

INVESTIGATION CONCERNING THE REICHERT-MEISSL NO. AND
THE RATE OF DISTILLATION OF THE VOLATILE
ACIDS IN BUTTER FAT.

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In 1906 J. Delaite and J. Legrand (Bul. Soc. Chim. Belg.) investigated the determination of the volatile acid. He found the R. M. No. to increase when saponification was continued from one-fourth to six hours. This they claimed was due to depolymerization.

In the regular work of the laboratory no such variation was observed in the routine work of determining the volatile acids. The time of saponification varied from one to one and one-half hours.

To determine the effect of continuing the saponification on the per cent. of volatile acids obtained by the Reichert-Meissl process, 10 determinations were made using the same butter fat and following the A. O. A. C. method. (p. 189, 1908), the saponification being carried out under method (a), under pressure with an alcoholic solution of potassium hydrate. The saponification flasks were completely submerged in a steam bath at a temperature of 105° C. This was done to insure a more uniform temperature during the time of saponification.

The time of saponification varied from 15 minutes to two and one-half hours. The quantity of butter fat taken was as near five grams as could be weighed accurately. The result calculated on the basis of five grams.

In distilling the volatile acids the conditions were kept as uniform as it was practical, the rate of distillation being so regulated that 110 c.c., the required amount was distilled in 30 minutes.

Ten determinations were made. The results are shown in the following table.

TABLE I.

Showing the Effect of Time of Saponification.

Time in minutes.....	15	30	45	60	75	90	105	120	135	150
Reichert-Meissl No.....	28.32	28.25	28.35	28.29	28.29	28.28	28.35	28.32	28.28	28.31
Per cent. Vol. Acid as Butyric.....	4.93	4.97	4.99	4.98	4.98	4.97	4.99	4.98	4.97	4.98

Some allowance must be made in the time factor, slight saponification taking place before placing in the steam bath, also during the time of cooling after removing the flasks. But it will be observed that this factor was uniform for the 10 determinations.

From the figures in Table I. no such variations are indicated as reported.

Some of the factors which influence the Reichert-Meissl No.

Rate of distillation.

Failure to remove alcohol (when used).

Size of distilling flask.

Absorption of carbonic acid and quantity of fat taken.

All those factors are under the control of the operator and constant results are obtained by observing uniform conditions.

The rate of distillation of the volatile acid by the Reichert-Meissl process, also the rate of distillation of the volatile acids by distillation with steam.

In determining the rate of distillation of the volatile acids by the Reichert-Meissl process, the distillate was collected in fractions of 10 c.c.

and titrated with $\frac{N}{10}$ NaOH.

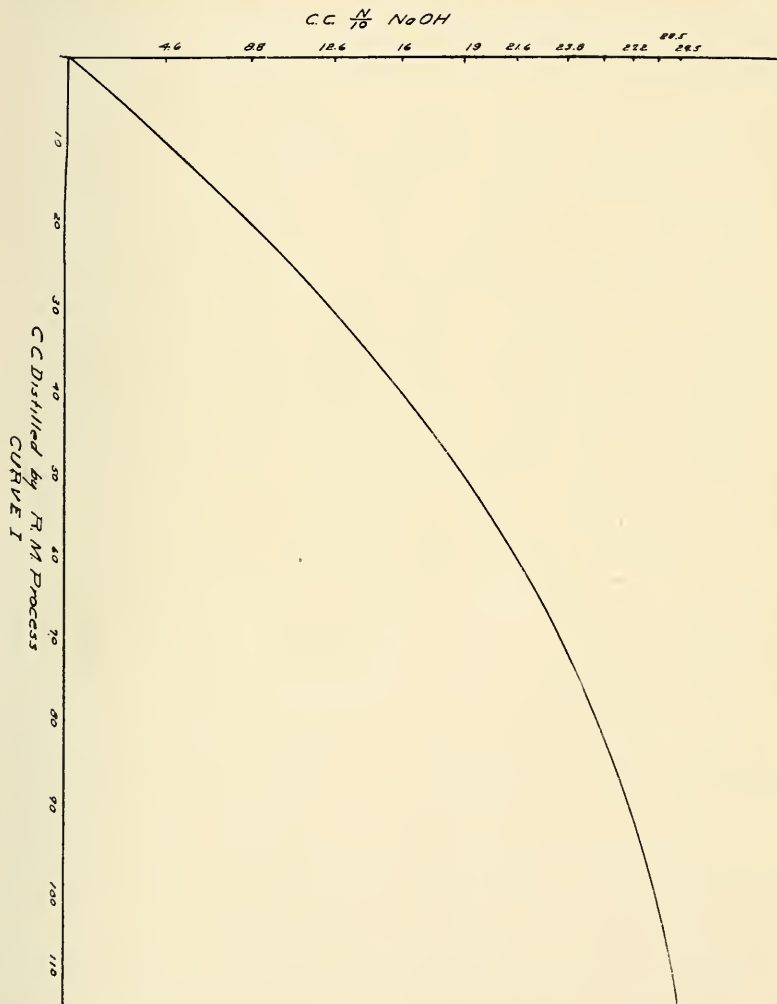
The number of cubic centimeters of $\frac{N}{10}$ alkali required to neutralize each fraction are tabulated in Table II, also the per cent. acid calculated as butyric acid based on five grams of butter fat taken.

TABLE II.
Showing Rate of Distillation by the R. M. Process.

No.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	Total
C. C. $\frac{N}{10}$ NaOH.	4.6	4.2	3.8	3.4	3.	2.6	2.2	1.9	1.5	1.3	1.0	29.50
Per cent. vol. acid as butyric.81	.74	.67	.60	.53	.45	.38	.35	.26	.23	.17	5.19

From Table II it will be seen that the first fraction of 10 c.c. of the distillate contains 15.6 per cent. of the total volatile acid, uniformly decreasing to the 11th fraction, which contains only three per cent. of the total volatile acid.

Plotting the above results, the volume distilled as abscissa and the number of c.c. of $\frac{N}{10}$ alkali used to neutralize the distillate, we obtain the following graphical representation of the R. M. process of distillation.



The total number of c.c. required to neutralize the volatile acid was 28.5 c.c. corresponding to 5.19 per cent. of acid calculated as butyric acid.

By the Reichert-Meissl process, we obtain only a certain fraction of the total volatile acids and which is fairly constant if carried out under standard methods.

To determine the relation of the volatile acids obtained from the R.-M. process of distillation to the total volatile acids, distillation was made with steam. By this means it is possible to estimate the total volatile acids. The usual method of saponification and precautions were taken as in the R.-M. process.

One thousand c.c. were distilled with steam and an aliquot portion titrated which gave a total of 6.03 per cent. volatile acid as butyric acid. In the R. M. process, 5.19 per cent. of acid was obtained from the same butter fat. Thus we see that only 86 per cent. of the total per cent. of volatile acids were obtained by the R. M. process.

The Rate at which the Volatile Acids Distill by Means of Steam.

The same method was used as in the previous experiment in determining the total volatile acids. The distillation was collected in portions of 50 c.c. and titrated with $\frac{N}{10}$ NaOH. Twenty fractions were titrated and the result shown in Table II.

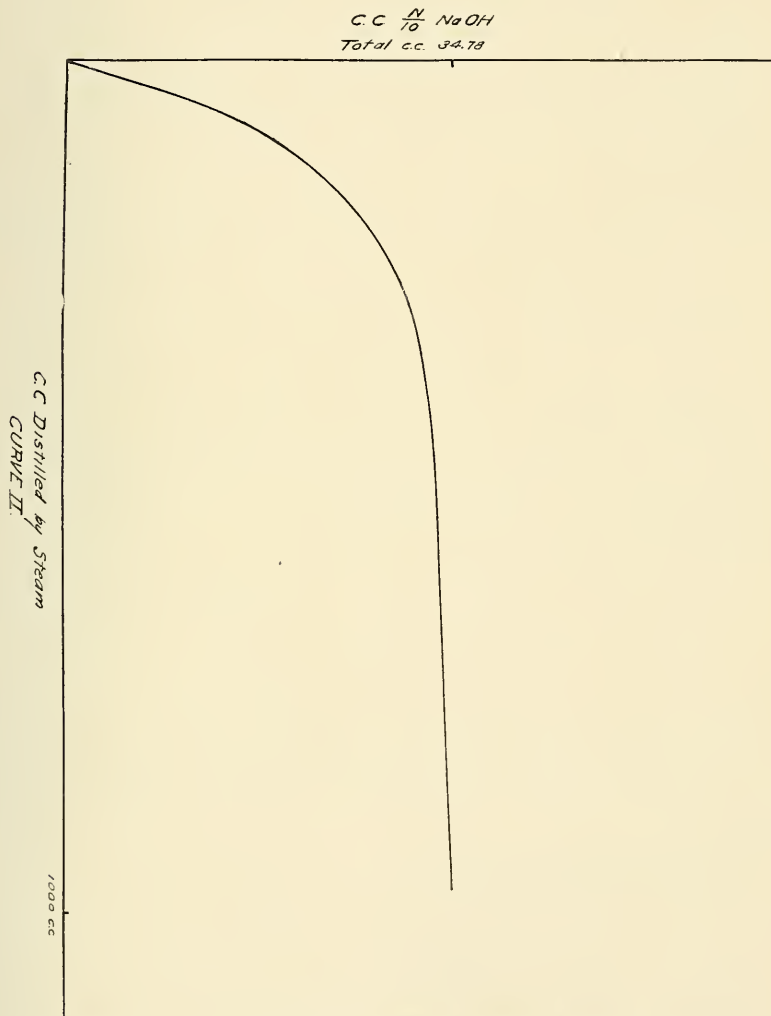
TABLE III.

No.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.
C. C. $\frac{N}{10}$ -NaOH	12.7	8.48	5.58	3.66	2.4	1.52	1.00	.70	.60	.45	.30
Per cent. vol. acid as butyric	2.03	1.36	.89	.59	.39	.25	.16	.11	.08	.06	.047
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No.	12.	13.	14.	15.	16.	17.	18.	19.	20.	Total	
C. C. $\frac{N}{10}$ -NaOH30	.25	.20	.20	.18	.15	.14	.13	.10	38.74	
Percent. vol. acid as butyric047	.04	.03	.03	.028	.023	.022	.021	.017	6.22	

The weight of butter fat taken for the experiment as shown in Table II was 5.50 grams. The per cent. of volatile acid and distillate is based on five grams of fat.

From Table II it is seen that the first fractions contained the greater part of the volatile acids, decreasing rapidly after the second fraction, and that the volatile acids are practically all distilled when 1000 c.c. have been collected.

Plotting the c.c. distilled as abscissa and the c.c. of $\frac{N}{10}$ alkali required to neutralize the distillate as ordinates, we get a curve quite different from the one obtained by the Reichert-Meissl process.



In distillation by the R.-M. process and distillation with steam, we meet with different conditions.

In neither case is the vapor saturated with the volatile acids of butter fat during the period of distillation and the liquids in the still are made up of water and the insoluble fatty acids.

The vapor pressures of the volatile acids differ and their solubility in water and fatty acids influence the water of distillation.

Of two acids having approximately the same vapor pressure, the one which is least soluble in the mixed liquid will distill the faster.

Combining the factors, solubility in water and in the fatty acids, a mathematical expression for the rate of distillation becomes only approximately true.

Theoretically,¹ if we do not keep the volume constant as is the case in the R.-M. process of distillation, that is by making no addition to the liquid in the still during distillation,

The equation is $\frac{dy}{dx} = a \frac{y}{x}$ and integrating we get

$$\text{Log } y = a \text{ Log } x + c \text{ or } y = x^a.$$

y equals amount of volatile acids left in solution and x amount of liquid left in still, the original amount being taken as 1.

On the other hand, if the volume is kept constant as is the case in steam distillation x becomes constant.

In this case we consider the quantity of water removed to the quantity of volatile acids left in the still.

We then write equation $-\frac{dy}{dx} = ay$. Integrating we get the equation

$$-\text{Log } y = ax + c, \text{ or } y = \frac{1}{ax}.$$

y = amount of volatile acid left in solution, original amount being taken as 1; x = amount of water and volatile acids distilled.

The above equations do not take into account the condensation in the still.

¹ H. D. Richmond, Analyst, 1908.
S. Young, fractional distillation.