## Summary

1. The conductance of tetramethylammonium iodide in phenol at 50° has been measured at concentrations from $1 \times 10^{-4}$ $N$ to 0.07 $N$. The constants of the conductance function found for this electrolyte are $\Lambda_0 = 16.67$, $D = 0.70$, $m = 1.28$, and $K = 2.25 \times 10^{-4}$.

2. The conductance of sodium iodide solutions in phenol at the same temperature has been measured at concentrations from $1 \times 10^{-4}$ to $1.6 \times 10^{-3}$ $N$. The ionization of sodium iodide is much lower than that of tetramethylammonium iodide.

3. The results are briefly discussed in relation to the freezing-point determinations of Hartung.

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# AN IMPROVED STILL FOR PRODUCING PURE WATER

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**Introduction.**—One of the difficulties encountered in the study of the properties of dil. aqueous solutions is due to the lack of a convenient method for the preparation of pure water in larger quantities. While water having a specific conductance approaching that of pure water, $0.042 \times 10^{-6}$ at 18°, has previously been obtained by several investigators, a convenient method for obtaining such water in larger quantities and without undue preliminary preparation appears not to have been developed. The still described below has been found to meet general laboratory requirements admirably.

A detailed discussion of previous methods is omitted for the sake of economy of space.[1] It may be noted, however, that the only methods which are practicable from a laboratory standpoint are those of Bourdillon and Weiland, who removed the chief impurity, which is carbon dioxide, by means of a stream of pure air passing through the condensing system during the process of distillation.

In the method of preparation described below, the use of pure air in the course of the distillation process has been eliminated. At the same time, the still has been so designed that it can be constructed by an ordinary tinsmith. Since the limit of the purity of the water obtained by the ordinary methods of distillation appears not to have been investigated systematically heretofore, a brief description may be given of some pre-

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[1] Kohlrausch, *Ann. Physik*, **44**, 577 (1891). Kohlrausch and Holborn, "Leitvermögen der Elektrolyte," Teubner, Leipzig, **1898**, pp. 111–115. Kohlrausch and Heydweiller, *Ann. Physik*, **53**, 209 (1894). Bourdillon, *J. Chem. Soc.*, **103**, 791 (1913). Weiland, This Journal, **40**, 131 (1918).

liminary experiments in which water of a high degree of purity was obtained by such methods.

**Preliminary Experiments.**—In the earliest experiments, a copper still of conventional form, having a capacity of 20 liters, was employed. This still was supplied with a special still-head about 30 cm. long and 15 cm. in diameter, at the bottom of which a baffle was located by means of which the steam was forced to pass through water before passing into the condensing system. Before entering the condenser, the steam was passed through a plug of glass or asbestos wool. The water condensed in the filter was carried off through a water-sealed drip tube. The condenser consisted of about 1.8 meters of block tin tubing, wound in the form of a spiral. The condensed water was collected in a measuring cell which was kept under a slight pressure of pure air.

Various reagents were employed for the purpose of determining which of these were most efficient for the purpose of purifying the water. In the end it was found that an alkaline permanganate solution was most effective, and that the more concentrated the alkaline solution, the better the grade of water obtained. The best water prepared had a specific conductance of $0.12 \times 10^{-6}$ mhos. It should be stated, however, that it was found difficult to obtain water of this degree of purity, consistently. At times, the specific conductance rose considerably above the minimum value, for no apparent reason. Presumably, one of the chief factors influencing the conductance is the amount of carbonate present in the alkali employed. A larger still was finally constructed having a capacity of 90 liters, which, initially, was operated in the same manner as the copper still. In part, this still is shown in the accompanying figure, in which *A* is the boiling chamber, and *B* is a filter provided with a water-sealed drip tube *D* and 2 nickel screens *C*, between which asbestos wool could be placed.

From the filter *B*, the steam passed through the settling chamber *E*, also provided with a drip at the bottom, and thereafter it passed directly into a block-tin condenser of the worm type. The best water obtainable with this still again had a specific conductance of $0.12 \times 10^{-6}$ mhos. During the operation of this still, as well as the preceding one, it was at times observed that, when the still was initially run at a low rate and the rate of distillation was then accelerated, the specific conductance was initially greatly decreased. It was inferred that this was due to the fact that the worm condenser had sagged on one side, trapping a certain amount of water. On increasing the flow of steam, a considerable amount of this water was swept forward and carried over into the cell. It was clear from this observation that the water initially condensed in the spiral had a lower specific conductance than the water finally collected in the cell; and to test the water supplied by the drip pipe *D* a cell was accordingly attached to this pipe. As was expected, it had a lower specific conductance than the water delivered by the condenser. On reflection, it was obvious that if carbon dioxide is the sole or chief impurity to be removed from the water, the removal may be simply effected by the fractional condensation of the steam. At the boiling point of water, the concentration of carbon dioxide in the vapor is about 5 times that in the liquid phase. It follows, therefore, that if a small amount of steam is allowed to escape uncondensed, it will carry with it practically the entire content of carbon dioxide.

**Final Design of Still.**—This led to the design of a still of the type shown in outline in the figure.

From the settling chamber E, the steam passes through a length of block-tin tubing, which is provided with a condenser H, to a separating chamber J. At the bottom, this chamber is provided with a trap through which the condensed water flows into the cell, being cooled on the way by means of the cooler M. The level of the water in J is kept just below the level of the tube I. The excess steam, not condensed by the condenser H, passes through J and is finally condensed by means of a condenser N. All the water condensed by the condenser H is thus separated from the steam in J and collected in the cell. The relative amount of steam condensed is regulated by the level of the water in the condenser H. In practice, it has been found desirable to allow approximately 20 to 25% of the steam to escape through the trap J. It has also been found that the filter B is unnecessary and may be omitted without detriment.

Ordinary distilled water is introduced into the boiling chamber A, and small amounts of sodium hydroxide and permanganate are added. In determining the effectiveness of the still, measuring cells were attached to the tubes L and K passing through the condensers M and N, and the specific conductance of the water in the 2 cells was compared simultaneously.

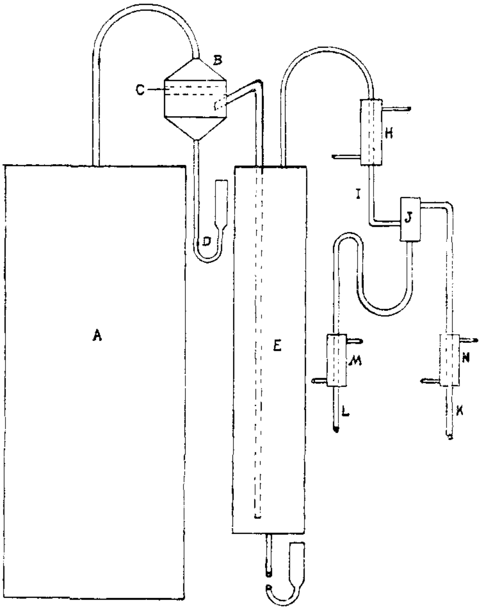

Fig. 1.—Still for producing pure water.

It has been found that, from the start, the specific conductance of the water condensed in H is approximately $^1/_5$ that of the water condensed in the condenser N. When approximately 20% of the water has been distilled, the conductance of the water condensed in H begins to decrease very markedly, while that condensed in N decreases only slowly. When approximately $^1/_4$ of the water has been distilled, the specific conductance of the water condensed in H falls below $0.1 \times 10^{-6}$, and thereafter very rapidly. With a still of this type it is possible to collect approximately $^1/_2$ the water with a specific conductance in the neighborhood of $0.05 \times 10^{-6}$ mhos. The best water collected had a specific conductance of $0.048 \times 10^{-6}$ mhos at 18°.

In practice, the still is filled with ordinary distilled water and maintained at the boiling temperature overnight. Approximately 25% of the water is then distilled, after which the water collected is sufficiently pure for practically all purposes. In order to avoid contamination of the water, when not in use; the still is kept heated very nearly to the boiling point. Under these conditions, water having a specific conductance of $0.05 \times 10^{-6}$ may be collected after operating the still for a short time. The water is collected under a slight excess pressure of air free from carbon dioxide in order to avoid contamination by the atmosphere.

The quality of the water collected is but little influenced by the rate of distillation. As a rule, the still is operated at the rate of 1 liter per hour.

In a still of this type, the steam itself performs the function of the air as employed by Bourdillon and by Weiland. Ordinary copper sheet may be employed for the construction of the still, but tinned copper should be employed for the construction of the settling chamber *E*. The various tubes and the separator *J* are constructed of commercial block tin.

## Summary

(1) Experiments are described in which water having a specific conductance of $0.12 \times 10^{-6}$ is obtained by the direct distillation of ordinary distilled water from an alkaline permanganate solution.

(2) A simple form of still is described by means of which water having a specific conductance of $0.05 \times 10^{-6}$ at 18° may be obtained in quantity. The principle on which the operation of the still depends is that of fractional condensation. About 20% of the steam is allowed to escape uncondensed. This carries with it practically all the carbon dioxide and all other volatile impurities present in the water.

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