

time should be long enough (several seconds) to prevent the manometer from building up a periodic oscillation.

When in operation the movement of the mercury surface at F is too small to be observed. Temperature measurements on a platinum thermometer in the mercury vapor at about 200° indicated that the pressure remained constant to well within 0.01 mm. while the temperature of the stabilizing volume was varied some 5°. The maximum change in room temperature that the heater can compensate for is, of course, dependent on such factors as the coil dimensions, the power input and the fraction that D is of the total volume. The fact that the control manometer is influenced by changes in room temperature introduces a slight error which, in precision work, could be avoided by the use of a thermostat.

This simple regulator should prove to be useful in many cases where a constant high or low pressure is required.

Other applications of an electrically heated wire in the construction of automatic laboratory devices will be discussed elsewhere.

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Adaptation of the Dimethylglyoxime-Benzidine Test for Cobalt to the Usual Scheme of Qualitative Analysis

BY ALFRED W. SCOTT

A. Chiarothino¹ states that a solution containing cobalt when treated with an alcoholic solution of dimethylglyoxime and benzidine gives an orange-red color. It is further stated that this test must be carried out in a neutral solution.

In nearly all schemes of qualitative analysis the cobalt and nickel precipitate, or the solution obtained from it, is divided into two parts in order to run confirmatory tests for these two cations. The author thought that if the dimethylglyoxime-benzidine test could be adapted to the usual scheme of analysis it might be of advantage since all of the cobalt and nickel precipitate could be used for each test. The following procedure, suitable for use in the ordinary scheme of qualitative analysis, was found to give satisfactory results even when the concentration of nickel was fifty times that of cobalt.

The precipitate of cobalt and nickel sulfides, after separation from the rest of the group three precipitate, is dissolved in hydrochloric acid containing a little nitric acid. The solution is filtered and the filtrate evaporated practically to dryness. The residue is dissolved in a few cc. of water containing 1 cc. of dilute acetic acid. To this solution is added 1 g. of solid

(1) A. Chiarothino, *Industria Chimica*, **8**, 32 (1932).

sodium acetate, 0.5 cc. of a 0.5% alcoholic solution of benzidine and an excess of an alcoholic solution of dimethylglyoxime. If nickel is present the solution is filtered. In the absence of cobalt the filtrate will be colorless to yellow. If cobalt is present the filtrate will have a red coloration. Should the concentration of cobalt be so small that the red color is not very pronounced, it may be intensified by adding solid sodium acetate and allowing to stand for several hours.

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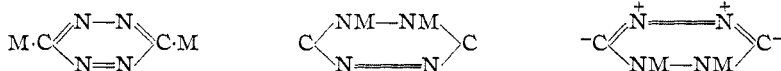
[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF STANFORD UNIVERSITY]

Tetrazine¹

BY DENNISTOUN WOOD, JR., AND F. W. BERGSTROM

In connection with our attempts to prepare nitrogenous derivatives of divalent carbon,² we were interested by Müller's statement³ that "1,2,4,5-tetrazine still contains two atoms of hydrogen which may be replaced by metals, and so have an acid character." In support of his statement, Müller has only his observation that tetrazine and silver nitrate form a precipitate of dark green needles,⁴ but no analysis of this has been made.⁵ On the other hand, Hantzsch and Lehmann⁶ state that tetrazine forms no salts with either acids or bases, but that it reduces silver nitrate and mercuric chloride.

In view of these conflicting statements, it became desirable to see whether or not tetrazine⁷ does form metallic salts corresponding to one or more of the formulas (M is a monovalent metal).



Preparation and Properties of Tetrazine.—Tetrazine was prepared from monochloroacetic acid through the following intermediates: glycine ethyl ester hydrochloride,⁸ ethyl diazoacetate,⁹ sodium bisdiazoacetate,¹⁰ bisdiazoacetic acid,⁸ tetrazine dicarboxylic acid.¹¹ Tetrazine¹² itself was prepared by heating an intimate mixture of its

(1) Abstracted from the doctoral dissertation of Dennistoun Wood, Jr., Stanford University, 1930. Presented at the Denver Meeting of the American Chemical Society, August, 1932.

(2) Wood and Bergstrom, *THIS JOURNAL*, **55**, 3314 (1933).

(3) Müller, *Ber.*, **47**, 3001 (1914).

(4) Curtius, Darapsky and Müller, *ibid.*, **40**, 86 (1907).

(5) Müller, personal communication.

(6) Hantzsch and Lehmann, *Ber.*, **33**, 3678 (1900).

(7) Tetrazine is at the same time a derivative of formamidine (ammono formic acid), hydrazine and diimide.

(8) Hantzsch and Silberrad, *Ber.*, **33**, 70-72 (1900).

(9) Curtius, *J. prakt. Chem.*, [2] **38**, 401-404 (1888); Silberrad, *J. Chem. Soc.*, **81**, 600 (1902).

(10) Curtius and Lang, *J. prakt. Chem.*, [2] **38**, 532 (1888).

(11) Curtius, Darapsky and Müller, *Ber.*, **40**, 1184 (1907).

(12) Curtius, Darapsky and Müller, *ibid.*, **40**, 84-85 (1907).