14; n^{20}_{D} 1.5425; d^{20}_{4} 1.130; $(R)_D$ calcd. 27.3, found 27.1; mol. wt. calcd. 97, found 104, 106 by cryoscopy in water. It was noted that the aqueous solution became slightly turbid during the measurements. By analogy with the reported behavior of 3-hydroxymethylindole,[3] probably self-condensation was occurring. The infrared spectrum showed the N–H and bonded O–H bands as described above.

Lithium Aluminum Hydride Reduction of 2-Pyrrolealdehyde.—A slurry of 0.8 g. of lithium aluminum hydride in ether was added dropwise to a stirred solution of 2.0 g. of 2-pyrrolealdehyde in 50 ml. of dry ether. The mixture was stirred for 15 minutes after the addition was completed, and the excess hydride and the organic salt were decomposed with a slight excess of water. The ether solution was filtered, dried and evaporated. Distillation of the residue gave 1.2 g. (59%) of 2-hydroxymethylpyrrole.

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DEPARTMENT OF CHEMISTRY
STANFORD RESEARCH INSTITUTE
STANFORD, CALIFORNIA

The Rosenmund Reduction of Trichloroacetyl Chloride

BY J. W. SELLERS AND W. E. BISSINGER

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It has been claimed that trichloroacetyl chloride is a product of the oxidation of tetrachloroethylene in the presence of light, chlorine or peroxides.[1] The Rosenmund reduction of trichloroacetyl chloride would be of interest as a method of preparing chloral. When the acyl halide was reduced under modified Rosenmund conditions dichloroacetyl chloride was produced in 50–60% yields. No chloral was found. If the reduction were carried out under ordinary Rosenmund conditions[2] (in xylene at 110° using 2% palladium–barium sulfate catalyst, and a quinoline-S regulator) wherein 0.25 millimole of hydrogen chloride was evolved per minute, the acid evolution abruptly stopped when 2 moles of hydrogen chloride had appeared per mole of acid chloride (6 hr.). The product did not contain any material having a boiling point below 125°. When the reaction was stopped after the evolution of 1 mole of hydrogen chloride per mole of acid chloride, the product was a complex mixture of high-boiling substances. Attempts to obtain the reduction in refluxing *n*-heptane produced no acid.

Experimental[3]

Rosenmund Reduction of Trichloroacetyl Chloride.—A suspension of 40 g. (0.22 mole) of trichloroacetyl chloride,[4] 4 g. of 2% palladium–barium sulfate catalyst, 0.4 ml. of quinoline-S regulator and 190 ml. of Insectisol (a highly refined petroleum fraction which was refluxed over sodium, distilled, and the fraction, b.p. 190–220°, collected) was heated to 140°. Hydrogen was passed through the stirred mixture at such a rate that a millimole of hydrogen chloride per minute was collected in the aqueous exit gas scrubber. After 0.22 mole of HCl had been titrated (5 hr.) the mixture was cooled, filtered, and distilled. The fraction, 15.5 g. (50%), b.p. 104–106° (729 mm.),[5] was collected.

Anal. Calcd. for C_2HOCl_3: mol. wt., 147.4; Cl, 72.17. Found: mol. wt. (titration of hydrolysis products in 0.1 *N* NaOH), 146; Cl, 71.0.

The preparation of the dichloroacetanilide in benzene at room temperature[6] gave colored products difficult to purify. When prepared in acetone[7] at −5 to −10° using a 10% excess of aniline (mole ratio of acid chloride: aniline, 1:2.2) and recrystallized from 95% ethanol, the anilide, m.p. 117.5–118.5°, was obtained in 72% yield calculated on the acid chloride.

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[1] F. Kirkbride, U. S. Patent 2,321,823, June 15, 1943; E. Hart and M. Matheson, U. S. Patent 2,472,946, June 14, 1949.

[2] E. Hershberg and J. Cason, *Org. Syntheses*, **21**, 84 (1941).

(3) All melting points uncorrected.

(4) H. Brown, THIS JOURNAL, **60**, 1325 (1938).

(5) E. Gustus and P. Stevens, *ibid.*, **55**, 374 (1933).

(6) E. Votocek and J. Burda, *Ber.*, **48**, 1002 (1915).

(7) P. McKie, *J. Chem. Soc.*, **123**, 2213 (1923).

RESEARCH LABORATORIES
COLUMBIA-SOUTHERN CHEMICAL CORPORATION
BARBERTON, OHIO

The Preparation of Chalcones from Hydroxy and Methoxy Aldehydes and Ketones[1]

BY HERBERT E. SMITH AND MARK C. PAULSON

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Four new methoxylated chalcones,[2] of possible interest as model substances for the investigation of the structure of lignin, have been prepared from hydroxy and methoxy aldehydes and ketones. The crude products were either semisolids or oils which were purified by repeated recrystallization. The recovery of significant amounts of starting materials from each preparation was not unexpected, since condensations of this nature are reversible and the yields of pure products were relatively low. The physical properties, derivatives and analyses of the chalcones are given in Table I.

Rapid cooling of the alcoholic solution of 2′-hydroxy-3,3′,4-trimethoxychalcone produced an intimate mixture of two crystalline varieties which proved to be polymorphic forms which were separable by fractional recrystallization. Controlled cooling of the clarified solution to 50–55° during recrystallization of this chalcone produced only the crystalline prismatic variety. The isolation of minute quantities of 3′,4′,8-trimethoxyflavanone and veratric acid from the reaction mixture indicated that acid isomerization of the 2′-hydroxychalcone[3] and oxidation of the aldehyde

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(1) This note is based on a portion of a thesis submitted by Herbert E. Smith in partial fulfillment of the requirements for the degree of Master of Science at Bradley University.

(2) The number system used for the substituted chalcones is that of *Chemical Abstracts*.

(3) St. von Kostanecki and W. Szabranski, *Ber.*, **37**, 2634 (1904).