APPARATUS FOR THE DETERMINATION OF AMMONIA IN WATER, BY THE WANKLYN METHOD, AND TOTAL NITROGEN BY THE KJELDAHL METHOD.

By Robert Spurr Weston.

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THE introduction of the ammonia method by Wanklyn[1] in 1867 has been followed by many modifications of the original apparatus. Like modifications have followed the introduction of the method introduced by Kjeldahl[2] in 1883.

Wanklyn originally used a tubulated retort and a Liebig condenser. The copper condenser contained a glass tube 90 cm. long and 3 cm. in diameter, thus allowing the beak of the retort to enter the condenser tube. The joint was made by wrapping a little writing paper around the beak of the retort where it entered the condenser tube. Such an apparatus is friable and unhandy.

This single still has been improved upon by various analysts, until there are several forms in use which satisfy most requirements.

An excellent form is described by Cairns[3] and consists of a 2 liter, glass stoppered flask with a side-neck tube. The tube is bent so as to point vertically downward at a convenient distance from the flask. A copper condenser carrying a block-tin zigzag is attached to the neck of the flask by a rubber tube, and the lower end of the condenser is arched upward to prevent the condensed atmospheric moisture from contaminating the distillate. Leffmann[4] makes use of a tubulated retort with a bent neck, connected with a spiral glass worm.

Perhaps the most durable and, all things considered, most convenient form of single still is the one designed by Dr. A. H. Gill and used for several years by Mrs. E. H. Richards in the laboratory of the Massachusetts Board of Health.

This still consists of a flask holding about 1300 cc., which is closed by a cork carrying a $\frac{3}{16}$ inch glass tube. This glass tube is bent so as to enter a $\frac{1}{4}$ inch straight block-tin condenser

[1] Wanklyn: "Water Analysis," 1868.

[2] *Ztschr. anal. Chem.*, **23**, 557.

[3] "Quantitative Analysis," New York, 1896, p. 271.

[4] Leffmann: "Examination of Water," Philadelphia, 1895.

tube. The joint between the glass and tin tubes is made with a cork which is bored half way on one side to receive the glass, and half way on the other side to receive the tin tube. A rubber collar is slipped on to the lower end of the condenser tube to prevent the contamination of the distillate. The condenser is perpendicular and the neck of the flask makes an angle of 45° with it.

This apparatus has many advantages. It is easily boiled clean. Breakage of parts is not frequent, and the broken parts themselves can be replaced at a small cost. The inclined flask is also a decided advantage when the contents have a tendency to bump.

When many stills are used at once, however, these single stills are not so convenient. They take up much bench room unnecessarily, and when the condenser tubes are run through a common cooling tank, the distilling and receiving vessels are on opposite sides and are therefore somewhat inconveniently placed. Perhaps the best arrangement in using a common condenser is that described by Mason.[1]

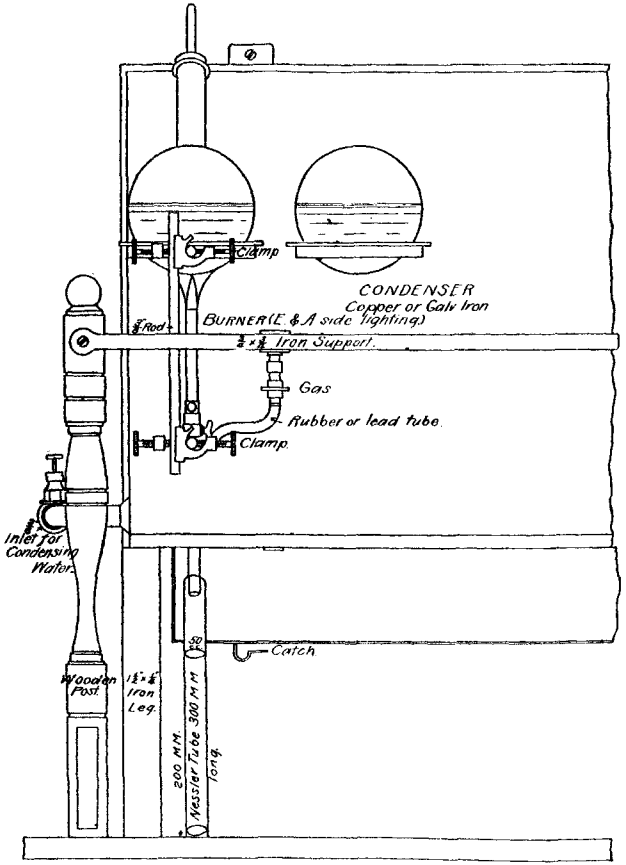
For the determination of nitrogen according to the Kjeldahl method, many forms of still have been introduced. It has been desirable from the beginning, however, to arrange groups of stills. Two forms are in general use.

At the Halle Agricultural Experiment Station an apparatus[2] is used which makes use of an air condenser, but the general arrangement of parts is similar to that used by Mason for water analysis, though Erlenmeyer flasks are used for distilling- and receiving-vessels.

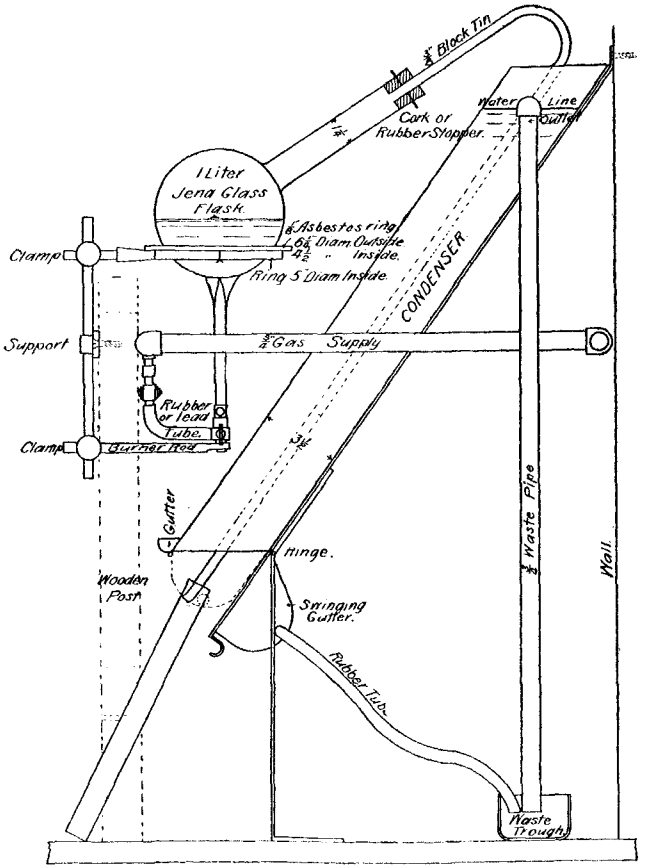
The most important improvements in the construction of sets of stills were made by the chemists of the United States Department of Agriculture, and are described by Professor S. W. Johnson in Bulletin No. 10 of the Division of Chemistry.

This apparatus,[3] of course, is well known and possesses the advantage that it can be operated from one side of the condenser tank. The distilling flasks are raised above the bench, so that it is possible to reach under them for the purpose of attending to the receiving flasks. The distilling flasks are supported upon

[1] "Water Analysis," New York, 1899.
[2] Wiley: "Agricultural Analysis," Vol. II, 203.
[3] *J. Anal. Chem.*, 4, 179; also Wiley: *loc. cit.*, 208.



FRONT ELEVATION.



SIDE ELEVATION.

an iron shelf. They are joined to the helical block-tin condenser tubes by a safety bulb and rubber connectors. This apparatus was adapted to water analysis by Hazen and Clark.[1]

This apparatus was further improved by the chemists of the Massachusetts Board of Health by bringing the condensing tubes out above the open top of the condenser tank, and by connecting them directly with the stoppers of the distilling flasks, thus avoiding a rubber connection between the distilling flask and the condenser tube. This apparatus was used by the writer for several years, and was found to be quite convenient; nevertheless, some improvements suggested themselves during that time, and in designing a group of ten stills the form described below was developed.

The distilling flasks of the Johnson apparatus are supported on an iron distilling shelf, and the burners are supported on another shelf placed beneath. The metal shelves are hard to keep clean and are, of course, non-adjustable. The distilling flasks, moreover, are placed vertically, and the contents are more liable to be projected into the condenser on that account than if they are inclined. The condenser is unnecessarily large, and the receiving tubes are placed so far under the apparatus as to be inconvenient to tend.

The apparatus described below was designed to overcome the faults of the older apparatus. The drawing on page 470 shows the construction quite plainly. Any desired number of stills can be provided for.

CONDENSER TANK.

The condenser tank is built of copper or galvanized iron. The galvanized iron should be japanned. It is inclined so as to bring the lower ends well forward, and is 7.2 inches in length for each unit. The tank is 3.5 inches thick, and is rhomboidal in section, with sides 25 inches and 4.25 inches long respectively. The condenser tubes are of block tin $\frac{3}{8}$ inches in diameter and about 36 inches long. They are straight, not helical. They are soldered into the bottom of the condenser tank, projecting 2 inches below the bottom of the same. They are not supported

[1] Report of Massachusetts Board of Health on "Purification of Water and Sewage," 1890, p. 710.

except at the bottom of the tank, and are sufficiently pliant to allow connection with the flasks to be made with ease.

A swinging gutter is attached to the bottom of the condenser. This gutter can be swung under the condenser tubes when "boiling out" the apparatus. A small upturned gutter is fashioned around the bottom of the tank to catch any possible condensation. The inclined tank brings the tubes well within reach. The tank is supported by legs to the bench and by lugs to the wall.

DISTILLING STAND.

The whole system of flasks and burners is supported by means of a cross bar resting on two or more posts. The $\frac{3}{4}$ inch square, iron cross bar is drilled with holes $7\frac{1}{4}$ inches apart, and $\frac{3}{8}$ inch rods, 10 inches long, are driven into the holes. These rods project 6 inches above and 4 inches below the cross bar. Double clamps hold the rods for the burners and the 5 inch retort stand rings. The base of a Bunsen burner (E and A side lighting) is unscrewed and the burner itself is fitted with a $\frac{3}{8}$ inch rod. The above arrangement permits the flask and the burner to be moved (within limits) in any direction and at will. The flasks are supported by rings of asbestos ($\frac{1}{8}$ inch thick, $6\frac{1}{4}$ inches outside diameter, and $4\frac{1}{2}$ inches inside diameter); these asbestos rings in turn are supported by the iron rings, being secured to the latter by wires. This furnishes a very neat and springy support for the flasks, and one which, to a great extent, relieves the jar due to bumping and the consequent breaking of flasks. A water supply is provided for the condenser, entering near its bottom at one end and wasting near its top at the opposite end. All parts of the apparatus are accessible, and the flasks can be connected and disconnected with one hand, the spring of the condenser tubes helping to hold the stoppers in place.

The gas pipe is supported on the backs of the wooden posts and $\frac{3}{8}$ inch hose cocks are placed to the right of each still support; these cocks are connected with the burners by lead or rubber tubes. Either antimony rubber or selected cork stoppers can be used, the latter, perhaps, to be preferred. The ends of the condenser tube pass directly through the stoppers. If desirable, copper flasks can be used. The writer prefers to use the standard 1-liter, Jena glass round-bottomed flask.

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.