

tracted with acetone. The residue obtained by evaporation of the acetone was crystallized twice from ethyl acetate to yield 0.1 g. of VII, melting at 289° (corr.), with decomposition. A mixed melting point with a sample of this acid obtained from the dibenzal compound showed no depression.

The end-point in a direct titration of V with 0.1 normal alkali is not permanent. The neutral equivalent found was 436.0 and that calculated was 442.8. The titration was also performed by adding a large excess of 0.1 normal alkali, allowing the solution to stand overnight and then back-titrating with 0.1 normal acid. In this manner one mole of V neutralized four moles of alkali.

Summary

3-Acetyl-2,4,6-trimethylbenzoic acid (II) has

been prepared by ozonization of benzaldiaceto-mesitylene (I).

Hypohalites convert the acid into the corresponding trihaloacetyl compounds (III and V). These are sufficiently stable toward alkali to be titrated in cold solutions. Warm solutions of alkali cleave them to the corresponding dicarboxylic acid (VII).

The tribromo acid is partially hydrolyzed by alkali to give the corresponding dibromo compound (VI) and hypobromite. The latter acts upon the bromoform present to give carbon tetrabromide.

URBANA, ILLINOIS

RECEIVED APRIL 13, 1934

NOTE

The Isomerization of *n*-Butyl Phenyl Ether

BY RICHARD A. SMITH

Straight chain alkyl phenyl ethers have not as yet been rearranged to substituted phenols with the aid of rearranging agents. Previous attempts in this direction have been unsuccessful. There is one reference to the action of very high temperatures on phenetole to yield ethylphenol.¹

The author has described a satisfactory method of rearranging alkylphenyl ethers with anhydrous aluminum chloride.² By utilizing this method, it has been found possible to convert *n*-butyl phenyl ether to *o*- and *p*-butyl phenols. Interestingly enough, the yield of phenols is comparable to that in cases of branched chain isomerizations.

A more complete study of this phase of alkyl phenyl ether rearrangement is being conducted.

Experimental

n-Butyl phenyl ether was prepared by the conventional method of the action of *n*-butyl bromide on the sodium salt of phenol in absolute alcohol: b. p. 206°, n_D^{26} 1.5019, d^{26} 0.9547.

(1) Schering-Kahlbaum A. G., Austrian Patent 125,216.

(2) R. A. Smith, *THIS JOURNAL*, **55**, 849, 3718 (1933).

The agreement with data reported by other workers is satisfactory.³

o- and *p*-Butylphenols.—One molar equivalent of anhydrous aluminum chloride was added in small portions to the phenyl ether. The heat generated was absorbed with an external cooling bath. The reaction mixture was allowed to stand for thirty-six hours at room temperature. The resultant products were hydrolyzed with ice and hydrochloric acid. The organic layer was separated and water washed. It was 95% soluble in 10% sodium hydroxide, from which it was precipitated with hydrochloric acid. The product was then water washed, dried with anhydrous sodium sulfate and separated into the two butyl isomers by distillation. There was a small quantity (approximately 5–10%) of higher boiling material—probably polyalkylated butylphenols: *o*-butylphenol, b. p. 238°, n_D^{22} 1.5205, d^{22} 0.973; *p*-butylphenol, b. p. 248°, n_D^{22} 1.5165, d^{22} 0.976.

The agreement with data reported by other workers is satisfactory.⁴

THE CHEMICAL LABORATORY RECEIVED APRIL 17, 1934
WASHINGTON SQUARE COLLEGE
OF NEW YORK UNIVERSITY
NEW YORK, N. Y.

(3) Sekera and Marvel, *ibid.*, **55**, 375 (1933).

(4) Sandulesco and Girard, *Bull. soc. chim.*, [4] **47**, 1300 (1932).