

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
STATE COLLEGE, PENNSYLVANIA

RECEIVED MARCH 31, 1941

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. KORNBURG

RECEIVED MARCH 14, 1941

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
UNIVERSITY OF CHICAGO
CHICAGO, ILLINOIS

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 $C_{12}H_{13}O_7N$: 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.

CHEMISTRY DEPARTMENT
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RICHARD D. KLEENE
SHERMAN FRIED

RECEIVED FEBRUARY 17, 1941

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

Claisen flask. At the end of this time the mixture was distilled under reduced pressure. Water came over first and then 31.4 g. (71%) of *m*-bromopropenylbenzene boiling at 108–114° at 16 mm. was obtained. This was dissolved in 100 cc. of ethanol and reduced in a Burgess-Parr reducing outfit with hydrogen and 0.1 g. of platinum oxide catalyst.¹ The reduction was complete in two and one-half hours. The mixture was filtered and distilled. There was thus obtained 27 g. (85%) of *m*-bromo-*n*-propylbenzene boiling at 96–100° at 17 mm.; n_D^{20} 1.5354.

Anal. Calcd. for $C_9H_{11}Br$: Br, 40.15. Found: Br, 40.19.

***m*-Bromo-*n*-butylbenzene.**—Using the same general procedure 74 g. of *n*-propyl bromide and 92.5 g. of *m*-bromobenzaldehyde gave 71 g. of *m*-bromobutenylbenzene boiling at 126–130° at 22 mm.; which on reduction yielded 59 g. of *m*-bromo-*n*-butylbenzene, b. p. 113–116° at 18 mm.; n_D^{20} 1.5330.

Anal. Calcd. for $C_{10}H_{13}Br$: Br, 37.51. Found: Br, 37.50.

(1) Adams, Vorhees and Shriner, "Organic Syntheses," Coll. Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1929, p. 452.

NOVES CHEMICAL LABORATORY
URBANA, ILLINOIS

C. S. MARVEL
DONALD G. BOTTERON

RECEIVED MARCH 19, 1941

n-Heptylsulfonylacetic Acid

The reaction of sodium chloroacetate with the sodium mercaptide from 25 g. (0.17 mole) of *n*-heptyl mercaptan was carried out in the same way as described¹ for the reaction of butyl mercaptan. The α -(*n*-heptylthio)-acetic acid was liberated by the addition of a solution prepared by dissolving 40 ml. of concentrated sulfuric acid in 180 ml. of water. The oil was separated and the aqueous layer extracted with ether. The combined water-insoluble layers were dried over anhydrous sodium sulfate and the ether removed by distillation. The residue was dissolved in 90 ml. of a mixture of glacial acetic acid-acetic anhydride (1:1) and oxidized with 43 g. (0.38 mole) of 30% hydrogen peroxide under the conditions previously¹ described. After removal of the solvent under reduced pressure the residue was crystallized from ether. The yield was 35 g. (83%) m. p. 90–91°. Recrystallization from water gave large glistening laminae of *n*-heptylsulfonylacetic acid, m. p. 95.5–96° (cor.).

Anal. Calcd. for $C_8H_{18}O_4S$: S, 14.42. Found: S, 14.54, 14.59.

(1) Pomerantz and Connor, THIS JOURNAL, 61, 3144 (1939).

DEPARTMENT OF CHEMISTRY AND CHEMICAL ENGINEERING
UNIVERSITY OF PENNSYLVANIA
PHILADELPHIA, PENNA.

G. GORDON URQUHART
RALPH CONNOR

RECEIVED MARCH 11, 1941

COMMUNICATIONS TO THE EDITOR

THE CARBOHYDRATE IN COLLAGEN

Sir:

It has been reported by Grassmann and Schleich [*Biochem. Z.*, 277, 320 (1935)] that the carbohydrate complex of collagen contains glucose and galactose in equimolecular proportions. In connection with a determination of the amount of carbohydrate in samples of collagen that were being investigated, an attempt was made to destroy the sugar in hydrolyzed collagen by fermentation with yeast. It was found that the sugar was not fermentable with a galactose-active yeast. When *d*-galactose and yeast were added to samples of the hydrolyzate, the added sugar was destroyed and the sugar from the collagen remained. These experiments give conclusive proof that neither *d*-glucose nor *d*-galactose forms a considerable part of the carbohydrate in collagen. The presumption is that the sugar is a mixture of *l*-glucose and *l*-galactose, but it has not been established that this is the case. It is significant in this regard that Bell and Baldwin [*Nature*, 146, 559 (1940)] re-

cently found *l*-galactose as a component of a polysaccharide of animal origin.

NATIONAL BUREAU OF STANDARDS
WASHINGTON, D. C.

JOHN BEEK, JR.

RECEIVED APRIL 8, 1941

THE ISOLATION OF PURE LINOLEIC ACID BY CRYSTALLIZATION

Sir:

The only method previously available for the preparation of linoleic acid which is pure as evaluated from its iodine number is that of debromination of pure tetrabromostearic acid. However, Matthews, Brode and Brown¹ have shown that the debromination acid may contain 12% of an isomeric linoleic acid. When this contaminating acid is removed by repeated low temperature crystallization, the resultant product melts nearly 2° higher than any specimen of linoleic acid so far reported.

(1) Matthews, Brode and Brown, THIS JOURNAL, 63, 1064 (1941).