

## On the Purification of Mercury by Distillation *in vacuo*

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XXXVII. *On the Purification of Mercury by Distillation in vacuo.* By J. W. CLARK, *Demonstrator of Physics in University College, Liverpool.*

[Plate XIV.]

THE usual processes for the purification of considerable quantities of mercury may be roughly classed as (i.) chemical (*e. g.* treatment with dilute  $\text{NO}_2(\text{HO})$ ,  $\text{CrO}_2(\text{HO})_2$ ,  $\text{Fe}_2\text{Cl}_6$ ,  $\text{SO}_2(\text{HO})_2$ , and  $\text{SO}_2(\text{HgO}_2)$ , &c.); (ii.) mechanical (*e. g.* shaking, filtering through wash-leather, &c.); and (iii.) distillation. The last-named process may be conducted either *in vacuo* or under the ordinary pressure.

Of these processes distillation *in vacuo* is in all respects the simplest and most satisfactory. Preparatory to distillation the mercury may be advantageously filtered through a writing-paper cone with a very small orifice at the apex; and when considerable quantities of lead or zinc are present, the distillation *in vacuo* may be hastened by their previous removal by one of the usual chemical methods. It is stated that the presence of  $\frac{1}{10000}$  part of lead reduces the amount of mercury distilled in a given time from 67 to 55\*. Gold, iridium, silver, copper, tin, nickel, cadmium, and arsenic do not influence the rate of distillation†.

The distillation of mercury under the ordinary pressure is too inconvenient a process to be ordinarily used in laboratories; not so, however, at a temperature of  $180^\circ$ – $200^\circ$  C. *in vacuo*. The first apparatus for this purpose was described by Weinhold‡, and since then Weber§ and A. W. Wright|| have described other forms. The form shown in section in fig. 1 (Plate XIV.) differs from all the preceding chiefly in being supplied with the mercury to be distilled from a movable reservoir (in the form of a *constant level regulator*, fig. 2), the raising of which fills the distiller with mercury, which thus renders a Sprengel-pump unnecessary to set it in action. It is hoped moreover that its simplicity and efficiency and the ease with which it can be made may render its description useful.

\* Gmelin-Kraut, *Hdb. der Chemie*, Bd. iii. Abth. i. S. 740, 6te Aufl.; Millon, *Ann. Chim. Phys.* [3] xviii. p. 337.

† Gmelin-Kraut, *loc. cit.*

‡ *Progr. d. k. Gewerbsch. zu Chemnitz. Rep. für Physik*, Bd. xv. S. 1.

§ *Ibid.* Bd. xv. S. 52.

|| *Chem. News*, 1881, p. 311.

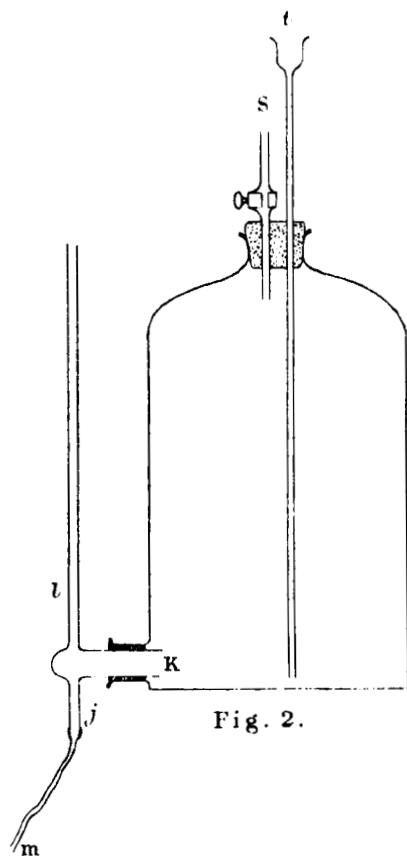


Fig. 2.

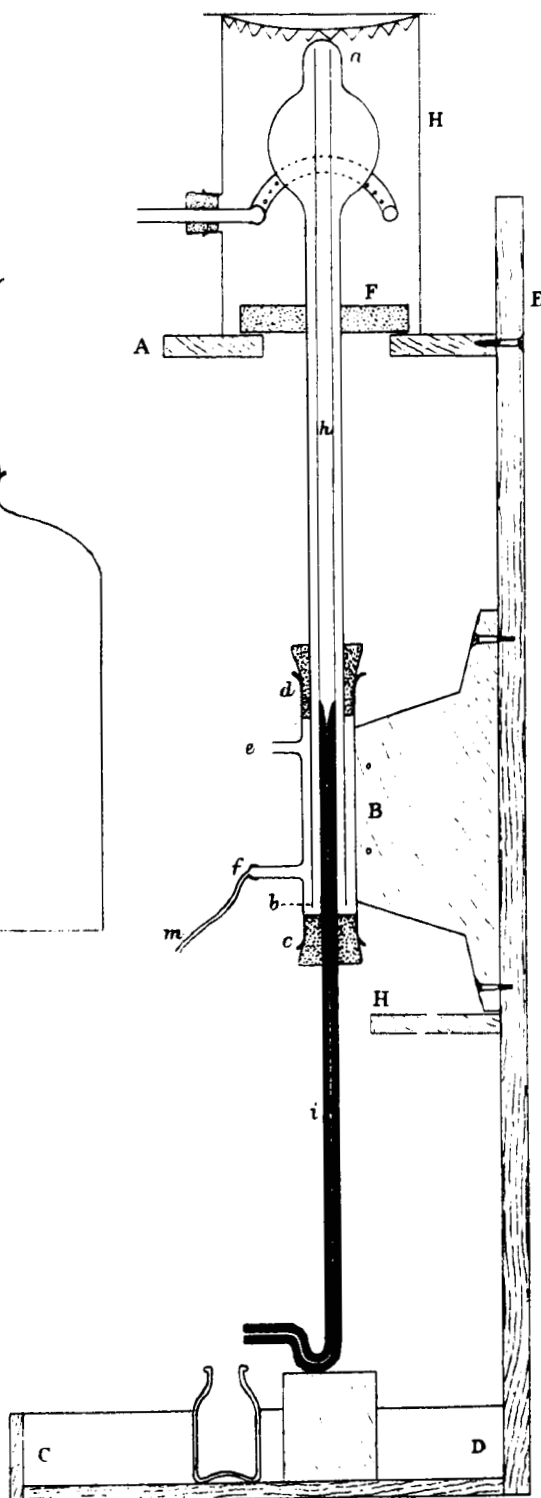


Fig. 1.

The distiller consists of a lead-glass tube, *ab* (fig. 1), 36 inches long, and about  $\frac{3}{8}$  of an inch in internal diameter. A bulb of about two inches diameter is blown two inches from its closed upper end. The lower end passes air-tight through a well-secured india-rubber cork which closes the top of the cistern *dc*, and terminates at *b* a little below the tube *f*. The cistern (*dc*) is made from a piece of glass tube 1 inch in diameter and from 8 to 12 inches long, with two short pieces of quill-tubing, *e* and *f*, sealed into it. The lower end is also securely closed with a cork through which passes a piece of ordinary Sprengel-tube, *i*, 36 inches long, with a piece of quill-tubing, *h*, about 24 inches in length, fused onto the upper end. The top of this tube is nearly in contact with *a*. The internal diameter of the Sprengel-tube should not much exceed 1 millim., and the bend at its lower end is best when not much more than one inch in radius. Instead of india-rubber corks, ordinary corks soaked in melted paraffin or covered with sealing-wax may be used, but the apparatus then loses in flexibility.

The base of the stand consists of a wooden tray, *CD*, from which rises a stout board, *DE*, carrying a shelf, *AE*, perforated in the centre with a hole of sufficient size to allow the glass bulb to pass through it. In the Physical Laboratory of University College the board *DE* which carries the distiller is fixed to the wall over the mercury table. This renders the tray *CD* unnecessary. A large cork, *F*, is bored with a hole of rather less diameter than the tube *ab*, and the cork is cut in halves. By placing the tube in the position shown in the figure and twisting a piece of copper wire round the periphery of the halves of the cork, the tube is firmly supported on the shelf. The cistern is secured by string which passes through holes in the projecting piece of wood, *B*. A block of wood may be placed as a support for the end of the tube *i*. A tin cylinder, slightly notched round the top and covered with a flat tin plate, keeps the bulb surrounded with hot air, whilst a mica window at the side allows the height of the mercury in the bulb to be easily seen. The pipe of the brass ring-burner passes through a hole in the tin case. The diameter of the ring is about half an inch greater than that of the glass bulb, and on the inner side it has a *large number of very small holes*.

The constant-level reservoir (fig. 2) is made from a large bottle provided with a tubulure at the side. Into this passes (through a cork if the tubulure be sufficiently wide—if not, cemented in with sealing-wax) a glass tube, *K*, about 3 inches in length and  $\frac{1}{2}$  inch in diameter. Its outer end is closed, and into the upper and under sides are sealed two pieces of quill-tubing, *l* and *j*. The top of the upper one is open, but the lower (*j*) is connected with the cistern of the distiller by a narrow piece of india-rubber tubing, *mm*, about  $3\frac{1}{2}$  feet long, enclosed in a canvas tube. By means of an india-rubber (or paraffined) cork the thistle-funnel and small glass stopcock are fitted *air-tight* into the neck of the bottle\*. Thus fitted, the reservoir is placed on an ordinary adjustable table-stand on the shelf *H* (fig. 1). To set the distiller in action, open the stopcock, *S*, of the reservoir and pour some of the mercury to be distilled through the thistle-funnel, *t*, into the reservoir, and with a short piece of india-rubber tubing and glass rod *securely* close the tube *e* (fig. 1) at the top of the cistern. Then raise the reservoir. The mercury gradually rises in the cistern, and by compressing the air in the upper part is forced up the tube *ab*, and then filling the bulb sprengels down the tube *hi*. The reservoir may then be lowered on to its stand on *H* and the india-rubber stopper removed from the tube *e*. The reservoir is set in action by attaching a piece of india-rubber tube to the stopcock *S* and sucking out air until, passing down the tube *l*, it bubbles up through the mercury in the reservoir. Then close the stopcock, and adjust the reservoir at such a height on its stand that the mercury is nearly at the top† of the bulb in the distiller. If needful, a little more air is sucked out of the reservoir, as before described. Thus set in action, the level of the mercury in the cistern *cd* will be retained constant until almost the

\* So perfectly does this form of constant-level cistern work, that it seems probable that it may prove useful for other purposes—*e. g.* keeping a Sprengel-pump in uniform action for many consecutive hours &c.

† The vapour-tension of the hot mercury will depress the level in the bulb. The extent of this depression is somewhat dependent upon the height of the gas. On this account a simple form of gas-pressure regulator may be advantageously used with this apparatus. In another connection I hope to describe a pressure regulator of very convenient construction for this and other purposes.

whole of the contents of the regulating reservoir have been distilled.

To start the distillation, remove the tin plate which covers the cylinder (H) and light the gas. Five to ten minutes later, sufficient mercury will have distilled over to have entirely displaced the impure mercury originally present in the narrow Sprengel-tube *i*.

The reservoir can be replenished with mercury without interrupting the distillation. For this purpose it is only necessary to place a screw pinchcock on the india-rubber tube leading to the cistern of the distiller, open the stopcock S, and pour the mercury into the reservoir through the funnel *t*. Then suck a few bubbles of air out of the reservoir, as before described, close the stopcock, and release the screw-clamp from the india-rubber tube. The level of the mercury in the distiller will remain as before.

When it is desired to empty the distiller of mercury, air must be introduced into the bulb either by alternately sucking and blowing through a piece of india-rubber tubing connected with the end of *i*, or by disconnecting the india-rubber tube leading from the reservoir, emptying the cistern *cd*, and cautiously inclining the distiller until small bubbles of air enter at the end of the tube *ab* and rise into the bulb. The mercury then sinks into the cistern, and may be withdrawn through the tube *f*.

The Sprengel-tube *i* should be carefully cleaned and dried before putting the apparatus together.

The first time of using the distiller, it is interesting to observe that, as the tube *ab* gradually becomes heated, the surface air-film detaches itself from the glass and rises into the bulb.

The quantity of mercury distilled by an apparatus of the size and form here described is about 2 lb. an hour: very little gas is used, as the flames should be less than a quarter of an inch high and never allowed to come in contact with the glass bulb. For commercial purposes, an iron mercury-bottle and iron gas-pipe might advantageously replace the glass bulb and tube.

It seems to be possible that the latter modification of the method may be applicable to the distillation of some other metals, such as zinc, magnesium, cadmium, arsenic, &c.