XXIX.—*Apparatus for "Steam Distillation."*

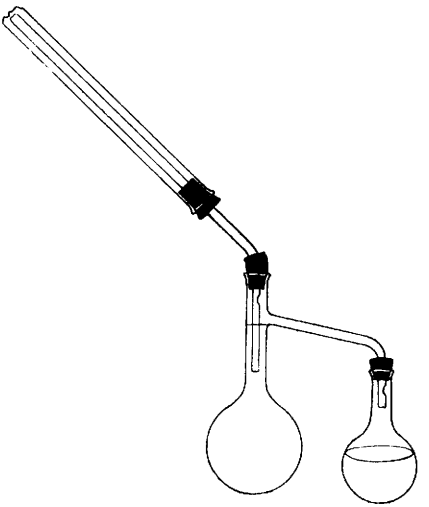
By Francis Edward Matthews.

For some time past, the author has taken advantage of the fact that during the process of steam distillation various solid substances of fairly high melting point are deposited in, and adhere to, the interior of the condenser, so that by substituting a reflux condenser for one in the

ordinary position, the process becomes a continuous one, it being merely necessary from time to time to remove the substance deposited in the condenser. By using condensers of sufficient interior diameter, the removal of this deposit need not be often repeated, and consequently the separation or purification of the substance can be left almost to itself until complete.

The advantages of an automatic method of steam distillation were so obvious, that attempts were made to construct an apparatus which could, in like manner, be used for the separation of liquid substances. In the case of liquids heavier than water, trial was first made of a reflux condenser having near its lower extremity a T-piece connected with a flask, so that the condensed mixture of water and the liquid should

FIG. 1.



flow down the T-piece into the flask, when the heavy liquid would sink to the bottom and the water flow back from the surface into the boiling-flask. It was found that this arrangement did not work at all, from the fact that the liquid was in the form of small drops, which remained floating on the surface of the water, and consequently returned to the boiling-flask along with it.

It was obvious that, in order to succeed, a quantity of the heavy liquid sufficient to produce a large drop must be collected, and then the drop, when it becomes of a certain size, will necessarily sink. This object was attained in the following way. The vessel in which the mixture was boiled was an ordinary flask, in size sufficient to readily contain the liquid to be steam distilled along with two or three times its volume of distilled water, this boiling-flask (Fig. 1) being connected

by a cork to a receiver, which consisted of an ordinary distillation-flask having a wide side-tube. This side-tube was bent downwards, 3 or 4 inches from the end, at an angle of about 120°, so that when the receiver was upright, the upper part of the side-tube was inclined downwards, the lower part being nearly vertical. About 1 inch from the end of the side-tube a hole was made large enough to carry off the steam from the boiling-flask. It was found that this hole allowed the diameter of the side-tube to be considerably reduced, for in a tube in which vapour is passing in one direction and water in the other, the chief point at which a block is usually produced is at the lower end of the tube where the water begins to form drops; by allowing the vapour to enter the tube at the side, higher up, this tendency to choking at the end is obviated to a very great extent, and consequently a much narrower tube can be used.

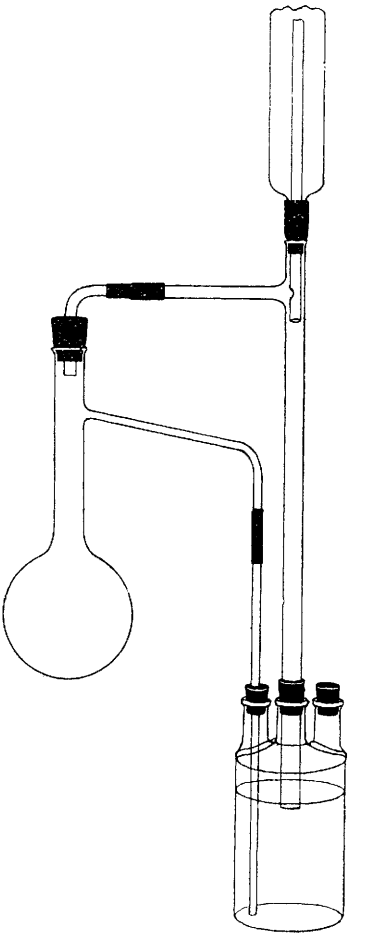
The reflux condenser connected with the receiver was of the following construction. Its inner tube was sufficiently narrow to pass readily into the neck of the receiver, and the portion below the water-jacket had to be of sufficient length (about 9 inches) to admit of a bend being made in it at an angle of about 120°, the portion below the bend penetrating the receiver to a depth of about 2 inches below its side-tube. In this part of the tube, about 3 inches from its end on the side away from the bend and just below the cork, a hole was made to admit the mixed vapours to the condenser.

The apparatus works as follows. The liquid to be distilled is placed in the boiling-flask together with two or three times its volume of water, the flask is connected to the side-tube of the receiver, which is filled with distilled water up to the junction of the side-tube, and the condenser is inserted into the receiver to a depth of about 2 inches below the water-level. On raising the contents of the first flask to the boiling point, a stream of the mixed vapours passes through the side-tube and into the upper part of the neck of the receiver, whence it is forced through the hole into the condenser; here it is condensed, and the liquid runs down the side of the condenser away from the hole until a drop of the heavy liquid sufficiently large to sink is formed in the lower end of the condenser; as the liquid condenses, it displaces an equal volume of water from the receiver, which flows down the side-tube back into the boiling-flask. As the receiver, if protected from direct radiation, remains fairly cool during the whole operation, the volume of liquid in the boiling-flask remains almost constant until the distillation is finished.

For automatically steam-distilling liquids lighter than water, it is obvious that the water must be transferred from the bottom of the receiver back again to the boiling-flask. This object was attained satisfactorily by one or other of the following arrangements, which, although identical in principle, differ in various details.

1. The boiling-flask (Fig. 2) is an ordinary distilling-flask of suitable size, having the side-tube bent vertically downwards 2 or 3 inches from the end when the flask is upright. The receiver is a Woulfe's bottle, with either two or three necks. Into the neck nearest to the boiling-flask, a straight, upright tube is fixed by means of a well-fitting cork, of such a length that, one end being at the bottom of the Woulfe's bottle, its other just touches the lower end of the bent side-tube of the boiling-flask, to which it is connected by an india-rubber joint. The second neck of the Woulfe's bottle is fitted with a vertical T-tube, which fulfils the following conditions. It must dip 2 or 3 inches into the Woulfe's bottle, and the T-joint must be sufficiently high up to allow of the formation of a column of (light) liquid sufficient to overcome the pressure of a column of water the height of which is the difference in level between the surface of the water in the receiver and the point where the side-tube meets the neck of the boiling-flask. Into the upright T-tube a condenser, of any suitable form, is fixed (a narrow vertical condenser with a hole made in it, as previously described, has been found to work well). The boiling-flask is connected with the side-tube of the vertical T-tube by means of a tube bent at right angles, a cork, and an india-rubber connector. It is essential that all the corks in the Woulfe's bottle should fit tightly.

FIG. 2.



To use the apparatus, distilled water is poured into the receiver till the bottom of the T-tube is just covered; the mixture to be distilled is placed in the boiling-flask and heated; the mixed vapours pass into the T-tube, and thence into the condenser; the condensed liquid soon forms a column in the upright T-tube, which gradually forces the water up the other upright tube back into the boiling-flask.

The receiver should be screened from direct radiation as much as possible, although the expulsion of a small amount of air from the upper part of the Woulfe's bottle is unimportant. The chief objection to this form of apparatus is the india-rubber connection between the boiling-flask and the T-tube, as this is liable to attack by the vapours of some liquids, whilst if no joint is made, the strain produced in fitting the apparatus together is dangerous. The second india-rubber

FIG. 3.

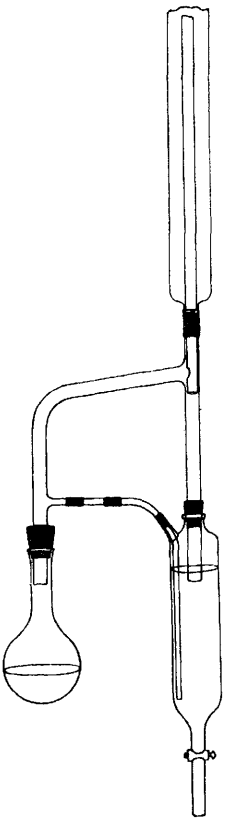
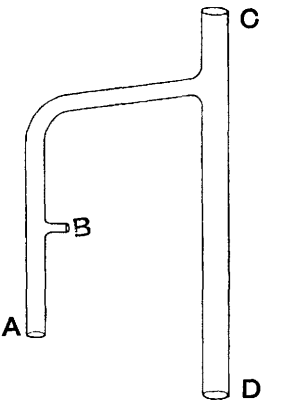


FIG. 4.



joint does not matter, as only cool water passes through it. Apart from this, the apparatus works perfectly; and it has the advantage that it can be constructed in a very short time from apparatus found in any ordinary laboratory.

II. In the apparatus Fig. 3, the Woulfe's bottle is replaced by a separating-funnel with two necks; this has the advantage that the distillate can be drawn off by the tap at the bottom without dismantling

the apparatus, whilst the boiling-flask is an ordinary flask, instead of the distilling-flask used in I. The connection between the boiling-flask and the receiver and condenser is made by means of a piece of apparatus with two T-tubes, as is shown in Fig. 4. The boiling-flask is connected at A by means of a cork, B corresponds to the side-tube of the distilling-flask in the previous arrangement, and is connected, best by a double india-rubber joint, to the tube passing to the bottom of the receiver; the condenser is connected at C, and the end, D, passes 2 or 3 inches into the receiver. By this means, the exposure of india-rubber to hot vapour is avoided. The apparatus works just in the same manner as the previous one, and a detailed description is unnecessary.

Liquids heavier than water may also be distilled in this form of apparatus, the tube through which the water returns being shortened, so as to allow of the water being drawn off near the surface.

From the relatively small amount of liquid necessary for these processes, "steam distillations" can readily be carried on in the vapour of higher boiling liquids, and I am in hope that some new separations may be made by these means. I have found that, in many cases, the rapidity of the distillation can be greatly increased by dissolving some substance, such as sulphuric acid or calcium chloride, in the water of the boiling-flask. Many organic liquids bump very badly when boiled with water, and to obviate this, a couple, made by soldering a piece of zinc foil to a piece of platinum foil, may be used with advantage, wherever possible; thus aniline, which bumps furiously when boiled with water, can be readily distilled without any bumping when the couple is added. In conclusion, I wish to express my thanks to Professor McLeod for the kindly interest he has taken in this work, and for some important suggestions he has made.

ROYAL INDIAN ENGINEERING COLLEGE,
COOPER'S HILL, STAINES.