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## Azoyl Derivatives of Sugars and Separation by Chromatographic Adsorption (Abstract)

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### Recommended Citation

Coleman, George H. and Farnham, Alford G. (1941) "Azoyl Derivatives of Sugars and Separation by Chromatographic Adsorption (Abstract)," *Proceedings of the Iowa Academy of Science*, 48(1), 246-247. Available at: <https://scholarworks.uni.edu/pias/vol48/iss1/47>

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Nothing, however, was known about this effect in the indanones substituted in the 7 position. In these ketones free rotation about the bond between the carbonyl group and the ring is prevented and the increased opportunity for resonance might influence the steric effect.

The ketone 4, 7-dimethyl indanone-1, in which one ortho position is occupied by a methyl group and the other by the end of aliphatic ring, was prepared. It readily formed an oxime (m.p. 175.5 - 177.5° C), a semicarbazone (decomposed 215 - 221° C) and a phenyl-hydrazone (m.p. 99- 101° C).

MOUNT VERNON,  
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### PYRROLIDINE FROM N-ALKYL-N-CHLOROUREAS

(ABSTRACT)

GEORGE H. COLEMAN AND GLEN ALLIGER

Previous work in this laboratory has shown that when N-n-butyl-N-chloro-p-toluenesulfonamide, N-n-butyl-N-chloroacetamide, or N-n-butyl-N-chloropropionamide is dissolved in sulfuric acid and heated, ring closure occurs with the formation of pyrrolidine in good yields. It was thought that N, N'-di-n-butyl-N, N'-dichlorourea might react in a similar manner. This was found to be the case. When this compound was dissolved in 95 per cent sulfuric acid and heated for a period of one hour at 120-130° C. pyrrolidine was formed in yields of 50 to 55 per cent. Under similar conditions N, N-dimethyl-N'-n-butyl-N'-chlorourea gave pyrrolidine in yields of 5 to 10 per cent.

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### AZOYL DERIVATIVES OF SUGARS AND SEPARATION BY CHROMATOGRAPHIC ADSORPTION

(ABSTRACT)

GEORGE H. COLEMAN AND ALFORD G. FARNHAM

Azobenzene-p-benzoyl derivatives of d-d-glucose and B-d-glucose, fructose, galactose, lactose, maltose, sucrose, trehalose, gentiobiose and cellobiose have been prepared. They have been analyzed for per cent of azoyl and their specific rotations in chloroform determined. The chromatographic separation of d-d-glucose and fructose azoates as described by Reich has been repeated using silicic acid as an adsorbent. Using a mixture of "magnesol"

and "dicalite" as an adsorbent the following separations have also been made: lactose and galactose, sucrose and d-d-glucose, trehalose and B-d-glucose.

IOWA CITY, IOWA.

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## EFFECT OF HYDROGEN ION CONCENTRATION UPON THE HYDROLYSIS RATES OF KETIMINES

(ABSTRACT)

I. JOHNSON AND J. B. CULBERTSON

Measurements have been made upon the hydrolysis rates of ketimine hydrochlorides to obtain information concerning the relation between their varied structures and stability toward hydrolysis.<sup>1</sup> The hydrogen ion concentration might appear as a catalytic factor in this hydrolysis.<sup>2</sup> It would thus seem necessary to determine these hydrolysis rates at the same hydrogen ion concentration if this factor has a marked effect. Different ketimine hydrochlorides alone in water produce, in the dilute solutions usually employed, pH values ranging initially from about 3.6 to 5.3 and increasing in each case toward the pH of the equivalent ammonium chloride formed as the result of hydrolysis. Certain measurements on the hydrolysis rates of ketimine hydrochlorides of widely different initial pH values in water, to which an equivalent of hydrochloric acid was added, showed that their speed was actually lowered by this hydrogen ion increase although not enough to affect the general order of the rate.

In the studies for this report the hydrolysis rates for two ketimine hydrochlorides, representing extremes in initial hydrogen ion concentration of their hydrolyzing solutions, have each been measured in buffered solutions at several pH values ranging from 2 to 5. The results reveal a decrease in rate of hydrolysis with increase of hydrogen ion concentration, although the effect is not sufficient to markedly affect the general comparative order of the velocity constants of various ketimine salts.

Without making any attempt to analyze more critically the role of hydrogen ion concentration upon the hydrolysis rate of ketimine salts at this time, we may conclude that hydrolysis velocity measurements made upon the simple unbuffered ketimine salts