mainly for awards to foreign scholars. With funds contributed to the American Council of Learned Societies there have been provided 40 fellowships in the humanities. Seventeen of these were granted during 1932. All the awards were post-doctoral fellowships intended to provide opportunity for further training and experience in humanistic research. In addition a grant was made to the American School of Classical Studies at Athens, for fellowships in archeology, in connection with the excavation of the Athenian Agora.

SCIENTIFIC APPARATUS AND LABORATORY METHODS

THE PURIFICATION OF MERCURY BY AN ELECTROLYTIC METHOD

IN addition to the many uses to which mercury is put in chemical and physical laboratories, it is becoming increasingly important in biological work as a confining and transferring agent for aqueous solutions and gas mixtures. After exposure to rubber tubing, stopcock grease and solutions of organic matter like blood, mercury acquires impurities, so that when it is shaken with water, large quantities of dirt, consisting of organic matter and mercury compounds, are liberated. Since large quantities of mercury are often needed at one time, a rapid and convenient method for cleaning it is desirable. After extensive experimentation with a variety of procedures, we have found the following method to be most satisfactory in combining effectiveness and convenience. The apparatus is inexpensive and easily prepared and the purification of 500 cc of mercury can be completed in one and one half hours.

In general the procedure is to make the mercury the positive pole of a 110 volt direct current, first in the presence of 10 per cent. H_2SO_4, then in the presence of 5 per cent. NaCl, and finally to make the mercury negative in the presence of 10 per cent. H_2SO_4, and to remove by filtration the scum which forms on the mercury during each of these steps.

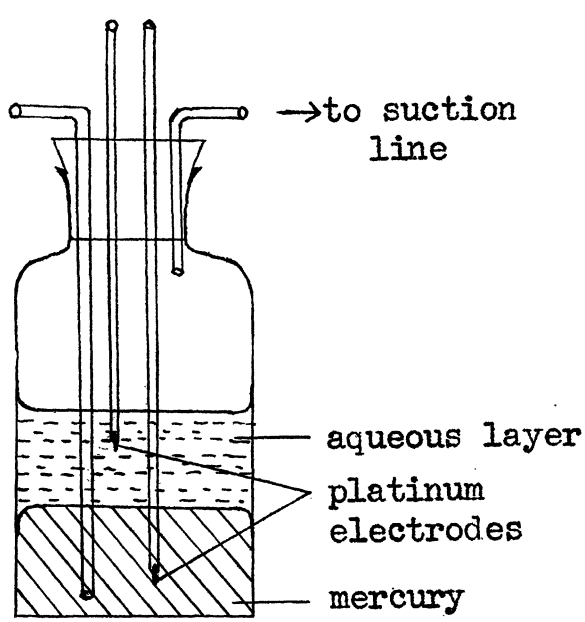

FIG. 1

APPARATUS

The apparatus consists of a wide-mouth bottle with capacity about four times the volume of the mercury to be purified, fitted with a stopper containing two glass tubes with platinum electrodes sealed in the ends, and containing also inlet and outlet tubes for air, the former reaching the bottom of the bottle, the latter ending close under the stopper. Platinum wires 0.5 mm diameter and protruding about 10 mm from the end of the glass serve well as electrodes. An ordinary water suction pump causes a stream of air to pass through the bottle, which not only agitates the mercury vigorously but also prevents the accumulation of explosive mixture of hydrogen and oxygen arising from the electrolysis. The electrodes are arranged so that they can be connected to the 110 volt direct current outlets with sufficient resistance in series, by means of a hot plate or lamps, to give a current of about 3 amperes.

PROCEDURE

One volume of mercury[1] is placed in the bottle with an equal volume of 10 per cent. H_2SO_4. The stopper with electrodes and glass tubes is inserted and air is bubbled through the mercury. The circuit is completed in such a way that the mercury is the positive pole and the electrode in the H_2SO_4 layer is then the negative pole. The electrolysis is carried on for fifteen minutes, during which time a gray precipitate forms in the acid layer. The circuit is broken and then the suction line is disconnected.

The mercury is now washed with alkali in the following manner. Tap water is run into the bottle to remove most of the acid, after which the mercury is poured into a bottle just large enough to contain it so that the water and acid are almost completely removed. The mercury is then poured into a larger bottle containing about one fifth volume N NaOH, and the aqueous phase becomes dark. The mercury and NaOH are poured back and forth between two

[1] We have found it impractical to purify more than 500 cc of mercury at one time because the great weight makes manipulation inconvenient.

bottles four or five times in order to increase the surface contact of the mercury and alkali, after which the alkali is removed by pouring the mercury into a bottle just large enough to contain it. The mercury is washed with distilled water by pouring it back and forth between two bottles, using a volume of water about equal to the volume of mercury. The water is removed by decantation and any water remaining is sponged off with a towel. The mercury has a green to brown scum on it which is removed by filtering through a piece of filter paper which has been perforated at the tip of the cone with a needle.

The mercury is again put into the electrolysis bottle and covered with an equal volume of 5 per cent. NaCl solution and a second electrolysis is carried out with the mercury the positive pole. A wrinkled bluish film forms on the surface of the mercury, a dirty green precipitate appears in the aqueous layer, and the solution becomes alkaline. This electrolysis is carried on until a red-brown precipitate appears, which requires five to twenty minutes, depending upon the amount of impurities in the mercury. The precipitate may adhere to the walls of the bottle or may be suspended in the solution. The NaCl solution is removed with tap water, the mercury is washed with N NaOH, with distilled water, and then filtered as described after the previous electrolysis.

The mercury is again placed in the electrolysis bottle and an equal volume of 10 per cent. H_2SO_4 is added. The electrolysis is carried on for fifteen minutes, but this time the mercury is made the negative pole and the electrode in the sulfuric acid layer the positive pole. During this electrolysis the surface of the mercury appears clean and, except for gas bubbles, the aqueous layer remains clear and colorless. After this electrolysis is completed, the H_2SO_4 is removed and the mercury is washed with N NaOH, with distilled water, and filtered as before. Then the mercury is washed with about one fifth volume of 10 per cent. HNO_3 and again with distilled water. This washing with HNO_3 not only removes traces of alkali but apparently removes some scum and thus makes the mercury sparkle to an unusual degree. In order to remove the last traces of acid or alkali, we have found it necessary to drop the mercury in a fine stream through a 50 cm column of running distilled water. After the supernatant water has been removed by decantation and sponging with a towel, the last droplets of water are conveniently removed by pouring the mercury through a dry perforated filter paper.

DISCUSSION

This procedure is the result of an extensive study of a variety of procedures for purifying mercury. If any one of the steps is omitted, or if the order of steps is rearranged, the mercury obtained is less pure than that obtained by the method as described. Also more extensive electrolysis, additional treatment with chemical reagents, distillation or aeration does not improve the quality of the mercury beyond that obtained by the method as described. By this method we have obtained mercury which looks clean, has a bright reflecting surface, is free from scum, has a high surface tension and does not adhere to the walls of glass apparatus. It forms only a very slight black precipitate when aerated with neutral water for 24 hours, as is characteristic of the purest reagent mercury.

The following theoretical considerations guided us in developing this method. That organic and metallic impurities in mercury can be removed by oxidation is an old idea, and preliminary experiments where impure mercury was shaken with $FeCl_3$ or $K_2Cr_2O_7$ showed that such reagents are very effective. The method of oxidizing the impurities by making the mercury the positive pole in a direct current electrolysis was then tried, and it was found that this electrolysis method is not only superior in removing impurities but also involves less loss of mercury by oxidation than does the use of solutions of oxidizing agents. The electrolytic method usually involves a loss of 2 to 4 per cent., depending upon how much impurity is present, whereas during aeration with ferric chloride the loss may be 10 to 15 per cent., with less improvement of the mercury. Further study showed that one electrolysis with an acid and one with an alkaline solution is more effective than any other combination, and the presence of the chloride ion during the alkaline electrolysis is especially effective. It was then observed that pure mercury treated by these oxidizing electrolyses would show more black precipitate when agitated with water than similar pure mercury not so treated, and it was felt that this might be due to the presence in the mercury of mercury compounds, perhaps oxides, formed in the positive electrolyses. These should theoretically be removable by a negative electrolysis, which would reduce such mercury compounds to metallic mercury. The observation that a final negative electrolysis definitely improves the quality of the mercury led us to include this step.

The scum removed by decantation and filtration after the first two electrolyses contains considerable mercury. When collected separately and treated with dilute nitric acid it coalesces to form globules of impure mercury. When enough is collected, it may be purified and a large fraction of it converted into pure mercury.

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