CONCERNING THE USE OF ELECTRICAL HEATING IN FRACTIONAL DISTILLATION.

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In the course of a research[1] now in progress in this laboratory it became necessary to fractionate a number of organic liquids in order to prepare them in a state sufficiently pure for investigation. The process of distillation was at first carried out in the usual manner, but some of the substances required very many successive systematic distillations in order to furnish enough material boiling within a reasonable limit of temperature, and indeed, in more than one case the task seemed hopeless.

A part of the research in question involved the determination of the latent heat of vaporization of the various substances by means of a modification of Kahlenberg's method,[2] to be described later. In the course of these experiments, it was noticed that each organic liquid boiled at a much more constant temperature when heated electrically by the platinum coil of this apparatus than it had during its previous fractional distillations in an ordinary boiling flask. This led to the use of the hot platinum coil instead of the gas burner as a source of heat in the preliminary fractional distillation, with a very great gain in the efficiency of this process.

Probably the reason for this difference in efficiency between the two methods of heating lies in the difference in the extent of superheating. The success of fractional distillation might be supposed to be impaired when superheating occurs, for in this case the higher boiling fractions would naturally have more tendency to come over with those of lower boiling point. In order that the most effective separation may be made, the temperature of the liquid should never exceed the true boiling point of the mixture. Very considerable superheating occurs when a liquid is boiled in a glass flask by the application of heat from outside. On the other hand we found that very little superheating of a liquid occurs when the liquid is heated by means of an electric current passing through a suitable resistance wholly immersed in the liquid. S. Lawrence Bigelow has suggested this method of heating in the determination of the molecular weight of a substance in solution by measuring the elevation in boiling point; its satisfactory application to this problem is an indication of its efficiency in obviating superheating. It is clear therefore that the electrical method of heating might be expected to give more complete separation during the process of practical distillation than the ordinary method.

The matter is so obvious that probably others have thought of this

[1] This Journal, **30**, 8 (1908).

[2] Kahlenberg, *J. Phys. Chem.*, **5**, 215 (1895).

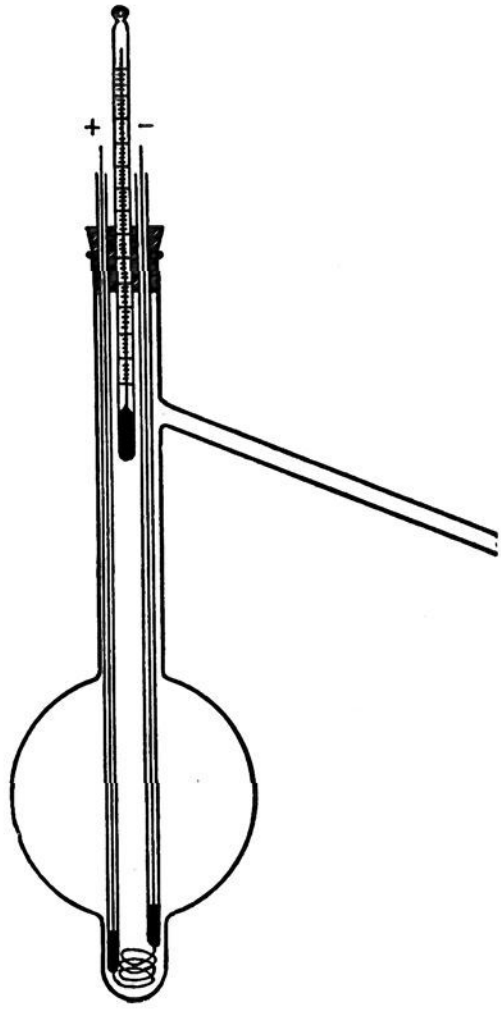
before; but because we have never seen the method in use nor have been able to find a reference to it in chemical literature, we venture to call attention to it in this brief paper.

The extent of the increased efficiency is best indicated by two parallel experiments, alike in every essential respect except the difference in the source of heat, and the fact that into the ordinary boiling flask Markovnikov capillary tubes were placed to relieve the superheating to some extent. Even with this precaution added to the old way, the difference in result was very marked, as the following figures show.

0.1 liter of a specimen of normal butyl alcohol, dried with anhydrous copper sulphate, needed *six* distillations in order to secure 75 mm. of liquid boiling within the limits of 1° (117.0°–118.0° at 759 mm.), using the ordinary method of outside heating by a gas flame.

The same volume of the original liquid by only *two* fractional distillations with electrical heat yielded the same volume of distillate of a much higher grade of purity, having boiling point limits only 0.6° apart (117.3°–117.9°).

Similarly, 120 mm. of cresol which in one distillation gave 100 mm. within 0.8° (190.0°–190.8° at 765.0 mm.) gave an equal amount boiling within 0.3° (189.9°–190.2° at 758.5 mm.) by the new method. Numerous other examples might be cited, but these are sufficient to show the great advantage to be derived from electrical heating.



A word concerning an advantageous form of apparatus is not out of place, although a heating resistance-coil may be immersed under the liquid in any ordinary distilling apparatus. In order to economize material, a narrow cistern was blown into the bottom of a common stout distilling flask. Into this depression the heating coil was placed. The coil consisted of about 40 cm. of platinum wire having a resistance of about 0.7 ohm. A current of from ten to fifteen amperes was led to the resistance wire from above by heavy copper wires encased in glass tubes, into the ends of which the ends of the platinum wire were sealed, contact being made by a drop of mercury. It is necessary that these copper wires be heavy (about 2.5–3.0 mm.

in diameter) so that they may not become heated by the current and thus superheat the vapor coming into contact with the glass tubes encasing them. For this reason it might be well to introduce the electrical connection from below, through the glass walls of the cistern; but obviously the present arrangement can be most easily made. It is necessary that the coil and mercury contacts be entirely covered by the liquid at all times. The diagram illustrates the arrangement. The coil was more compact than that represented in the figure, so that it was possible to distil all but 4 or 5 mm. without uncovering the resistance.

It is almost needless to call attention to the fact that short-circuiting through the liquid may cause slight decomposition when electrolytes are thus heated; hence the method is not well applicable to liquids of this class.

Because the bubbles of vapor arise only from the small area of the hot resistance wire, ebullition proceeds quietly, and there is never any tendency to "bump." This method of heating is therefore especially applicable to fractional distillations under reduced pressure, where so much trouble is usually experienced from the explosive formation of vapor. Concentrated sulphuric acid, for example, boils as quietly under greatly reduced pressure when so heated as does water or alcohol under ordinary pressures. The method of heating dispenses entirely with the necessity of passing air through the liquid in vacuum distillations, and heavy viscous liquids may be advantageously distilled in this way. By combining this method of heating with the Hempel, Wurtz, Linnemann or other fractionating towers great efficiency may be expected. However, where the amount of material is small, the towers cannot be advantageously used, because of the loss of material required to wet the considerable area of their condensing surfaces; and it is very convenient to have at hand an economical method fully as efficient as the ordinary method where the tower is used.

The method may also find successful application in the distillation of inflammable liquids and may therefore be of some industrial importance where power may be obtained cheaply. Moreover, low boiling liquids, ordinarily requiring special precautions, can be distilled as expeditiously as those of high boiling point, since superheating is impossible.

In brief, this article describes experiments showing the great gain in the efficiency of separation obtainable by the use of electricity as a source of heat in fractional distillation. An advantageous form of apparatus for this purpose is described.

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