The neutral salts selected were salts that would naturally be present in the living tissues.

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AN ELECTRICALLY HEATED VACUUM FRACTIONATION APPARATUS.

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In developing methods for the analysis of essential oils, it was found desirable to improve, if possible, upon the ordinary apparatus for fractional distillation *in vacuo* and, as a result of a year or more experimentation with various styles of apparatus, the following has finally been adopted.

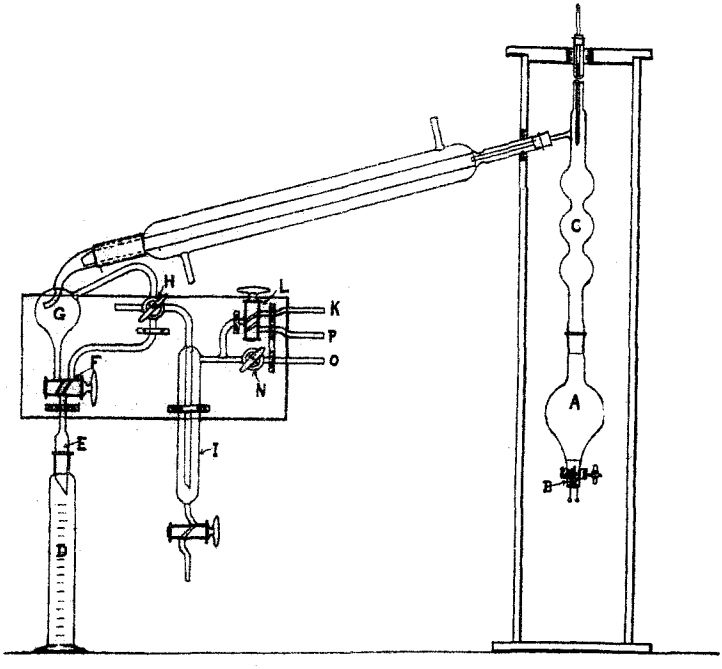
The suggestion made by Richards,[1] that fractional distillations can be carried on much more expeditiously by the use of a heating coil within the distillation flask itself than when the heat is applied externally, led us to use a flask of the style shown in the accompanying sketch, A.

For working with 50 cc. portions of oil, this should have a capacity of approximately 150 cc. The heater B is a small coil of German silver or nichrome wire attached to platinum leads which in turn are sealed into the bottom of the flask. As it was desirable to distil off all but the last 10 per cent. (5 cc.) from the sample of oil and to leave as near as possible exactly this amount in the flask, it was necessary to have the bottom of the container as small as practicable. For this reason, it was made long and narrow, the 5 cc. mark being a little above the top of the heater. A fractionating head, C, of the Ladenberg type, is attached to the flask by a ground joint, the upper end of this head being extended some distance above the outlet tube so that the stopper through which

[1] THIS JOURNAL, 30, 1282; 31, 1200.

the thermometer passes is in no danger of being attacked by hot vapors. The flask and head are enclosed in a wooden box to protect them from drafts as it was found otherwise impossible to carry on the distillation at a uniform rate.

The receiving end of the system is so constructed that either the distilling flask or the receiver may be evacuated, the one independently of the other. Thus, in beginning a distillation, the graduated cylinder D is slipped on to the ground joint E, stopcock F turned so as to connect this receiver with the safety bulb G and stopcock H which is a four-way cock bored with an L-shaped opening set so that the



safety bulb is connected with the evacuating system through a trap, I. This trap serves to catch any of the distillate which, in case of an accident, might otherwise be sucked through into the pump connections and lost. Tube K, attached to the house vacuum, is then connected with the system through stopcock L and the major portion of the air in the entire apparatus quickly withdrawn. With the vacuum available in the Bureau of Chemistry, the pressure, as indicated on the manometer, will fall to about 20 mm. L is then closed and stopcock N, which is attached to an ordinary Chapman pump, opened, and by means of this the pressure still further reduced, in cold weather to about 12 mm. Cock N is then shut off, which, as it is a three-way, makes at the same

time connection between the water pump and the outside atmosphere, preventing any backing up of the water, and L opened so as to connect I with the Geryk pump through the tube P. With this machine we are able to work readily below 5 mm. pressure. The current being turned on to the heater through a suitable rheostat, distillation is begun and the rate regulated as required by merely varying the external resistance.

When, for any reason, it is desired to separate one fraction from another, this is easily accomplished by reversing stopcock, F, thus closing G and connecting E directly with cock H, which may then be set so as to close the distillation system and open the receiver to the atmosphere. The first cylinder may now be removed and another one, which is ground to fit the taper of the adapter a little further down, slipped into place. Stopcock H is again turned, this time so as to connect the new receiver with the vacuum pump, the pressure in the boiling flask being unaffected. After the receiver has been evacuated, cock H is set to connect the pump with the distilling system and F reversed and the distillate which has collected in trap G during the exchange of cylinders, run into the new receiver. If proper precaution be taken to have all joints tight, the vacuum in the distilling flask and condenser will drop very little while the change of receivers is being made. Distillation then proceeds as before.

The apparatus, while it may look complicated, is in reality a comparatively simple one such as can be made up, with the exception of the distilling flask itself and the ground joints between the receivers and the adapter, in almost any laboratory from stock articles. The ground connections may be replaced by rubber if tube E be made with a long taper so as to fit into an ordinary graduated cylinder leaving just sufficient space for a rubber sleeve between the two.

It is not absolutely necessary that provision be made for connecting with the house vacuum and water pump previous to turning on the mechanical Geryk but this is desirable wherever practicable as it saves considerable time, and as a rule, oil immersion pumps work much better when they have only a small quantity of air to handle.

While there are no radical improvements in this apparatus over various others upon the market, still we have found that it combines the advantages of all of these without their disadvantages. In the method for separating fractions used by Brühl and others, it is impossible to work with one fraction until the distillation is complete or at least without interrupting it, but here as many fractions as desired may be taken off, one after the other, without changing the rate of distillation. This we have found to be very important where empirical methods are involved.

We have substituted in the heating coil other resistance materials

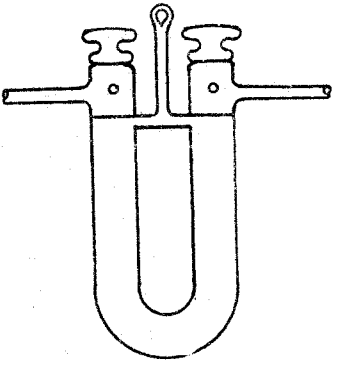
for platinum, not only as they are much less expensive, but also because with them the superheating and consequent bumping seems to be less. The joints between the heavy platinum leads used through the glass and the coil are made by twisting the two together and covering the union with a little bead of melted "Einschmeltz" glass. Probably because there is a high resistance at this junction, the bubbles of vapor originate here and rising through the body of the liquid keep it as well stirred as it would be with a stream of air. Of course, in working with liquids which would attack the base metals, the alloys cannot be used but they can safely be employed with most organic substances.

The fact that the source of heat is under absolute control makes it possible to regulate the rate of distillation very closely and to duplicate one's fractionations almost exactly. This is of great advantage where a number of samples of the same material are being examined, as often comparatively small variations in the rate of distillation materially affect the properties of the various fractions.

NOTE.

A Modified Drying Tube.—In view of the large percentage of breakage of U tubes and the resultant loss to the chemist of time and determinations when engaged in carbon analyses, the writer has endeavored to modify the usual form of tube in order to minimize this trouble.

In the accompanying photo is shown the tube as used in this laboratory and made for us by Eimer & Amend at an additional cost of only 25 cents each. The tube is of thick glass and is of the same bore throughout.



The brace and perpendicular bar are of glass possessing practically the same coefficient of expansion as the body of the tube, in order to properly withstand temperature changes, while the end of the upright bar is drawn into a loop, by means of which the tube may be suspended from the balance beam or train support. The advantage to be gained from this loop is great in having all the arm openings upon correct alignment during an analysis and in eliminating the use of wire in suspending.

Then, too, we do away with the changes of weight due to chemical action upon copper wire, which is usually used, and an entire glass surface is presented for wiping before weighing.

It can readily be seen that the breakage at the curve of the U, due of pressure or pull, is reduced to a minimum, and we believe the small