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## Preparation and Stability of Diphenyl Amine Hydronitrate

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compose over a period of weeks or less even when under refrigeration and protected from light.

Trialkylgold compounds at a temperature of about a  $-65^{\circ}$  may be cleaved with hydrogen chloride in anhydrous ether to give the corresponding hydrocarbons and dialkylgold halides. This fact has been utilized in an improved preparation of dialkylgold halides from gold tribromide and the desired alkyllithium compounds.

AMES, IOWA

## PREPARATION AND STABILITY OF DIPHENYL AMINE HYDRONITRATE

ROBERT E. JONES AND J. B. CULBERTSON

In connection with certain proposed studies the question of the preparation of diphenyl amine hydronitrate, or diphenyl ammonium nitrate, arose. A search of the literature revealed no record of this substance. The nearest equivalent reported was the perchlorate salt of diphenyl amine (Ber. 43, 1085-86. 1910). This compound was obtained upon treatment of a carbon tetrachloride solution of the amine with 70% perchloric acid added dropwise.

Aside from the fact that salts of diphenyl amine are very largely hydrolyzed in any aqueous media, at the outset it was recognized that the formation of a nitrate salt would be further complicated not only because of the strongly oxidizing character of the nitrate radical but also because of the nitrating property of concentrated nitric acid.

After a number of preliminary experiments involving various dilutions, a concentration of 0.25 M. pure (practically 100%) nitric acid in carbon tetrachloride was added dropwise to a 0.5 M. solution of diphenyl amine in the same solvent. The temperature was kept at  $-15^{\circ}$  C. while the solution was thoroughly mixed by mechanical stirring. The nitrate salt separated at once as a yellowish-colored amorphous solid. The product was quickly filtered with suction and washed thoroughly with cold carbon tetrachloride. This material had a light yellow color, no doubt due to traces of impurity. It was subjected to analysis for nitrogen according to the usually modified Kjeldahl procedure applied to organic compounds containing oxidized nitrogen. Duplicate results gave 11.73% and 11.89% nitrogen. The theory for diphenyl amine hydronitrate is 12.07% nitrogen.

On standing for several days, the light yellow material acquired a dark greenish-blue color, later becoming practically black. Presumably this color change was due to intra-molecular oxidation and nitration giving a mixture of products very difficult to identify.

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## RING CLOSURE OF N-METHYLCYCLOHEPTYLAMINE

GEORGE H. COLEMAN AND JOSEPH J. CARNES

Previous work in this laboratory has shown that secondary N-chloroalkylamines lose the elements of hydrogen chloride to form heterocyclic amines when heated in sulfuric acid solution. This reaction has now been applied to the preparation of the bicyclic compound tropane (8-Methylazabicyclo [3, 2, 1] octane).

N-Methylcycloheptylamine was prepared from cycloheptanone and methylamine by condensation and reduction of the resulting imine. This compound formed 85-95% yields of the chlorimine when treated with chlorine. The N-chloro-N-methylcycloheptylamine when heated at 65°-67° in 84% sulfuric acid gave tropane in 40-42% yields. This was identified by its physical constants and by the melting points of the picrate, chloroplatinate, and chloroaurate.

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## STABILITY OF CYSTEINE SOLUTIONS

RONALD E. PYLE AND JOSEPH I. ROUTH

The stability of cysteine in acid solution increased with increase in acidity (0.1 to 6N HC 1). When the solutions were made in conductivity water and stored under nitrogen, less than 1 per cent of the cysteine was oxidized in 7 days.

Cysteine did not appear to be oxidized when it was subjected to the conditions obtaining in the hydrolysis of proteins by acids. Treatment of cysteine solutions with decolorizing charcoal pro-