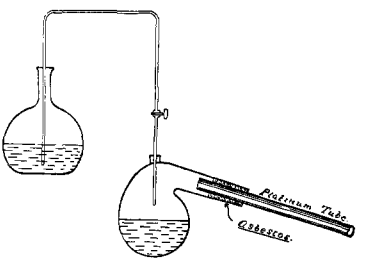
PURIFICATION OF WATER BY DISTILLATION.

BY G. A. HULETT.

Ordinary distilled water suffices for almost all analytical purposes, but for atomic-weight work and many operations in physics water of a higher degree of purity is required. There seems to be little definite information as to apparatus and methods. Stas distilled his water from an alkaline solution of potassium permanganate after rejecting 1/20th, but still found ammonia. After distilling from potassium bisulphate and condensing in a platinum condenser he found it wholly free from organic and mineral matter. Kohlrausch[1] in preparing pure water, used potassium permanganate, potassium hydroxide and potassium bisulphate, in order to get rid of organic matter, volatile acids and ammonia; while Nernst[2] suggests purifying water by recrystallization.

In the present investigation the apparatus used possesses some points of advantage over that ordinarily employed. The condenser is a platinum tube 19 mm. in diameter, and about 60 cm. long. At the lower end the tube is contracted to 5 mm. diameter, and bent so as to pass into the neck of the receiving flask, thus preventing the distillate from coming in contact with the air about the still. This platinum condenser is provided with a short glass cooler, leaving about 20 cm. of the upper end free. About 15 cm. of this free end extends into the neck of a 4-liter retort, and the space between the platinum and glass is packed with asbestos. By this arrangement *only the water which is condensed in the platinum tube is collected*, while the water which condenses in the neck of the retort drips out at the asbestos packing.



[1] Pogg. Ann. Erg. Bd. **8**, 4 (1878.)

[2] Zeit. phys. Chem. **8**, 120 (1891).

When there were more than two liters of the liquid to distill, it was found convenient to siphon water into the retort as fast as distilled. If it was necessary to reject the first portion of the distillate the larger portion, in a glass flask, can be boiled the desired length of time before the siphon is started. With this arrangement it is possible to distill large quantities of water and the apparatus needs no attention for hours at a time.

The most simple and delicate test for the purity of water is its electrical conductivity which can be determined by the method employed by Kohlrausch. The resistance cell was of the single bottle form, and the electrodes were each 25 sq. cm. and were not platinized. They were arranged in the form of concentric cylinders three mm. apart, and held in place by little pieces of glass fused to the lower ends. The resistance capacity of this cell was determined by comparing it with another of known resistsnce capacity, and also by using a 1/10000th normal sodium-chloride solution. Its value was found to be 1148×10^{-10}. Repeated determination showed an experimental error of 0.5 per cent. This cell gave a very sharp telephone minimum when used with the best distilled water.

VALUES OF $K \times 10^{10}$.

I	II	III	IV	V
10.8	3.75	3.01	4.44	1.17
6.78	4.11	1.40	1.67	31.0
4.85	1.58	0.88	0.99	102.0
3.68	1.25	0.76	0.79	3.01
4.03	0.77	0.76	0.80	2.90
3.20	0.96		0.80	3.93
2.93	0.76	0.76	0.79	47.9
			0.76	
		0.72	0.77	6.80
2.20	0.76		0.76	
		0.71	0.76	
	0.76	0.71		22.10
1.63		0.72	0.76	
	0.74	0.72	0.77	
1.46				
		0.76		
1.40	0.87			
1.47		0.87	0.78	

In general two liters of the water to be investigated were put into the retort, and the distillate collected in 100 cc. flasks, so that successive portions of the distillate could be tested. The first water investigated was from a surface well and an analysis by Mr. McLauchlan showed it to be unfit for drinking purposes. Two liters of this water were distilled, and the successive hundred cubic centimeters of the distillate gave the conductivities indicated in column I, of the table.[1] It appeared that after one-fourth had been distilled over, the remainder was quite good water. Following this suggestion and using the continuous distillation, fifteen liters of water were obtained with a conductivity $K \times 10^{10} = 3.3$. This water was then made distinctly purple with potassium permanganate, and after standing two days, was acidulated with 75 cc. sulphuric acid and carefully redistilled. Finally 2000 cc. were treated with 50 cc. of a saturated solution of barium hydroxide, and again distilled. The conductivities for the successive portions of this final distillate are shown in column II.

It is to be observed that the conductivity of the successive portions[2] of the distillate decreases rapidly, and after about a quarter of the water has been distilled, it reaches the value $K \times 10^{10} = 0.77$, where it remains practically constant during the remainder of the distillation.[3] It is to be further observed that the distillation may be continued without affecting the quality of the distillate until only about 100 cc. of the liquid are left in the retort.

A second series of observations was made, starting with the distilled water regularly used in the laboratory. Ten liters of this water, mixed with 50 cc. of a saturated solution of potassium bichromate and 50 cc. of sulphuric acid, were allowed to stand several days and then distilled. (The acid solution of potassium bichromate is a much stronger oxidizing agent than potassium permanganate, and seems to boil with less bumping.) Two liters of the distillate were distilled with 50 cc. of the barium hydroxide solution. The results are shown in column III of the table,[3] and are in close agreement with those in No. II. It thus appears that the methods here

[1] It took from 8 to 9 minutes to distill off each portion.

[2] The time of the distillation was 6 to 8 minutes per portion.

[3] Each portion distilled in about ten minutes.

described yield distilled water which leaves nothing to be desired as far as any impurity affecting the conductivity is concerned. It needs to be added that repeated distillation of this final distillate did not lead to any noticeable reduction of the conductivity below 0.76. Kohlrausch concludes from his research that the best water distilled in the air has a conductivity of 0.70.

Barium hydroxide is a strong base and seems to be an admirable reagent for retaining the carbon dioxide and volatile acids. The results show no indication of barium hydroxide having been mechanically carried over during the distillation. To test this point further, two liters of the best water with 50 cc. of the solution of barium hydroxide were distilled at a slow rate[2] until the distillate showed a conductivity $K \times 10^{10} = 0.76$., and then the rate of distillation was gradually increased until it was 17 cc. per minute. The results are found in column IV. Comparing these results with those in I and II, it apears that *the quality of the water is independent of the rate of distillation* when barium hydroxide is employed to fix the acids. The last 100 cc. gave a conductivity of $K \times 10^{10} = 0.87$, leaving only 55 cc. in the retort! With sulphuric acid, however, the case is altogether different, as appears from the following series of observations: Two liters of water, conductivity $K \times 10^{10} = 0.80$, and 50 cc. of sulphuric acid were slowly distilled. All portions of the distillate gave high conductivities, as shown in column V. A portion of the distillate was concentrated and, when tested with barium chloride, showed sulphuric acid. An inspection of these irregular results suggests that some of the liquid in the retort was mechanically carried over, but as all portions of the distillate show a conductivity much above that of the water employed (0.80) it raises the question whether some sulphuric acid does not *distill* over with the water.

In order to test the quality of water to be had by the method of continuous distillation five liters of the distillate, from potassium bichromate and sulphuric acid, were divided between the retort, and a 3-liter glass flask from which it could be slowly siphoned. After redistilling and rejecting the first 400 cc., during which time the water in the flask was also boiled, the siphon was started and two liters of the distillate collected. The sample showed $K \times 10^{10} = 0.87$

[2] 12–24 minutes for each of the first eight portions.

The next liter gave 0.78. Then successive portions of 100 cc. gave respectively : 0.76, 0.80 and 0.87, leaving but 100 cc. in the retort. It is possible that a tin tube of the same construction would answer for water. According to A. C. McGregory[1], a condenser of good glass answers every purpose.

In conclusion, I wish to express my thanks to Dr. E. H. Loomis for many suggestions and kindly interest in the above work.

Princeton Chemical Laboratory, June, 1896.

[1]Inaugural Dissertation, Strassburg, 1893.