For the determination of nitrogen by the Kjeldahl method it is best to connect the ends of the condenser tubes with glass tubes, the latter to dip into standard acid contained in the receiving vessels. Safety bulbs may also be placed at the tops of the distilling flasks connecting the same with the condenser tubes.

The first set of stills of this design was built for the laboratory of the Cincinnati Water Commission in 1898. It consisted of 10 units. Since that time three other sets have been built, all of which give satisfaction.

To determine the free and albuminoid ammonia in sands and in sewage, it has been found most convenient to place them in a 250 cc. Kjeldahl flask, and to pass through them, by means of a glass tube which should extend nearly to the bottom of the flask, steam from ammonia-free water. This ammonia-free steam is best generated in a closed copper vessel placed at one side of the condenser tank and heated by a large burner. The steam is conducted along the top of the condenser in a $\frac{3}{8}$ inch metal pipe. At suitable intervals $\frac{1}{4}$ inch tees with metal cocks are placed, from which steam can be taken for sand and sewage work.

If desirable, the burners can be easily removed and smaller rings can be used to support the Kjeldahl flasks.

Reagents are added to the flasks by means of a long-stemmed funnel.

A PROCESS FOR THE DETERMINATION OF CARBON DIOXIDE IN CARBONATES.

BY R. E. DIVINE.

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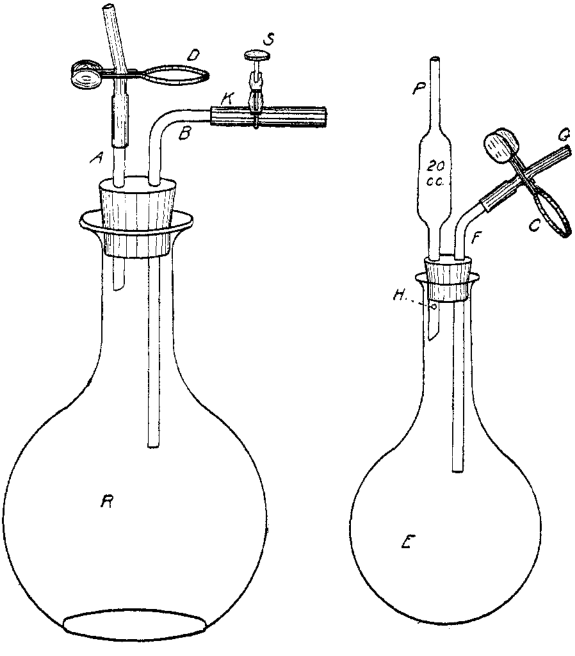
THIS process is based on the principle of Pettenkofer's process; namely, absorption of the carbon dioxide by a measured amount of standard baryta water (solution of barium hydroxide), and titration of the excess of the latter with a standard acid. The apparatus about to be described is simple in construction and may be assembled from materials available in almost every laboratory, and the process, if carried out with a reasonable amount of care, should yield fairly accurate results with anyone. It does not require the time and experience necessary in order to obtain good results by the ordinary gravimetric method.

The apparatus consists of a round-bottomed evolution flask E of about 300 cc. capacity, fitted with a rubber stopper and two tubes P and F, and as a receiving flask a 1 quart Bunsen filtering flask R fitted with two tubes A and B.

P is a 20 cc. pipette cut off so as to leave about 3 inches on each side of the bulb. The lower end is ground to a point on an ordinary grindstone, and at about 1 inch above the point there is a small hole, H, blown into the side of the tube which enables the condensed drops of liquid to fall freely from the end.



F is a tube bent sufficiently to clear the bulb on P, projecting lower into the flask than P, and fitted on the upper end with a short piece of rubber tube G and a pinch-cock C.

The tube B of the receiving flask is fitted with a piece of heavy pressure tubing K, and a screw-cock S. Tube A is fitted with a short piece of soft tubing and a pinch-cock D.

The empty receiving flask R is first placed on a steam-bath in a place free from draughts and steam generated in a small flask or can (I use an ether can) is passed through A until the flask is hot, and steam issues through B quite freely. S is now closed tightly, then D, and the apparatus disconnected from the source of steam and placed where it may cool. The evolution flask is now connected with the source of steam at G, and, after the air is expelled and steam issues strongly through P, the rubber tube K of the receiving flask is forced on P, then C is closed and the source of steam disconnected; the apparatus after a minute's cooling is ready to receive the carbonate. This is introduced by placing a small cut-off funnel in the end of G, washing 1 gram of the finely pulverized carbonate into the funnel, then by cautiously opening the pinch-cock C, the carbonate may be completely washed into the flask.

The screw-cock S is now opened wide. Twenty-five cc. of 10 per cent. sulphuric acid[1] are now allowed to flow on the carbonate and E is then heated over a low flame to boiling and boiled until the tube G which has been collapsed by the external pressure swells out nearly to normal size. S is then closed tightly and the heat removed. During the boiling E is allowed to hang supported only by the heavy tube K of the receiving flask. This tends to lessen in great measure the force of bumping in E which takes place as soon as the carbon dioxide is nearly boiled out of the liquid. The boiling must, however, be continued for some time in order to completely displace the carbon dioxide in E by aqueous vapor; E is now disconnected from R at K, and after three or four minutes' cooling 500 cc. of the standard baryta water are allowed to flow into R. This is accomplished without contact with the carbon dioxide of the air in the following manner: An ordinary graduated half-liter flask is fitted with a rubber stopper having three holes. Through one of these passes a tube to the bottom of the flask after the manner of a wash-bottle but fitting closely to the flask so as to remove the contents completely. Through another hole passes loosely a short tube connecting with a wash-bottle containing strong caustic potash solution for washing the air admitted to the flask. Through the remaining hole passes a short tube connecting by means of a long rubber tube fitted with a pinch-cock with a bottle of distilled water on a shelf. The standard baryta solution is of

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[1] If the substance forms insoluble sulphates use tartaric acid.

such a strength that 500 cc. require 48 to 50 cc. of half-normal hydrochloric acid for neutralization.

The stopper and tubes are first removed from the measuring flask which is filled with air free from carbon dioxide, and it is then quickly filled to the mark by the siphon on the stock baryta bottle. The stopper is then replaced and the siphon of the measuring flask connected with K of the receiving flask. By opening the screw-cock S the baryta water flows into R. S is closed as the last drops enter the siphon of the measuring flask, the tube connecting with the wash-bottle pulled out of the stopper, and 20 cc. or 30 cc. of distilled water allowed to flow into the flask. This is then sucked over after replacing the wash-bottle connection, and then 20 or 30 cc. more distilled water sucked over in like manner, taking care that not much air enters R which is now shaken thoroughly, and after a minute or two washed air is allowed to enter. The flask is then shaken occasionally for about ten minutes, then air allowed to enter, disconnected from the measuring apparatus, the tubes and stopper rinsed quickly with the jet of distilled water and then immediately titrated with half-normal hydrochloric acid with phenolphthalein as indicator. The acid must not be run in too quickly and the contents of the flask must be thoroughly agitated while the acid is run in so as to avoid decomposition of barium carbonate with loss of carbon dioxide and consequent lowering of the result. The whole process including the standardizing of the baryta solution, weighing the carbonate, etc., can be quite expeditiously carried out and the results are as accurate as those of most technical processes. In working with C. P. sodium carbonate, I have obtained the following figures: 41.33, 41.42, 41.51, 41.32, 41.37, 41.34, and 41.36 per cent. Theory requires 41.51 per cent. Some trials with barium carbonate gave 21.99, 22.05, 21.98, and 21.91 per cent. Theory requires 22.33 per cent.; the barium carbonate contained quite a little chlorine. In this case tartaric acid was used to decompose the carbonate.

Freshly precipitated barium carbonate is sufficiently soluble to give a very faint alkaline reaction to the liquid, and this must be borne in mind if the color returns faintly after titrating.

BUFFALO, May 25, 1900.