THE PREPARATION OF VERY CLEAN MERCURY

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ANY PROCESS which proves adequate for the purifying of a metal used as the active substance in a photoelectric cell can also be employed with considerable confidence in other fields of experimentation where extraordinary purity is desired. This follows from the fact that the highest obtainable degree of purity is the most essential requirement in the investigation of the photoelectric properties of a substance.

In preparing mercury for photoelectric purposes the mercury not only must be freed from foreign metals and other substances, but it must be protected during the process of distillation from contamination. Especially must contamination with organic substances be avoided. Stopcock grease, which is a mixture of rubber and vaseline, and certain other organic substances produce pronounced changes in the photoelectric behavior of mercury, as has been shown by W. B. Hales and F. L. Poole in unpublished work, and by the author (Phys. Rev. 32, 323, 1928). The removal of occluded gases from the mercury is also important, for it is well known that absorbed and adsorbed gases often produce marked changes in photoelectric properties.

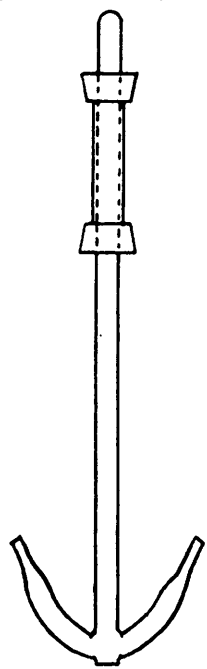

Fig. 1. Centrifugal mixer.

The following technique for cleaning mercury for photoelectric purposes has been used by the author with success:

(1) Remove grease by washing the mercury thoroughly with a 10 percent solution of potassium hydroxide.

(2) Shake or mix the mercury for several hours with an equal volume of 3-normal nitric acid, thus removing most of the metallic contamination.

(3) Wash the mercury with distilled water, dry by heating it to about 110°C. in a porcelain dish, and filter by forcing it with compressed air through chamois skin, or by passing it through a pinhole in the apex of a glazed paper funnel.

(4) Distill the mercury several times by the method of Hulett and Minchin (Phys. Rev. 21, 388, 1905); in this method, air is bubbled through the mercury in the still during distillation, thus oxidizing volatile foreign metals.

(5) Pass the mercury through a filter consisting of a glass tube with one end drawn to a capillary opening; repeat this process until all traces of oxide have disappeared from the surface of the mercury.

(6) If it is desired to remove occluded gases, as is necessary in photoelectric work, distill the mercury repeatedly in the highest obtainable vacuum. The still used in this process should form an integral part of the apparatus in which the mercury finally is to be used. This method of re-

moving occluded gases from mercury was first used by Kazda (Phys. Rev. 26, 643, 1925).

The washing processes described in steps (1), (2), and (3), above, are best accomplished with a centrifugal mixer (Fig. 1). This device can be made easily and it is more effective than the "fall" tube commonly employed in cleaning mercury, or than the ordinary mechanical stirrer; Bray (Am. Chem. Soc. J. 49, 2572, 1927) used a centrifugal mixer in preparing mercury, but does not describe it. This mixer is much more efficient than the ordinary stirrer and undoubtedly can be put to many uses besides the present one, particularly in the laboratory of the organic chemist. The vertical shaft of the mixer consists of a 30 cm length of 7 mm glass tubing. The upper end of the shaft is sealed shut and to the lower end is sealed a 10 cm length of similar tubing, shaped as in Fig. 1, and drawn out at each end to an opening approximately 2 mm in diameter. This tube contains a third opening in its lower center, opposite the point where it is sealed to the shaft. The upper end of the shaft is fitted with a piece of short glass tubing of slightly larger diameter than the shaft, this being held in place by two perforated corks which fit the shaft tightly. It is necessary to lubricate the part of the shaft between the two corks with a small amount of vaseline or graphite.

A clamp is attached to the larger tube, between the corks, and the mixer is supported vertically in a battery jar containing the mercury and washing liquid. The liquid should cover the openings in the ends of the mixer and the shaft should be so adjusted that air and mercury alternately enter the center opening during rotation. It is essential that the mixer be operated at high speed, preferably by means of a small motor attached to the shaft with rubber tubing or with a belt.

The still used in step (4) of the mercury cleaning process should be constructed in the manner described by Hulett and Minchin, except that all joints made with rubber tubing or stoppers should be replaced by ground glass joints. Experience also shows that a receiving bottle for the distillate, connected to the condensing tube by a ground glass joint, is much more convenient and also less fragile than the usual tube of barometric height.

Since phosphorus pentoxide is often used as a drying agent in apparatus containing mercury, it might be well to say here that the phosphorus and lower oxides, often present in impure phosphorus pentoxide, are likely to soil mercury. Phosphorus pentoxide can be purified sufficiently for this purpose by heating it thoroughly in a current of dry air.