

as follows: Use two platinum wires, dipping one in a pure hydrochloric acid solution of a calcium salt and the other in the solution to be tested. Insert the middle of the wires in opposit sides of the Bunsen flame near its base and draw them slowly forward until the looped ends are in the flame. The sodium (and potassium if dichromate has been used) burns off first and leaves the more persistent calcium and strontium in the flame. A decided difference in the flame colors thus brought side by side is caused by even a trace of strontium.

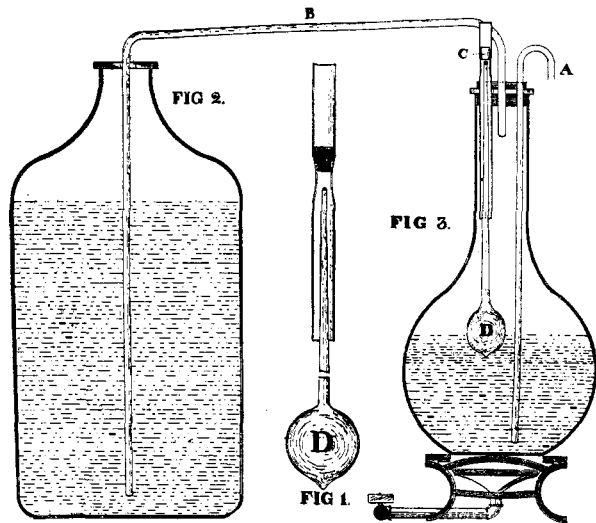
7. For further confirmation of strontium add calcium sulfate solution to a portion of the solution of 3 and boil. A slowly forming precipitate indicates strontium.

The chief advantage of this method lies in the easy and unmistakable identification of calcium in the presence of strontium.

UNIVERSITY OF NASHVILLE,
NASHVILLE, TENN.

NOTES.

A Wash Bottle for Continuous Hot Water Supply.—It is often found very desirable, where a large amount of routine analytical work is done, to have a more convenient means of washing precipitates than that furnished by the common wash bottle. A wash bottle which has given



very good satisfaction in this laboratory is described below in detail.

It consists of a heavy, 5-liter boiling flask (Fig. 3) connected with a large supply bottle (Fig. 2). The boiling flask has a tight-fitting three-holed rubber stopper as shown in the diagram. Through one hole passes a glass tube, A, which is connected by rubber tubing

to the washing-jet. The tube B connects the supply bottle with the boiling flask. As the liquid in the boiling flask is lowered, there is a partial vacuum created which causes water from the supply bottle to pass through B into the boiling flask, thus maintaining the supply.

To allow the escape of steam from the boiling flask and to prevent

the latter from filling to the stopper, due to the condensation caused by the cold water from the supply flask, a special valve (Fig. 1) is provided. This valve consists of a main tube which passes through the rubber stopper of the boiling flask. This tube, which may be ordinary 8 to 12 mm. tubing, is constructed as shown in the diagram in Fig. 1. Just above the constriction the tube is fitted with a ground glass plug. This plug is made the proper size by rotating in the upper part of the constriction a glass rod of proper size, the end of which has been covered with powdered emery. The ground end is then cut off, making the plug (C) as shown. The upper end of the tube is partly closed to prevent the escape of the plug. Into the lower portion of the tube is inserted a float, *D*, which is free to move up through the tube and lift the plug *C*. The length of the float is regulated by the amount of water desired in the boiling flask, which should not be lower than as shown in the diagram.

When the water from the supply flask raises the height of the liquid in the boiling flask, the float *D* is pushed upward till it raises the plug, allowing an inrush of air which destroys the vacuum formed by the condensation.

In order to obviate the risk of bursting the flask by the vacuum produced through condensation of the steam, a safety mercury valve is advisable. This is easily made by bending a glass tube so that one end may be fitted through the stopper of the boiling flask and the other end dipping into a small vessel of mercury. About six inches from the lower end of the tube should be a bulb large enough to hold twice the amount of mercury used.

The bulb of the float should be as large as the neck of the flask will admit. The whole apparatus can be constructed by any one of ordinary skill.

G. E. BOLTZ.

OHIO AGRICULTURAL EXPERIMENT STATION, WOOSTER, O.

Some Experiments on the Formation of Ammonia from Its Elements.—The recent papers by Lipski¹ and by Haber,² on the direct synthesis of ammonia from its elements, have brought to mind some few random observations made by the writer with a similar aim in mind, during the last two years. The experiments were almost entirely qualitative, and were discontinued partly for the reason that they did not seem to offer very much encouragement for further study. The experiments may be divided into three classes: (a) purely catalytic, (b) semi-catalytic, and (c) electrical.

(a) The catalytic experiments consisted in the passage of the mix-

¹ "Ueber Synthese des Ammoniaks aus den Elementen," Dissert., Herzogl. Tech. Hochschule Carolo-Wilhelm. Braunschweig, Halle, 1909.

² *Chem. Ztg.*, 34, 345.

ture $3\text{H}_2 + \text{N}_2$ over various reagents which seemed to offer some possibility of effecting the union of the nitrogen and hydrogen. The gases were passed over at atmospheric pressure, and at temperatures of 20° , 100° , 300° and red heat, and the catalytic agents tried were platinized asbestos, palladium-asbestos, reduced iron, and finely powdered commercial 85 per cent. ferromanganese. Traces of ammonia were found in the water through which the outflowing gases were passed, from each of these agents, the ferromanganese showing decidedly the greatest activity. The ammonia was tested for by means of Nessler's reagent, and in most cases showed amounts comparable to the higher standards used in sanitary water analysis—say a maximum of 0.00006 gram of NH_3 —after the passage of volumes of gases varying from one liter to ten liters.

(b) The semi-catalytic experiments consisted in passing mixtures of steam and nitrogen at 100° and atmospheric pressure over substances which would decompose the water vapor, and taking up the oxygen, theoretically leave the hydrogen in the nascent condition. Iron filings, zinc dust, and sodium amalgam (2 per cent.) were tried and as in the (a) experiments, traces of ammonia were found in the water through which the gases were drawn, but again not in sufficient quantity to warrant further development with the materials used.

Steam passed over calcium cyanamide yielded ammonia in abundance, as is well known, but this is a two-stage process, and not strictly in line with the other experiments.

(c) The third series of experiments was conducted at the suggestion of Dr. Norman Roberts, and with his aid and direction. For the most part they consisted in the passage of an electric spark between platinum electrodes in a mixture of hydrogen, nitrogen and hydrochloric acid gases, under both atmospheric and increased pressure. Fumes were frequently seen in the reaction vessel following the sparking, and these appeared to settle on the walls of the container. Yet when the vessel was washed out with ammonia-free water, and the wash water tested with Nessler's reagent, the amount of ammonia present was too small to warrant these fumes being considered as ammonium chloride.

Apparently the synthesis of ammonia *can* be effected in any of these ways, and it is possible that if the action were continued for a sufficient length of time upon a limited quantity of mixed gases, under conditions such that the ammonia would be absorbed as soon as formed, the yield of ammonia might be approximately quantitative, but the time element is of such magnitude as to render any of these processes of no merit practically.

Of course, one difficulty which is inevitably present in any work of this kind is the ease with which ammonia dissociates at high temperatures, and it is of interest to note that in those experiments which have yielded

the best results in the papers above cited, pressures ranging from 11 to 200 atmospheres have been employed. F. ALEX. McDERMOTT.

WASHINGTON, D. C.

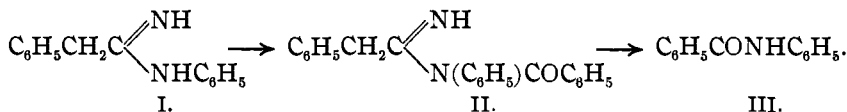
[CONTRIBUTIONS FROM THE SHEFFIELD LABORATORY OF YALE UNIVERSITY.]

BENZOYLPHENYLACETAMIDE.

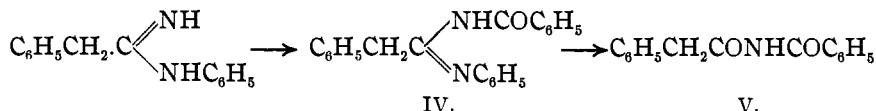
BY TREAT B. JOHNSON AND LEWIS H. CHERNOFF.

Received January 25, 1911.

In a paper entitled "The Molecular Rearrangement of Unsymmetrical Acylamidines into Isomeric Symmetrical Derivatives", Wheeler, Johnson and McFarland¹ described the behavior of benzoylchloride towards phenylacetophenylamidine, I. They observed that they reacted in anhydrous ether, at ordinary temperature, giving an unsymmetrical benzoyl derivative, II, melting at 110-111°. The structure of this compound was established by the fact that it smoothly underwent hydrolysis with hydrochloric acid, giving benzanilide, III.



In one experiment, however, in which chloroform was used as the solvent instead of ether, the reaction was less smooth and the primary products of the reaction—acylamidines—were not isolated. Under these conditions a mixture of benzanilide and a compound, to which they assigned the structure of benzoylphenylacetamide, V, was obtained. The latter contained 6.1 per cent. nitrogen and it was soluble in cold alkali and melted at 129-130°. An amide of this structure would result by the hydrolysis of a symmetrical benzoyl derivative, IV.



Our attention has recently been called, by the revisers of Beilstein's Handbuch, to the fact that this diacidamide, V, had previously been described by Colby and Dodge,² who assigned to it the much higher melting point of 171°. They obtained it by heating benzonitrile, VI, and phenylacetone, VIII, with phenylacetic, VII, and benzoic acids, IX, respectively at a high temperature. They state that the same compound, melting at 171°, was obtained in both reactions, and that it underwent hydrolysis with alkali, giving phenylacetic and benzoic acids. Their syntheses are expressed by the following equations:

¹ THIS JOURNAL, 25, 787.

² Am. Chem. J., 13, 6.