fering acid was introduced, gave a distinctly visible, black precipitate in a transparent, light brown mother liquor.

An inspection of Tables II and III shows that by boiling the reaction mixture, the interference may in most cases be overcome. There are, however, many exceptions to this generalization.

Summary.

(1) Attention has been called to the interference of thiocyanates, ferrocyanides and ferricyanides in the detection of iodides with palladium.

(2) Data have been furnished to show the nature and extent of the several interferences.

(3) It has been found that an excess of palladium favors the detection of iodides in the presence of thiocyanates.

(4) The interference occasioned by ferrocyanides and ferricyanides may in most instances be overcome by boiling the reaction mixture.

NEW YORK, N. Y.

NOTES.

Determination of Carbon Dioxide in Carbonates by Diminished Pressure.—Realizing the difficulty experienced by students in obtaining concordant results when determining the percentage of CO_2 in carbonates by the absorption method, it seems worth while to mention a slight modification which has been adopted in this laboratory together with some of the results obtained.

The apparatus is arranged in the usual manner for the production and absorption of the gas but instead of the ordinary water suction pump an eight-liter aspirating bottle is used for the testing and finally for drawing air through. The aspirator is provided with a glass stopcock, which is opened wide before adding the acid to the reaction flask, thus bringing the system under *slightly diminished* pressure and it is left open during the entire operation, the flow of gas being regulated by means of a stopcock in the dropping funnel. The flow of gas ceases automatically when the aspirator is empty of water, which commends itself favorably to institutions having short laboratory periods.

The following results were obtained by four men in their first semester work in quantitative analysis: Correct percentage of CO_2, 43.94, 44.96, 45.67, 46.40. Percentage as determined, 43.95, 44.95, 45.64, 46.36. Eight liters of water were used in the aspirator in the above experiments. The calcium chloride used in these experiments was prepared by evaporating to dryness a solution of calcium chloride containing a few grams of ammonium chloride and then volatilizing the excess of ammonium chloride.

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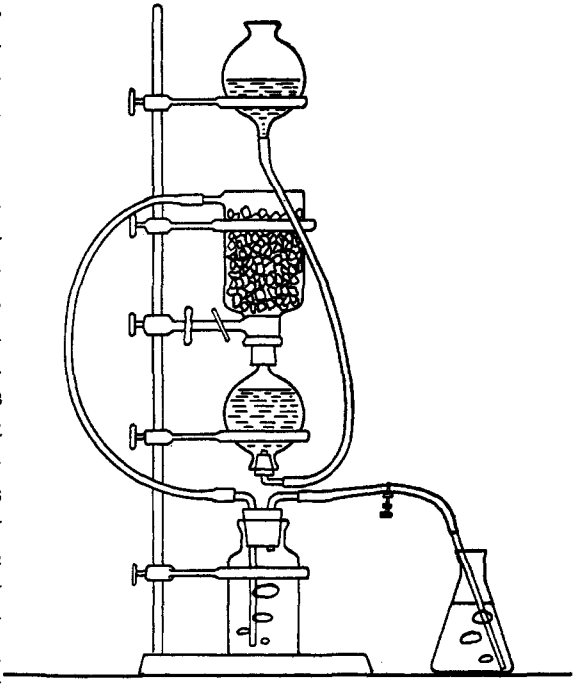
A Simple Gas Generator.—During the present shortage in laboratory glassware, we have found it difficult to purchase Kipp generators. The generator as indicated in the accompanying diagram is constructed with apparatus found in the stock room of every laboratory. A gas washing bottle is added, making the outfit compact and quite portable. The generator has proved quite satisfactory for all purposes where a Kipp was previously used. It is more easily cleaned, and generation may be positively stopped at any time by lowering the acid storage bulb.



R. O. FERNANDEZ.

An Efficient Desiccator.—Most systems of desiccation involve exposure of the material to an atmosphere of gas which has been, or is being, gradually dried over some drying agent, the moisture absorbed by the gas being allowed to pass over to the drying agent simply by diffusion. This process is slow and tedious. Recently, a form of desiccator was proposed in which the gas is kept in motion by means of a fan. This allows more rapid desiccation, but absorption of moisture from the material by the gas still depends on diffusion alone.

The author has recently employed an apparatus which seems to obviate these objections. It is shown in the accompanying figure. The apparatus consists of a Büchner funnel (*A*), connected below, through a stopcock (S′), with a safety flask, which, in turn, is attached to a water pump. Resting on the funnel is a rather heavy brass disk (B), somewhat larger than the funnel used, on the lower surface of which is cemented a ring, or sheet, of rubber (R), to make an air-tight joint when pressure is applied. Pressure is brought to bear on the disk by means of three screws acting between the disk and a metal collar (C). Heavy rubber tubing (R′) prevents the collar from exerting unequal pressure on the funnel. In

the center of the disk is set a brass tube a few centimeters in length. To this is attached a stopcock (S) by means of heavy rubber tubing (R″). The stopcock is further attached to a system of drying bottles or towers.

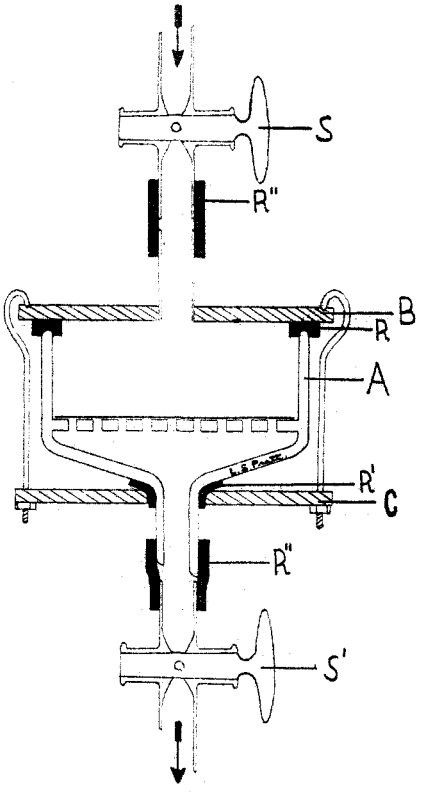


Fig. 1.

In case the material can be desiccated at an elevated temperature the apparatus shown in Fig. 1 may be inclosed in an air bath, which is then heated to the desired point. If the temperature used is over 100°, some material more stable than rubber should be used for packing.

This method could also be applied to Gooch crucibles as well as to Büchner funnels.

The material to be desiccated is either filtered into or subsequently placed in the funnel. The disk (B) is placed in position and pressure applied to close the joint tightly. The pump is then started and a slow current of air (or inert gas if desirable) drawn through the system.

In this case carefully dried gas is *forced through* the material and desiccation is very rapid.

By means of the stopcock S, it is possible to hasten the action by creating a partial vacuum in the funnel. If the stopcock be so set that a very slow current of gas passes, all the advantages of vacuum desiccation are obtained, still combined, however, with *forced penetration* of the material by the gas, and constant removal of the partially saturated gas.

The stopcock S′ permits the maintenance of a vacuum in the funnel in case it may be desirable to use the pump intermittently. The advantages of this type of desiccator are the following:

1. Simplicity of construction and operation.

2. Rapidity of desiccation, owing to *forced penetration* of the material by the dry gas, and the constant removal of the latter when partially saturated.

3. Adaptability to vacuum desiccation.[1]

[1] If the stopcock above the funnel is so set that a vacuum of 100 mm. or less is maintained within the funnel it is scarcely necessary to dry the air which enters since, with an initial humidity of 60%, the reduction of pressure within the funnel to 100 mm. reduces the humidity of the air entering the funnel to less than 10%.

4. Adaptability to desiccation at elevated temperatures.

L. S. PRATT.

UNIVERSITY OF VIRGINIA,
January 2, 1917.

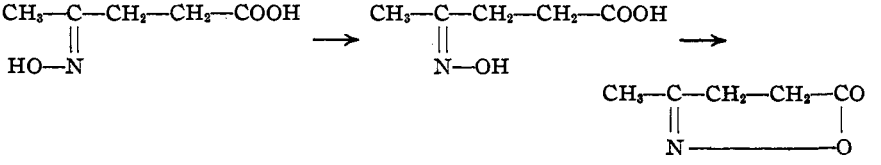
[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF THE UNIVERSITY OF WASHINGTON.]

ANHYDROXIMES. II.

BY ROBERT EVSTAFIEFF ROSE AND WINFIELD SCOTT, JR.[1]

Received November 8, 1916.

The β, γ and δ oximino acids, *i. e.*, the oximes of β, γ and δ carbonyl acids, show a strong tendency to lose water between the carboxyl and oximino groups, being thereby converted into ring structures containing carbon, oxygen, and nitrogen. For these cyclic compounds, whose properties are intermediate between those of lactones and acid anhydrides, one of us has suggested the name "anhydroxime."[2] The tendency towards ring formation in the oximino acids is greater than it is in the case of dicarboxylic acids but less than it is in the hydroxy acids. Thus acid anhydrides are usually formed only under the influence of heat or of a dehydrating agent and the rings formed are five or six membered;[3] lactones form spontaneously in the case of γ and δ hydroxy acids, while lactone rings of four to eight members have been prepared. Anhydroximes are formed spontaneously to give five and six membered cyclic compounds. Too little has been done to allow of stating that the ring cannot contain more or less atoms, but it would at least appear that ring formation does not proceed as readily as in the case of hydroxy acids. In this connection it should be remembered that configuration may influence the ease of formation of an anhydroxime, *e. g.*, the oxime of levulinic acid loses water only on prolonged heating with sulfuric acid,[4] but its behavior on undergoing the Beckmann rearrangement shows it to possess the *anti* configuration; inversion must then precede dehydration.



[1] The material presented here is used by Mr. Winfield Scott in his thesis in part fulfillment of the requirements for the M.S. degree of the University of Washington.

[2] Rose, THIS JOURNAL, 33, 388 (1911).

[3] The existence of malonic anhydride is due to a special reaction, that of carbon suboxide on acetic acid. The so-called anhydrides of the higher homologs of glutaric acid appear to possess much higher molecular complexity than they should were they really internal anhydrides.

[4] Rischbieth, *Ber.*, 20, 2669 (1887); Hantzsch and Werner, *Ibid.*, 23, 11 (1890).