FURTHER NOTE CONCERNING THE EFFICIENCY OF FRACTIONAL DISTILLATION BY HEAT GENERATED ELECTRICALLY.

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In a recent note[1] concerning the use of electrical heating in fractional distillation the convenience of the method was emphasized, especially for distilling *in vacuo*, and a few experiments were described. According to these experiments, which involved the separation of both the lower and higher boiling impurities from certain esters, it was shown both that superheating was much reduced by using this source of heat and that a constant-boiling fraction at the true boiling point was obtained much more readily by the electrical method than by the older method. These facts may easily be verified experimentally by any one who takes interest in the matter.

Further, the inference was drawn from these facts that the separation by electrical heating was more efficient than by the usual method. Others seem to have agreed with this assumption, since no doubt of its validity, either publicly or privately expressed, has reached us during the ten months which have elapsed since the appearance of the paper. Indeed Beckmann[2] has pointed out somewhat eagerly that he had independently discovered the method—his first publication having been made only about a month after ours, which he could not have seen.

Nevertheless it seemed to us desirable to test the conclusion further. Some other test of purity beside the boiling point should be applied, for the mechanism of boiling from a hot platinum wire is not sufficiently understood to make it possible for any one to predict beforehand whether or not the vapor thus produced represents merely an average of the vapors of the various components, or whether it corresponds to the particular component whose boiling point is represented by the temperature of the liquid at the moment.

A wide choice of further experiment is open. The separation of almost any two liquids capable of being separated by fractional distillation would serve the purpose. Accordingly the simplest and most obvious case was chosen, namely the fractional distillation of dilute ethyl alcohol. The experiments were carried out by distilling under otherwise similar conditions separate portions of dilute alcohol, by electrical heating on the one hand and by the usual method on the other; and the densities of the corresponding fractions were compared. Such an experiment presents very different conditions from the mere purification of the liquid from lower boiling and higher boiling impurities. In the present case the

[1] *Proc. Am. Acad.*, **43**, 521 (1908); THIS JOURNAL, **30**, 1282 (1908); *Z. physik. Chem.*, **64**, 120. It is worthy of note that the first of these communications was received by the American Academy on May 18, 1908.

[2] *Z. physik. Chem.*, **64**, 506 (1908).

initial and the final fractions of the extreme boiling points are the fractions to be purified, whereas in the case previously considered the middle fraction was the interesting portion. The need of the performance of such an experiment in order to obtain a complete knowledge of the process is very clear.

The first test was made as follows:

A sample of dilute ethyl alcohol was prepared by mixing absolute alcohol with water in the proportion of 44 per cent. by weight of alcohol to 56 per cent. of water. This mixture had as its specific gravity (at 25° referred to water at 15.6°) the value 0.92345. Two portions of 210 cc. each of this solution were taken: one was placed in the electrical distillation flask, of the type already described, while the other was placed in an ordinary boiling flask. Each was distilled into three portions of 70 cc. each, the distillations being carried on simultaneously.

In order to have the conditions as nearly parallel as possible, the flame and the current were respectively regulated so as to keep the rate of distillation practically constant and equal in each case throughout the whole proceeding, the rate being kept at between seventy and eighty drops per minute. In the ordinary distillation a piece of asbestos with a circular hole, one inch in diameter, in its center was placed under the flask, so that the flames struck the flask only at this point. In this way the upper part of the flask was prevented from being superheated. Several capillary boiling tubes were placed in the flask. The electrical distillation was conducted in the usual way, the current being regulated by means of a rheostat.

Below is a table giving the results. The densities were taken at 25° by means of an Ostwald-Sprengel pycnometer. They were referred to water at 15.6°, so that the contents of the fractions in alcohol might be taken from the convenient tables given in Bulletin 107 of the United States Department of Agriculture.

FIRST COMPARISON OF METHODS OF DISTILLATION.

	Ordinary distillation.		Electrical distillation.	
Designation of fraction.	Density of fraction.	Per cent. alcohol by weight.	Density of fraction.	Per cent. alcohol by weight.
I.....	0.8447	78.1	0.8412	79.5
II.....	0.8918	58.5	0.8943	57.4
III.....	0.9938	2.4	0.9946	2.0

Thus it is evident that the two processes gave nearly identical results, the electrical distillation, however, being somewhat the more efficient of the two. The gain is not, however, as great as that inferred in the other cases, judged solely by means of the constancy of the boiling point.

In order to obtain confirmation of the verdict of the densities, the refractive indices of these various samples were determined by means of a Zeiss immersion refractometer. The results obtained in this way

essentially confirmed those above, showing a small margin in favor of the electrical distillation.

Because a single experiment is never conclusive, it was thought worth while to repeat the comparison, in this case taking the temperatures at intervals of two minutes throughout each distillation. The volumes also at each of these intervals were measured, but need not be detailed here. The distillation was carried out essentially as before, care being taken that the conditions should be essentially similar in the two cases. In this case the two were not carried out simultaneously, however; and it happened that the pressure during the electrical distillation was 5 mm. less than in the ordinary distillation, and the rate of distillation was slightly greater in the latter case. The difference is so slight, however, that it could hardly have had any essential effect upon the results.

SECOND COMPARISON OF METHODS OF DISTILLATION.

	Ordinary distillation.			Electrical distillation.		
Fraction.	Range of boiling point.	Density.	Per cent. of alcohol.	Range of boiling point.	Density.	Per cent. of alcohol.
I.....	80– 83°	0.8445	84.0	78–80.5°	0.8404	85.2
II.....	83– 98°	0.8934	65.5	80.5–98°	0.8899	66.9
III.....	98–100°	..	..	....	..	..

These results agree essentially with the previous ones, giving a verdict slightly in favor of electrical distillation.

The range of temperature in the first fraction was somewhat less when the distillation was conducted electrically than when the vessel was heated from outside, but the gain in purity, as indicated in the preceding table, was not quite great enough to correspond to this diminished temperature range. The superheating evidently at first amounted to about 1.8° in the case of the ordinary distillation, but was negligible in the electrically conducted process.

In brief, this note shows that while distillation by means of a wire heated electrically effects a somewhat better separation than the ordinary method and causes much less superheating in the liquid, the gain in the efficiency of separation is not always as great as had been at first inferred from the great constancy of boiling point observed in a special series of cases.

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SOME ORGANIC COMPOUNDS OF BERYLLIUM.

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It has been repeatedly pointed out by one of us[1] that solutions of the

[1] THIS JOURNAL, **26**, 1444; **28**, 555. *Z. anorg. Chem.*, **49**, 178. *J. Physic. Chem.*, **11**, 651. "Chemistry and Literature of Beryllium," p. 61.