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XLVI. *On an Apparatus for the Distillation of Mercury in a Vacuum.* By WYNDHAM R. DUNSTAN, M.A., F.C.S., Professor of Chemistry to the Pharmaceutical Society of Great Britain, and T. S. DYMOND, F.I.C., F.C.S.*

[From the Research Laboratory of the Pharmaceutical Society.]

DISTILLATION of impure mercury constitutes the best method of removing foreign metals, and distillation in a vacuum is the only feasible plan of conducting the operation in the laboratory. It is doubtful whether mercury can be completely freed from zinc by distillation, and it is therefore safer to remove this metal, before distillation is resorted to, by agitating the mercury with warm concentrated hydrochloric acid.

Different forms of apparatus for distilling mercury in a vacuum have been described by Weinhold, Weber, Wright, and Clark. The action of these different pieces of apparatus is primarily dependent on the same principle; but that described by Clark (Phil. Mag. 1884, xvii. p. 24) is undoubtedly the most convenient and is the form which is ordinarily used. It possesses, however, several disadvantages. It is inconveniently long, being at least 5 feet in length, and needs a special place in the laboratory where it must be permanently fixed. Moreover the apparatus is only applicable when a comparatively large quantity of mercury is available, since at the conclusion of the operation there remains in the apparatus between one half and one quarter of a litre of undistilled mercury, the amount being chiefly dependent on the diameter of the distillation-tube. To this residue more mercury must be added before distillation can be recommenced.

The still we are about to describe does not entail these disadvantages. It is easy and inexpensive to construct, and when once started is automatic in its action. It is rather more than a metre long, and when required for use can immediately be clamped on a retort-stand and may be as readily dismounted again. The residue of undistilled mer-

* Read March 7, 1890.

cury which remains in the apparatus is less than 20 cubic centimetres.

The apparatus is constructed in the following manner:—A bulb A is blown on one end of a piece of thick-walled soft glass tubing B, and is made to assume somewhat the shape of

an oblate spheroid, so that nearly the whole of the mercury may be distilled out of it. The other end of the tube has a small elbow-bend to admit of its connexion with the reservoir H by means of the stout rubber-tubing G. The diameter of the bore of this glass tube is 3 millim., and its length 1 metre,

measured from the point at which it joins the bulb to the bend at the other end. To the summit of the bulb is sealed a piece of tubing which is bent round one side of the bulb and terminates in the limb C, which acts as a Sprengel pump. The length of the tube from the lower surface of the bulb to the bend at its other end is about 1 metre, and the bore has a diameter of 1.5 millim. The upper part of this limb, at a distance of about 5 centim. from the bulb, is expanded to form a chamber D for the condensation of the mercury vapour; this chamber is 1 decim. long and 1 centim. wide. The other end of the limb is for convenience bent twice at right angles F, and beneath it is placed a receptacle for the distilled mercury. The movable reservoir H, attached by means of at least 1 metre of stout rubber-tubing, known as "pressure" tubing, to E is a large tap-funnel having a capacity at least twice as great as that of the bulb A. It is provided with a tightly-fitting indiarubber cork carrying a small tapped tube K, which projects just below the cork, and a thistle-funnel L which extends to the bottom of the reservoir. The apparatus is supported by a clamp immediately below the chamber D, and it rests on the bend E of the stout limb B.

Before the still is set in action it must be thoroughly dried. The reservoir H is then filled with dry mercury and raised until the liquid has ascended to the summit of the bulb A, and has commenced to fall over into the limb C. The reservoir is now slightly lowered, and by means of the tap J the flow of mercury is checked until it falls only moderately fast (as in a Sprengel pump) down the limb C. In about five minutes the chamber D will have been exhausted of air. As soon as the sharp "click" of the falling mercury is heard and the descending column in C is almost continuous, the reservoir is lowered until the bulb A is about half full. The reservoir H is now adjusted to act automatically in maintaining a constant level of mercury in the bulb A. The stopcock J having been shut, the bulb is heated and the distillation allowed to proceed for about half an hour until the vapour-pressure of the mercury for the maximum temperature to which the bulb will be raised has been attained. Air is then sucked out of the tube K until bubbles pass up through the mercury, when the tap in K is instantly closed. The tube of the funnel L now con-

tains no mercury, the pressure of the air and of the mercury in the reservoir being exactly equal to the atmospheric pressure acting through L. The level of the mercury in the reservoir must now be adjusted until the surface of the mercury in the bulb is distant from the bottom of the reservoir (above J) by the height of the barometric column less the vapour-pressure of mercury for the temperature at which distillation occurs. This temperature having been ascertained, the adjustment may be made after direct measurement of the mercurial column. It may also be effected perhaps more readily by raising the reservoir little by little until on momentarily opening the stopcock J air is observed to bubble through the mercury. As soon as this occurs, the stopcock J is left open. The mercury from the reservoir H will now be automatically supplied to the bulb A. As the mercury distils out of the bulb more rises from the reservoir to take its place, and air enters from the funnel L and bubbles through the mercury in the reservoir into the closed space above. Through this action of the reservoir the level of the mercury in the bulb will remain the same until the whole of the mercury contained in the reservoir has been distilled.

In order to heat the bulb A the tubes just below it are surrounded by the ring-burner M, which may be a circular piece of metal or glass tubing perforated at regular intervals with small holes; the flames proceeding from these perforations should be very small, and must on no account be allowed to impinge on the bulb or on either of the tubes. The bulb is now enclosed on all sides so that a hot air-chamber may result. This is best done by cutting a circular disk N, whose circumference is somewhat greater than that of the ring-burner, out of asbestos millboard, incisions being made to correspond with the tubes so that the disk may slide under the bulb and rest on the burner itself. On this disk as a base a cylinder of the same material, or of glass, is placed, the cylinder being wider and taller than the bulb. The cylinder is closed at the top by a rather larger and circular piece of asbestos millboard, in the centre of which a small round hole should be cut. By partially or entirely covering this hole with a small disk of millboard, it is easy to regulate the admission of air to the chamber. The cylinder

is prepared from the millboard by thoroughly wetting it and rolling it round a bottle or wooden block of the proper size. When dry the cylinder is removed and secured by twisting a piece of thin copper wire round it. The bulb is thus heated in the air-chamber, and not by direct contact with the flames, and the bulb is effectually protected from the disastrous effects of draughts of cold air.

The temperature of the air-bath when distillation is proceeding ranges between 200° and 300° C. Since fresh mercury is automatically supplied to the bulb the still requires no attention, and may safely be left at work day and night provided that the gas pressure is almost constant. This may of course be ensured by fixing a gas-regulator in the air-bath. We have found, however, that an ordinary Sugg's dry governor introduced between the gas supply and the burner answers the purpose sufficiently well.

When the still is started for the first time the air-film on the glass is detached and passes away down the limb C. The distillation consists in continuous evaporation from the liquid surface unaccompanied by actual ebullition, or by the "bumping" and spirting which are so noticeable when mercury is distilled under the ordinary atmospheric pressure.

The rate of distillation is mainly dependent on the capacity of the bulb A. We have generally worked with a bulb rather smaller than that described above, because it is easier to blow it of sufficient thickness. As the still is automatic in its action and needs no attention the quick distillation of a large quantity of mercury is not a point of much importance. It has frequently happened that the distillation has come to an end during the night, both the reservoir and the bulb being discovered nearly if not quite empty. This, however, has occasioned no inconvenience; it is only necessary to introduce more mercury into the reservoir and from thence into the bulb. This can be done without extinguishing the burner. The stopcock J is closed and the tap K is opened. Mercury is then poured through the funnel L. The stopcock F is now opened very gradually, so that cold mercury may not suddenly rush against the hot glass, until the bulb is half full, when the stopcock J is closed again. A piece of rubber-tubing is now attached

to K, and air is sucked out until bubbles begin to rise through the mercury in the reservoir, when K is shut ; after the lapse of about half an hour the stopcock J is opened. When a distillation has been finished and the apparatus is not further required for the present, it may be left standing with the stopcock J closed, and the end F dipping into mercury or otherwise sealed ; it can then be started again at any moment. To empty the still, F is closed and the apparatus is carefully tilted so that the mercury remaining in C may flow into the chamber D. If now the end F be cautiously unclosed air will enter the apparatus through the empty limb C.

When this apparatus is at work in a dark room the remarkable phenomenon of the "flashing" of mercury is observed. It is seen in the condensation-chamber D. As the mercury liquefies flashes of green light are produced, which exhibit the spectrum of mercury. It would seem that this is an electrical effect arising from friction between mercury and glass. We have noticed that it is enormously intensified when one of the dischargers of a Wimshurst machine is brought near the chamber D. Under these circumstances the entire chamber becomes illuminated with a magnificent green light.

The apparatus may also be conveniently employed for observing the electrical discharge through mercury-vapour at different temperatures. For this purpose the ascending column of mercury in B and the descending column of mercury in C are used as electrodes. One terminal from the coil dips into the mercury in the reservoir H, the other into the distilled mercury which has collected in the receptacle under F; and below the surface of which the extremity of the tube C is immersed.

We are now making observations on the character of the discharge which occurs under these conditions, in the absence of any foreign metal.

Prof. Thompson said he devised a simple form of distilling-apparatus some time ago which answered fairly well, and could be made by any amateur glass-worker. It consisted of a double barometer, one leg of which was of small bore, so as to act as a Sprengel-tube. The rising part of the bend at the

top of the larger tube was expanded, and served as the evaporating-chamber, below which a burner was placed.

The President asked why Clark's apparatus is made so lengthy.

In reply to this question Mr. Boys said that, as the fall-tube goes down within the rising one, the mercury near the top of the latter is heated by the condensing mercury (thus economising gas), and hence condensation does not take place until the vapour has passed a considerable distance down the fall-tube.

XLVII. *The Theory of Osmotic Pressure and its bearing on the Nature of Solutions.* By SPENCER UMFREVILLE PICKERING, M.A.*

No one can doubt the mathematical correctness of the conclusions which Arrhenius, van't Hoff, Ostwald, and others draw from the premisses with which they start in their arguments respecting osmotic pressure, nor can we doubt the value of connecting numerous actions with one and the same cause, or that there are a large number of instances in which the observed facts are in substantial agreement with their conclusions. But we may, I think, legitimately doubt whether the premisses of the arguments are sound, whether the conclusions harmonize as well as they should with experimental data, whether the theory is more than a mathematical exercise, or more than a convenient working hypothesis of a rough character, instead of being, as its supporters maintain, an hypothesis established so firmly that we may build upon it a physical theory of solution.

The direct measurement of osmotic pressure has been made in but a few cases, and those cases are ones in which the substances examined are eminently unfitted for showing the presence of any chemical action which may be present. Of the phenomena correlated with osmotic pressure, the lowering of the freezing-point of a solvent by the addition of foreign substances is the one which has received the greatest attention, thanks to Raoult's classical work, and is the one which forms the main support of the theory.

* Read March 7, 1890.