

ally dissolved, while crystals of the elongated form retained their sharp characteristics and increased in size. After twelve hours, only elongated prisms could be detected. The experiment demonstrated the elongated prisms to be the form least soluble in methanol-water mixtures.

We wish to acknowledge our indebtedness to Dr. Raymond M. Hann, of the National Institute of Health, for a supply of *d*-galactose, and to Mr. Robert L. Brown, of Dr. Wolfrom's laboratory, Ohio State University, for an authentic specimen of *d*-galactose diethylmercaptal pentaacetate.

FOOD AND DRUG ADMINISTRATION  
FEDERAL SECURITY AGENCY  
WASHINGTON, D. C.

RECEIVED AUGUST 13, 1941

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## NEW COMPOUNDS

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### Preparation of Tri-*m*-nitrophenyl Orthoformate

In an attempt to prepare *p*-nitrosalicylaldehyde, a modified Reimer-Tiemann reaction was used as outlined by D. E. Armstrong and D. H. Richardson [*J. Chem. Soc.*, 496 (1933)]. Instead of the desired compound, tri-*m*-nitrophenyl orthoformate was obtained. Fourteen grams of *m*-nitrophenol was dissolved in a solution of 56 g. of potassium hydroxide in 70 cc. of water. The solution was heated to 90° and trichloroacetic acid was added slowly. After violent boiling ceased, the mixture was diluted to three times its original volume and filtered. The light colored precipitate was washed with methyl alcohol and dried. Two and one-half grams of this substance was obtained. The solution was acidified and extracted with ether, which recovered 7.5 g. of *m*-nitrophenol. The acid solution was filtered after the extraction and 3 g. of tar was obtained.

The original precipitate (2.5 g.) from the alkaline solution, which melted at 182–183°, was recrystallized from butyl alcohol and small brownish colored crystals were obtained which melted at 182–183°.

When this compound was treated with warm sulfuric acid, it dissolved and a gas was evolved which was found to be carbon monoxide. Upon dilution of the sulfuric acid solution and filtering, the resulting precipitate was found to be *m*-nitrophenol.

A weighed sample of the compound was treated with

concentrated sulfuric acid in an atmosphere of carbon dioxide and the gas collected over 40% potassium hydroxide. This gave an equivalent weight of 480 g. per mole of carbon monoxide. The molecular weight of  $(\text{NO}_2\text{C}_6\text{H}_4\text{O})_3\text{CH}$  is 427. The high value can be accounted for by incomplete decomposition of the sample.

*Analysis* of the compound gave the following results: calcd. for tri-*m*-nitrophenyl orthoformate: C, 53.4; H, 3.04; N, 9.85. Found: C, 53.45; H, 2.95; N, 10.44.

The yield in this procedure was 17.5%.

DEPARTMENT OF CHEMISTRY  
UNIVERSITY OF CALIFORNIA  
BERKELEY, CALIFORNIA

M. CALVIN  
J. R. SEGESSER

RECEIVED NOVEMBER 3, 1941

### $\gamma$ -Phenoxypropyl *p*-Anisyl Ketone

The ether was removed from the Grignard reagent prepared from 62 g. of *p*-bromoanisole in 150 cc. of anhydrous ether, and the residue was cooled in an ice-bath. To this was added slowly, with swirling and cooling, a solution of 39 g. of  $\gamma$ -phenoxybutyronitrile<sup>1</sup> in 150 cc. of dry benzene. A considerable amount of heat was evolved. After the mixture had been refluxed for twelve hours, it was cooled and hydrolyzed with ice-cold ammonium chloride solution. The two layers were separated quickly and the benzene solution was shaken with cold dilute hydrochloric acid (1:5). The oily imine hydrochloride which separated was removed with the water layer, and the acid extraction was repeated twice, as rapidly as possible. The combined acid mixture was then warmed for two hours on a steam-bath and the resulting ketone extracted with benzene and ether. The solution was washed with sodium bisulfite solution and water and distilled. The fraction boiling at 200–210° (0.5 mm.) was collected and recrystallized from methanol; yield, 26 g. of colorless leaflets; m. p. 58–60°. After another recrystallization the compound melted at 59–60.5°. The original benzene layer, after the acid extraction, was distilled, and from the fraction of b. p. 195–210° (0.5 mm.) there was obtained an additional 11.5 g. of slightly less pure ketone; m. p. 56–60° to a cloudy liquid.

*Anal.* Calcd. for  $\text{C}_{17}\text{H}_{19}\text{O}_3$ : C, 75.5; H, 6.7. Found: C, 75.4; H, 6.9.

DEPARTMENT OF CHEMISTRY  
UNIVERSITY OF MICHIGAN  
ANN ARBOR, MICHIGAN

W. E. BACHMANN  
A. L. WILDS

RECEIVED OCTOBER 25, 1941

(1) Marvel and Tannenbaum, *This Journal*, **44**, 2647 (1922).