

Of course, the reaction here described constitutes a limiting case in which the treatment with sulfuric acid of 2,2,4-trimethylpentane was allowed to proceed until no further action was noted. Less prolonged treatment would not lead to as great a destruction of the product.

Experimental

A sample of Röhm and Haas isooctane certified by the Bureau of Standards was fractionated through a column equivalent to 85 theoretical plates. All of the distilled material and the residue had n_D^{20} 1.3915–16. It was thus very pure 2,2,4-trimethylpentane.

In a 22-liter flask equipped with a sealed stirrer and reflux condenser attached to a trap cooled by dry-ice and acetone was placed 7836 g. of 2,2,4-trimethylpentane and 5 liters of 95% sulfuric acid (c. p. Baker "Analyzed"). A slow evolution of sulfur dioxide started after a few minutes and was very pronounced after three hours. The mixture was stirred for ten days at about 20°, after which time the evolution of sulfur dioxide had practically ceased.

The cold trap contained 610 g. of material about half of which was sulfur dioxide. The acid layer from the flask was removed and diluted with five times its volume of water and 1 kg. of black powder, presumably carbon, filtered off. The oil layer was washed, dried over potassium carbonate, and fractionated through a 15-plate column to give 39% of material boiling below 96° at 740 mm. and 31% boiling above 123°. Less than 25% of the starting material was recovered as fairly pure 2,2,4-trimethylpentane. The nature of the other products could not be determined even after painstaking fractionations through the 85-plate column. Indications were obtained, however, that neohexane and 2,2,4,4-tetramethylpentane were present.

SCHOOL OF CHEMISTRY AND PHYSICS
PENNSYLVANIA STATE COLLEGE
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NEW COMPOUNDS

3-Phenylphenyl Acetate

The method which was used for the preparation of this compound was the same as that reported previously for 4-phenylphenyl acetate,¹ except that after pouring the reaction mixture into water the ester was extracted with ether; the ethereal solution was dried with anhydrous sodium sulfate and filtered, and the ether was removed by distillation. The crude product was obtained as a light brown viscous oil in a 96.5% yield. After distillation at 135–136° (2 mm.), it solidified, m. p. 34.0–34.2° (cor.). (The melting point was determined from a freezing point curve; the thermometer which was used had been calibrated by the United States Bureau of Standards.)

(1) Hazlet and Kornberg, *THIS JOURNAL*, **61**, 3037 (1939).

Anal. Calcd. for $C_{14}H_{12}O_2$: C, 79.24; H, 5.66. Found: C, 79.6; H, 5.76.

The 3-phenylphenol which was used in this preparation was generously furnished by the Dow Chemical Company, Midland, Michigan.

DEPARTMENT OF CHEMISTRY
STATE COLLEGE OF WASHINGTON STEWART E. HAZLET
PULLMAN, WASHINGTON HARRY A. KORNBERG

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1-Phenylcycloheptanol

To an ethereal solution of the Grignard reagent prepared from 31.4 g. of bromobenzene and 5 g. of magnesium, after fifteen minutes, 22.4 g. of cycloheptanone was added with cooling and the alcohol isolated as usual, distilled in a vacuum, and the water droplets thereby formed removed over anhydrous sodium sulfate. The product was a viscous, colorless, fragrant liquid: n_D^{20} 1.5515, D_D^{20} 1.049, M_D (calcd.) 57.97, M_D (obsd.) 57.83.

Anal. Calcd. for $C_{13}H_{18}O$: C, 83.0; H, 9.0. Found: C, 82.79; H, 9.30.

DEPARTMENT OF CHEMISTRY
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RICHARD D. KLEENE

RECEIVED JANUARY 24, 1941

Derivatives of Tetrahydrofurfuryl Alcohol

3-Nitrohydrogenphthalate.—This compound was prepared according to the directions of Nicolet and Sacks.¹ It was obtained as a crystalline white solid which was recrystallized from hot water, m. p. 175–177°.

Anal. Calcd. for $C_{13}H_{13}O_7N$: N, 4.75. Found: N, 4.88.

α -Naphthylurethan.—This compound was prepared according to the directions of Shriner and Fuson.² It was obtained as clusters of needles which were recrystallized from petroleum ether, m. p. 88–90°.

Anal. Calcd. for $C_{16}H_{17}O_3N$: N, 5.17. Found: N, 5.27.

The tetrahydrofurfuryl alcohol was furnished by the Quaker Oats Company, Chicago, and the analyses were performed by Dr. T. S. Ma.

(1) Nicolet and Sacks, *THIS JOURNAL*, **47**, 2348 (1925).

(2) Shriner and Fuson, "Identification of Organic Compounds," John Wiley & Sons, Inc., New York, N. Y., 1935, p. 143.

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m-Bromo-*n*-alkylbenzenes

***m*-Bromo-*n*-propylbenzene.**—The Grignard reagent prepared from 61 g. of ethyl bromide was treated with 92.5 g. of *m*-bromobenzaldehyde and the crude *m*-bromophenylethylcarbinol thus obtained was heated for five hours at 160° with 30 g. of freshly fused potassium bisulfate in a