5-10% of retained water the films were heated at 160° for one hour immediately before spectral analysis. Determinations showed that during the time it took to measure the spectra no appreciable absorption of moisture occurred.

Instrument.—The spectral measurements[11] were made on a Perkin-Elmer infrared spectrometer Model 12-A using a 40-cycle chopper, a Strong nickel-strip bolometer, an a. c. amplifier and a Brown Instrument Company potentiometer. The data were obtained on a point-to-point basis, the points being taken from 5 to 10 cm.$^{-1}$ apart at frequencies up to 1900 cm.$^{-1}$ and at larger intervals at higher frequencies.

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[11] We are indebted to Mr. Dwight Merrill for some of the spectral measurements.

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A Convenient Synthesis of Octahydropyrrocoline and 2-(γ-Hydroxypropyl)-piperidine

BY V. BOEKELHEIDE AND S. ROTHCHILD[1]

It has been found that octahydropyrrocoline, which has previously been prepared in poor yield by procedures involving three or more steps,[2] can be obtained readily in excellent yield by the direct hydrogenation of 2-(γ-hydroxypropyl)-pyridine[3] at 200° and 2500 lb. pressure with Raney nickel catalyst.

When the reduction was carried out at 125° instead of 200°, the product was not octahydropyrrocoline but rather 2-(γ-hydroxypropyl)-piperidine. The yield of 2-(γ-hydroxypropyl)-piperidine is practically quantitative and this is undoubtedly the best method for its preparation.[4] At temperatures intermediate between 125 and 200° the reduction gave mixtures of octahydropyrrocoline and 2-(γ-hydroxypropyl)-piperidine. Although it is probable that 2-(γ-hydroxypropyl)-piperidine is an intermediate in the formation of octahydropyrrocoline, 2-(γ-hydroxypropyl)-piperidine is not readily affected by heat and can be distilled repeatedly without change.

Experimental[5]

Octahydropyrrocoline.—A mixture of freshly distilled 2-(γ-hydroxypropyl)-pyridine (9.0 g., 0.066 mole), Raney nickel (2 g.), and absolute alcohol (10 cc.) was heated at 200° with shaking under an initial pressure of 2540 p. s. i. of hydrogen. At the end of one hour the drop in hydrogen pressure corresponded to that calculated. After removal of the catalyst by filtration, the oily product was distilled yielding 6.4 g. (78%) of a colorless oil; b. p. 71-72° at 32 mm.; $n^{21.2}$D 1.4702.

For purposes of identification, there were prepared the following derivatives: picrate, m. p. 228-9° (lit.,[2] m. p. 226°, 231-232°); gold chloride double salt, m. p. 188-192° (lit.,[2] m. p. 191-193°).

The methiodide of octahydropyrrocoline was formed in benzene and proved to be unstable in the presence of air. The amorphous solid, obtained from benzene, decomposed at 280-283° and was analyzed directly without further purification.

Anal. Calcd. for $C_9H_{18}NI$: C, 40.46; H, 6.79. Found: C, 40.00; H, 7.10.

2-(γ-Hydroxypropyl)-piperidine.—A mixture of freshly distilled 2-(γ-hydroxypropyl)-pyridine (14.0 g., 0.102 mole), Raney nickel (2 g.), and absolute alcohol (4 cc.) was heated at 125° with shaking under an initial pressure of 2500 p. s. i. of hydrogen. At the end of four hours the pressure drop corresponded to that calculated. After removal of the catalyst and solvent, the product was distilled yielding 12.5 g. (85%) of a colorless oil; b. p. 101-102° at 3 mm.; $n^{21.5}$D 1.4882.

For identification there was prepared the hydrochloride, m.p. 128-129° (lit.,[2b] m. p. 127-128°), and the mercuric chloride complex, m. p. 180-182° (lit.,[2a] m. p. 182-183°).

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[1] Aided by a Grant from the National Foundation for Infantile Paralysis.

[2] (a) Loeffler and Kaim, *Ber.*, **42**, 94 (1909); Loeffler and Flugel *ibid.*, **42**, 3423 (1909), and Tullock and McElvain, THIS JOURNAL, **61**, 961 (1939); (b) Clemo and Ramage, *J. Chem. Soc.*, 2969 (1932); (c) Wibaut and Beets, *Rec. trav. chim.*, **60**, 905 (1940); (d) Diels and Alder, *Ann.*, **498**, 16 (1932).

[3] 2-(γ-Hydroxypropyl)-pyridine is available from Reilly Tar and Chemical Co., Indianapolis, Indiana.

[4] Previous methods for preparing 2-(γ-hydroxypropyl)-piperidine are given in ref. (2a).

[5] Analysis by Mrs. G. L. Sauvage. All melting points are corrected.

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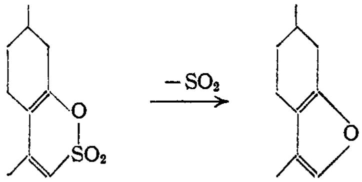
The Sulfonation of Mesityl Oxide

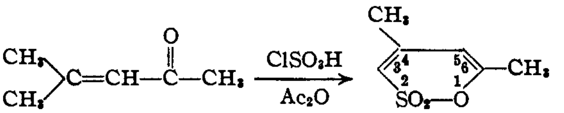
BY RICHARD H. EASTMAN AND DON GALLUP[1]

In seeking a convenient method for the synthesis of alkylated furans we have sought to extend to simple α,β-unsaturated ketones and aldehydes the observation of Treibs[2] that pulegone on treatment in acetic anhydride at ice temperature with concentrated sulfuric acid yields cyclopulegenol-sulfonic ester (I) from which menthofuran (II) is obtained in excellent yield on pyrolysis with zinc oxide at atmospheric pressure.



I II

We have attempted the sulfonation under varied conditions of concentration and temperature, using both sulfuric acid and chlorosulfonic acid, of crotonaldehyde, cyclohexylidenecyclohexanone, 3,4-dimethylhexene-3-on-2 and mesityl oxide. A clean-cut reaction was obtained only in the case of the sulfonation of mesityl oxide which on sulfonation with chlorosulfonic acid in acetic anhydride gave the cyclic sulfonic ester III in good yield. Pyrolysis of the cyclic sulfonic ester of mesityl oxide (III) at atmospheric pressure,



III

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[1] The work reported here is taken from a thesis presented by Don Gallup to Stanford University in partial fulfillment of the requirements for the degree of Master of Science.

[2] W. Treibs, *Ber.*, **70B**, 85 (1937).