Notes

A Simple Automatic Pressure Regulator

By C. C. Coffin

One of the most satisfactory and convenient methods of obtaining constant temperatures in the laboratory is to use as a bath the vapor of a liquid boiling under a constant pressure. A pressure regulator is therefore necessary for accurate work with this type of thermostat particularly if the desired temperature corresponds to a vapor pressure much below one atmosphere. If the liquid is mercury, for example, a pressure change of 0.1 mm. at 200° (17.3 mm. Hg) corresponds to a temperature change of 0.17°. At 350° (697.8 mm. Hg) a pressure variation of 0.1 mm. produces a change of only 0.008°. The need of accurate pressure control at the lower pressures is obvious.

The purpose of this note is to describe a simple pressure regulator which has been found to be very satisfactory. It has been used particularly for obtaining constant temperatures between 210 and 280° with the vapor of boiling mercury[1] and would appear to the writer to possess certain advantages over some other regulators[2] which invariably involve the annoyance of mechanically operated valves to vacuum or pressure.

The operation of the regulator is made obvious by the accompanying diagram. The tube A connects with the space above the boiling mercury and with a 20-liter stabilizing volume the pressure changes in which move the manometer M to operate a relay R controlling the nichrome heating coil C. To set the apparatus in operation the pressure in the system is brought by means of tap B to a value slightly less than that desired and the amount of mercury in the manometer is adjusted by taps G and H until the surface on the vacuum side is slightly below the platinum point F. The switch S is then closed, C heats, and when contact is made at F the relay operates to break the heating circuit. The gas in D then cools, contact at F is broken and the cycle is repeated. The amount of mercury in M and the current in C should be adjusted to the rate of cooling of D so that the times of "make" and "break" are approximately equal. This

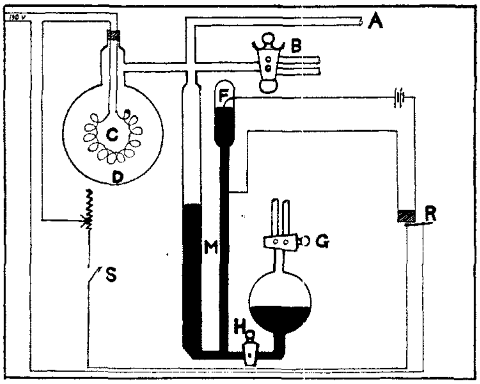

Fig. 1.—Automatic pressure regulator.

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(1) C. C. Coffin, *Can. J. of Research*, **5**, 636 (1931); **6**, 417 (1932); **7**, 75 (1932).

(2) Mathews and Faville, *J. Phys. Chem.*, **22**, 3 (1918); D. F. Smith, *Ind. Eng. Chem.*, **16**, 22 (1924); Carroll, Rollefson and Mathews, This Journal, **47**, 1791 (1925).

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. Chiarothino[1] 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

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(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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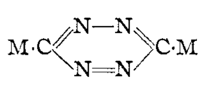
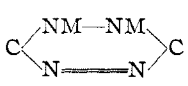
Tetrazine[1]

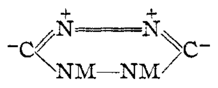
BY DENNISTOUN WOOD, JR., AND F. W. BERGSTROM

In connection with our attempts to prepare nitrogenous derivatives of divalent carbon,[2] we were interested by Müller's statement[3] 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 Lehmann[6] 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 tetrazine[7] 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] Tetrazine[12] itself was prepared by heating an intimate mixture of its

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[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 di-imide.

[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).