THE DISTILLATION OF COCOANUT OIL AT ESPECIALLY LOW PRESSURE

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Chevreul has already distilled not only fatty acids but also fats without decomposition in a Torricelliam vacuum[1]. Caldwell and Hurtley have distilled[2] butter-fat and cocoanut oil in a high vacuum and without decomposition. In the case of cocoanut oil almost 50 per cent is distilled up to 210° C. But their investigations are occupied more with fatty acids than with fats. Later on Bömer repeated the distillation of cocoanut oil[3]. He worked in a vacuum of the cathode light and obtained 87 per cent distillation at a temperature of 210-285° C. Bömer did not distill to completion, and moreover the residue underwent decomposition as distillation progressed, while in addition the distillate already obtained from redistillations under the same conditions exhibited decomposition phenomena.

We have made a further study of this question and have succeeded in completely distilling over cocoanut oil in vacuo without the occurrence of appreciable decomposition. This is made apparent by the constants obtained from analysis. But as far as smell and taste were concerned, the white fractions obtained showed no edible oil characteristics. The first fraction had a 'sharp (burning) taste and a somewhat pungent odor. This was caused by traces of decomposition products. It is also possible to re-distill the obtained fractions in vacuo.

The material forming the starting point of the tests consisted of 200 gr. of pure refined cocoanut oil obtained from one of the Dutch oil factories. There were obtained four fractions with a weight of 60, 35, 55, and 43 gr., or respectively 30, 17.5, 27.5, and 21.5 per cent. These fractions went over respectively at 206-223° C., 223-229, 229-231, and 231-254. The attenuation of the air during the distillation was not measured but throughout the entire experiment the Geissler tubes *J* and *X*, (see figure) remained unillumined, so that the pressure was too small to be measured by means of these tubes. Measurement of the temperature was carried out by means of a thermocouple; an ordinary thermometer inserted in place of the thermocouple *Q* was also used. The temperature at the point *R* was also capable of being determined by means of a thermocouple.

The figures obtained for the original cocoanut oil and for the distillates are given in the tables on the page following.

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* Reprinted from the Journal of the German Oil and Fat Industry.

Refraction n_d (60° C.)

Original oil	1.4420
Fraction 1	1.4391
Fraction 2	1.4398
Fraction 3	1.4408
Fraction 4	1.4447

Acid number (Milligrams KOH per gram)

Original oil	0.945
Fraction 1	1.4
Fraction 2	0.9
Fraction 3	0.9
Fraction 4	0.27

Saponification number (Milligrams KOH per gram)

Original oil	256	Accuracy ± 2
Fraction 1	280	
Fraction 2	274	
Fraction 3	262	
Fraction 4	237	

Iodine number (Wijs)

Original oil	8.2
Fraction 1	2.4
Fraction 2	1.5
Fraction 3	4.3
Fraction 4	23.3

From these figures it appears that for practical purposes no decomposition of the fats into fatty acids has taken place.

If saponification numbers of the fractions obtained are considered additively, we find 25,600 per 100 gr. for the original oil;

for fraction 1	30.0 × 282 = 8,460
for fraction 2	17.5 × 274 = 4,795
for fraction 3	27.5 × 262 = 7,205
for fraction 4	21.5 × 237 = 5,095
Total	25,555

The losses during distillation were 3.5 per cent. In the fractions we find a total saponification number of 25,555—a figure of considerable size when considered in relation to the losses. In the same way we find a small loss in the case of the acid number, a result which must be acknowledged as favorable.

The iodine number proves that those substances which fix iodine in Wijs's method are only recovered in the distillates of higher boiling point. This is in accordance with the observations of Bömer and Baumann.

The apparatus used will be found detailed in the figure cited. The vacuum was at first maintained by means of a preliminary pump I, aided by flasks containing carbon (H_1 and H_2), and placed in Dewar flasks containing liquid air, while at the same time a mercury pump *G* according to the design indicated by Langmuir[4] was shunted in.

From the distilling bomb *A* made of Pyrex glass and heated with the air bath *P* the fat was distilled. The distillate passed via *D* to the

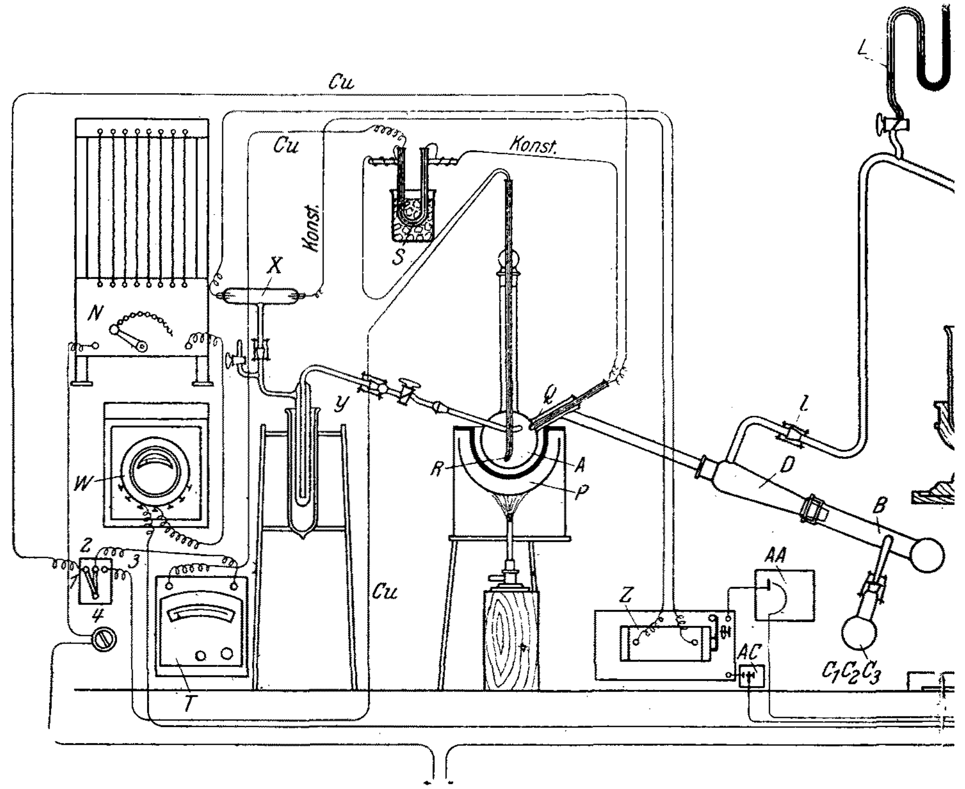


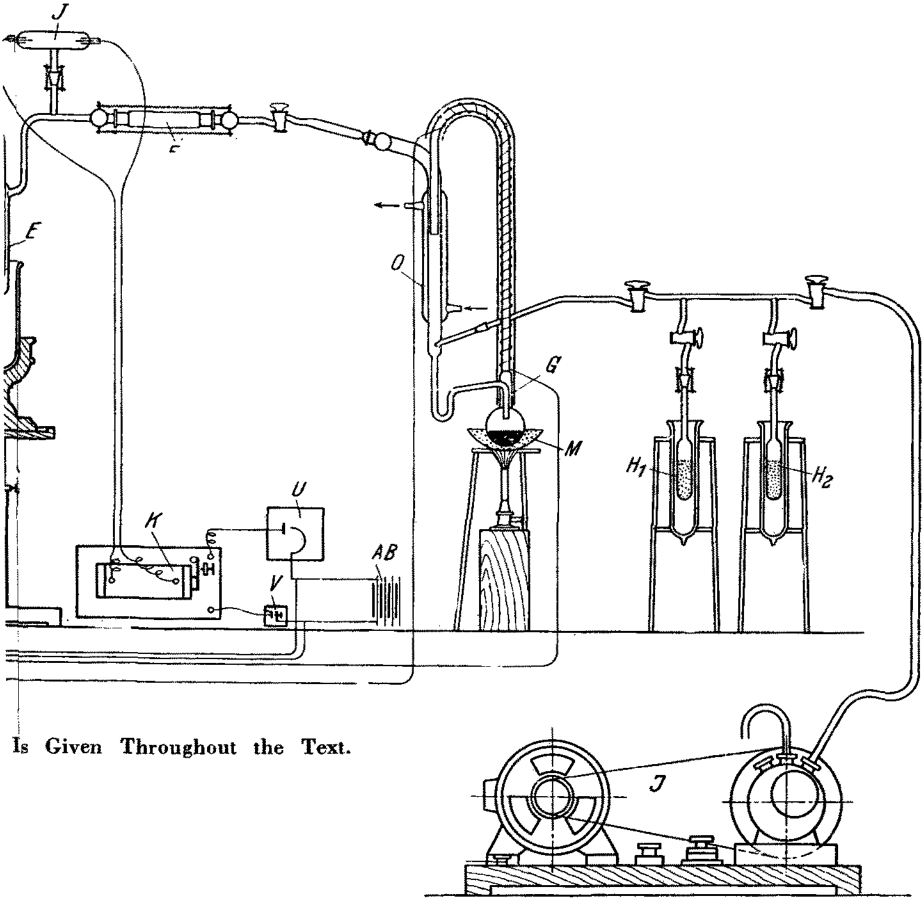
Diagram of Apparatus Used in the Study Reported in This Paper. Explanation of Figure

receiver *B* equipped with the small bulbs connected by ground joints C_1, C_2, and C_3.

The space, which was highly evacuated, was made entirely of glass and so far as necessary, was fitted with carefully ground cocks and connecting pieces.

The vessel *E* (in a Dewar beaker containing liquid air) served to condense very volatile constituents which do not leave the receiver as condensates. In F there was placed a dish of P_2O_5.

The mercury pump was heated by means of the sand bath *M*. In order to prevent the condensation of mercury vapor in the riser, this tube was surrounded by asbestos wired on with nickel wire. This wire was connected with a direct-current circuit by means of a rheostat *N*. A cool-



Is Given Throughout the Text.

ing water connection served to cool the jacket *O* of the mercury pump.

We observed that during the distillation process (an undertaking of only a few hours) the liquid in the bomb *A* was entirely quiet[5] and did not exhibit the well-known "phenomena of boiling."

Because of the fact, already stated, that the pressure was too small to be measured even approximately by means of a Geissler tube passing an electric current, the distillation was repeated and in the course of this the vacuum was measured by means of a MacLeod manometer[6]. In this case also 200 gr. of cocoanut oil were distilled. The weights of the fractions recovered were respectively 26, 49, 71, and 47 gr. or respectively 13, 24.5, 35.5, and 23.5 or altogether 96.5 of the original product.

The temperatures at which these fractions distilled over (measurements at *Q*) were this time respectively 208 to 218° C., 218-223, 223-237, 237-259, measurements which were at the same time carried out with the ordinary thermometer.

The pressure at the beginning of the distillation was 0.0023 m.M. of mercury and at the end 0.0034, the vapor pressure of the mercury not being considered.

For the calculation of these pressures from the observed heights of the mercury column in the capillary tubes of the manometer we used the calibration values established for the manometer by Dr. G. Meyer, for which we sincerely thank him.

We are convinced that the ease with which distillations of this kind may be carried out can become of great importance for the analysis of fats. It is now possible to distill without decomposition those glycerides such as occur in cocoanut oil, and the analysis will undoubtedly be capable of being rendered more precise.

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1) Journal of the Chemical Society XCV, Trans. I page 853 (1909).

2) K. S. Caldwell and W. H. Hurtley, The distillation of butterfat, cocoanut oil, and their fatty acids, Journal of the Chemical Society, XCV, Trans. I page 853 (1909).

3) A. Bömer and J. Baumann, Contributions to the knowledge of glycerides of fats and oils IX. The glycerides of cocoa butter, Zeitschr. Unters. Nahr.-u. Genussmittel 40, 97 (1920). Cf. also Ch.-Ztg. 38, 844 (1914).

4) See for instance B. J. Heerdt, Overzicht van de theorie en de toepassingen van gassen, waarin de onderlinge botsingen der mcleculen kunnen verwaarloosd worden, Utrecht-Nijmegen 1923, blz. 274.

5) Caldwell and Hurtley op. cit. page 855.

6) F. H. Newmann, The production and measurement of low pressures, London, 1925.

7) An apparatus from the collection of the laboratory for inorganic chemistry of the Technical College was kindly placed at our disposal.