bility. It is hoped, therefore, that this simple method may be of value in furnishing chemists with a means of determining within reasonable limits the relative availability of the various nitrogenous materials submitted to them for examination.

LABORATORY OF THE NEW JERSEY STATE
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AN APPARATUS FOR DETERMINING FAT.

BY H. J. WHEELER AND B. L. HARTWELL.

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THE Knorr apparatus[1] for the extraction of fat, which was a great improvement over preceding forms, enabled one to dispense with the ground-glass and cork connections formerly in use, by the employment of a mercury seal.

The mercury is carried in a channel which encircles the neck of the flask at the base. In order to maintain the connection between the flask and the glass part immediately above it, the two are held together by rubber bands. To avoid the use of the bands the condensers may be made movable so they can be lowered sufficiently to connect with the flasks, or if the condensers are stationary the bath which supports the flasks may be made movable. In many instances, particularly in the case of batteries of considerable size, neither of these plans is convenient and at the best, unless great care is exercised, the flasks are liable to break during the adjustment. The use of rubber bands is unsatisfactory owing to their liability to slip or break.

A later modification of the Knorr flask was one which had a glass channel attached to the neck of the flask near the top. This modification enabled one to reduce the quantity of ether liable to collect outside of the neck, but the flask was necessarily fragile and expensive. The apparatus described below was designed to remove some of the difficulties connected with that devised by Knorr and to accomplish certain other desirable objects. The improvements attempted may be summarized as follows:

1. The use of a simple flask which can be readily cleaned and replaced at small expense.

2. The employment of a rubber cup to carry the requisite

[1] Bull. 28, U. S. Dept. of Agriculture, Div. of Chemistry (1890), p. 96.

amount of mercury for sealing, and at the same time bind the flask firmly to the other parts of the apparatus.

3. The reduction to a minimum of the amount of ether, which can collect around the outside of the neck of the flask, by the ready adjustment of the cup to any point upon the neck.

4. The bending of the end of the tube from which the liquid ether drops upon the substance which is undergoing extraction, so that the ether will be delivered from the center and not flow down the glass.

5. The prolongation of the tube mentioned in (4) so that it may support the upper ends of extraction tubes or thimbles of different lengths.

6. The collection of the ether in one receptacle at the end of the extraction without disconnecting the apparatus.

7. The maintenance of the ether in a dry condition.

8. The loss of the least possible amount of ether vapor during the extraction and at its close.

Below is given a description of an apparatus designed to meet the conditions just mentioned. This apparatus, made in accordance with our specifications by Richards & Co., of New York, has already been in successful operation at considerable intervals for more than a year without apparent depreciation in the quality of the rubber cups and without the breakage of an extractor or other expensive glass parts.

A. A plain flask of 50 to 60 cc. capacity, having a straight neck.

B. A rubber cup channeled to carry mercury for sealing the connection between the flask and the lower portion F of the extractor.

C. A channel for mercury, showing the latter forced upward by the glass so as to cover the rubber, at the same time sealing the connection between the neck of the flask and the glass extractor which passes into the rubber cup at the outer margin of the channel.

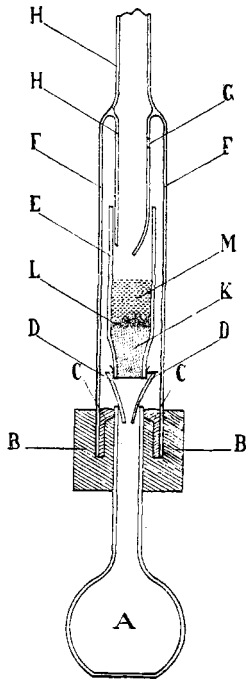

Fig. 1.

D. A small funnel to conduct the ether and dissolved fat into the flask. This is grooved where it enters the neck of the flask so that the small amount of ether which accumulates between the neck and the extractor may flow downward.

Resting upon the funnel D is the thimble which contains the material K to be extracted. The bottom of this thimble is covered with fat-free filter-paper fastened to the outside of the thimble by means of wire. The ends of the wire rest upon the edge of the funnel so as to leave space for the passage upward of ether vapor.

L. Fat-free cotton to facilitate the distribution of the ether at the beginning of or during the extraction.

M. Ether, which is maintained above the substance K in considerable quantity during most of the period of extraction. This is accomplished by raising the temperature of the flask A so that the ether may evaporate as rapidly as it filters into the flask.

E. The glass extraction tube or thimble.

F. Walls of the lower portion of the extractor.

H. The lower end of the condensing portion of the extractor, perforated at G to permit ether vapor to pass upward, and bent at an angle at the bottom so that the drops of liquid ether are delivered at the center. This part serves as a support for the thimble and is made sufficiently long to permit of the use of thimbles of various lengths.

The upper condensing portion of the extractor is of straight thin-walled glass and sufficiently long to insure condensation of the ether vapor.

The apparatus may be used singly or in battery form. The outer tubes of the condensers may be of glass, but if hot water is used in them, as described later, they should be made of metal. They are connected with the inner ones by means of rubber stoppers.

The rubber cup B binds the flask firmly to the extractor, so that rubber bands or devices for lowering or raising the bath below or the condensers above for maintaining the connection with the flask, as in the Knorr apparatus, are rendered unnecessary.

The cup also carries continually in its channel a proper amount of mercury for sealing.

In connecting and disconnecting the flask a straight cork fitting the inside of the neck of the flask A is inserted to prevent anything from falling into it as it passes through the rubber cup.

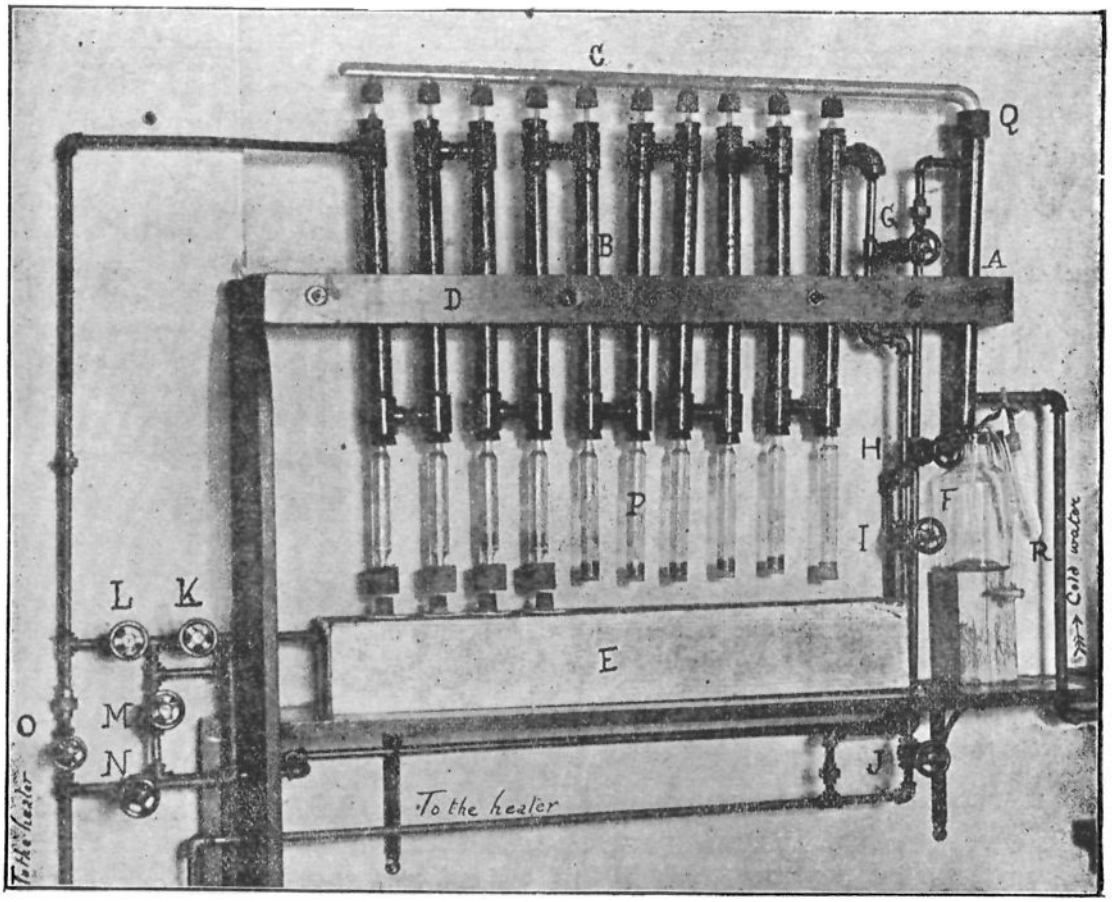

Fig. 2.

Fig. 2 shows a battery of extractors so arranged that the ether may, without disconnecting the flasks, be distilled at the close of the extraction and collected practically free from moisture. The source of heat for all the operations is an instantaneous water heater located somewhat below and (to avoid danger from fire) to the left of the battery.

BATTERY WITHOUT ARRANGEMENT FOR RECOVERING ETHER.

In laboratories where many fat determinations are made, the use of extractors in battery form is desirable. If, owing to the nature of the material to be extracted, the amount of ether necessary is considered too small to render its recovery unprofitable all of the piping necessary is that required to conduct water to

and from condenser B, and to connect the instantaneous water heater with each end of the bath E. If the apparatus is used in this manner the condenser A may be omitted, and the calcium chloride tube R attached directly to the right-hand end of tube C, which in that case may be made shorter than otherwise. A rubber tube is placed on the end of the calcium chloride tube so that it may be closed by means of a pinch-cock.

After beginning the extraction the end of the calcium chloride tube is kept open until the extractors are filled with ether vapor when it is closed and need not be opened again during the entire operation. If preferred, an automatic valve opening by pressure from within may replace the rubber tube and pinch-cock.

BATTERY COMPLETE AS SHOWN IN FIG. 2.

For the recovery of the ether the condenser A is necessary and also additional piping. The detailed arrangement of the piping will be determined by the location of the heater, the waste pipes and water supply pipes in individual laboratories.

As shown in Fig. 2, cold water after passing condenser A flows to the water heater and thence into the bath E. When the bath is once filled the water is allowed to pass through the condensers B to the waste pipe located below and to the right of valve K. During the extraction a continuous circulation is maintained from the lower right-hand end of bath E to the water heater, and thence back through the left-hand end of the bath. At the close of the extraction the cold water passing through condenser A flows to the water heater, thence to the left-hand member of battery B, forcing the cold water out underneath bath E into the waste pipe. As soon as hot water appears in the last member of battery B at the right, the current is diverted into bath E to vaporize the ether and assist in driving it over through condenser A. The water-level in bath E in the meantime is controlled by valve K in the pipe leading from the bottom of the bath to the waste; this pipe may also serve to siphon the water from the bath.

The inner tube of condenser A is straight and small enough to pass inside of tube C at the point Q where the latter is turned downward to meet it. This connection is sealed by mercury just as in the case of the flask and extractor (Fig. 1).

The tips of the inner tubes projecting through the tops of the

metal condensers B are drawn down so that they can enter the downward projections on the tube C. Short pieces of heavy-walled rubber tubing are slipped over these, and over the pieces of tubing are passed bored rubber stoppers which clasp them and the outside of the downward projections of tube C. The stopper projects considerably above the tubing so as to form a cup (about the tip) which holds sufficient mercury to produce a perfect seal over the rubber and between the two glass parts.

In setting up the apparatus the extractors P may be inserted in the condensers B before the latter are clamped in position.

The flask F is connected with the inner tube of condenser A by an ordinary cork coated with plaster of Paris. The only vent to the apparatus during the extraction and distillation is through the calcium chloride tube R attached to flask F which is kept closed the entire time after the apparatus has first been charged with ether vapor. By this arrangement no odor of ether can be detected except when the flasks are being changed and for a few moments after the extraction begins. After removing a flask from the extractor another should either be attached immediately or else the lower end of the extractor closed by means of a stopper to prevent unnecessary escape of ether vapor. It is desirable to insert a second flask at once in order that oxidation of the mercury by exposure to the air may be avoided as far as possible. In case the bath E is full of water, the extraction tube may be introduced or removed from the extractor without wetting by sliding it partially into a test-tube.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF LAFAYETTE COLLEGE.]

METHOD FOR PREPARING STRICTLY TENTH-NORMAL, FIFTH-NORMAL, ETC., HYDROCHLORIC OR NITRIC ACID.

BY RICHARD K. MEADE.

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IN a paper before the Lehigh Valley Section of the Society,[1] the writer described a method for preparing strictly normal, semi-normal, decinormal, etc., sulphuric acid by decomposing copper sulphate with the electric current. Since the publication of this

[1] This Journal, **23**, 12.