

Translation of Technical Oil Mission Microfilm Reel #101
Item 21579

Foot 29 - Frame 9

Foot 30 - Frame 2

THE INVESTIGATION OF THE WATER PRODUCT FROM A MEDIUM
PRESSURE SYNTHESIS EXPERIMENT WITH AN INITIAL GAS
RICH IN HYDROGEN USING AN IRON CATALYST

The water layer in the resulting reaction mixtures obtained in the hydrocarbon synthesis was separated from the liquid and solid hydrocarbons. The water layer was originally colorless, changing to a light brown color after standing for a day. The solution was clear, acid to litmus, and contained an appreciable amount of dissolved iron salt.

Quantitative Determination of Alcohols
and Fatty Acids Present in the Water Product

The water layer, having a volume of 735 mls., was treated with an excess of caustic solution and refluxed for an hour. After neutralization, the solution was distilled to an overhead temperature of 100°C. The total amount of distillate which consisted of the alcohol fraction was 73.2 mls.

A qualitative test on the distillate showed the presence of ketones (acetone, methyl ethyl ketone) and absence of aldehydes.

The boiling point curve of the subsequent distillation of the alcohol fraction, obtained with a good microfractionating column (15 theoretical plates), is shown in Figure 1.

The constituents of the water product were calculated as the pure alcohols on the basis of the boiling point curve, and taking into consideration the constant boiling azeotropes of mixtures of ethyl and propyl alcohols with water. The results are tabulated in Table I.

Table I

CONSTITUENTS OF THE WATER PRODUCT AS PURE ALCOHOLS IN WEIGHT %

	<u>Weight %</u>
Methyl Alcohol	0.6
Ethyl Alcohol	3.2
N-Propyl Alcohol	1.3
N-Butyl Alcohol	0.5* (up to 0.6)

* On the boiling point curve in Figure 1 the fraction distilling at 92°C. was assumed to be from an analogy of the other alcohol fractions an azeotropic mixture of butyl alcohol and water. Due to the lack of literature data the exact value could not be obtained.

The fraction boiling over 100°C. was evaporated on the steam bath after the neutralization of the water product with caustic and the separation of the alcohols. The sodium salts were decomposed with dilute sulfuric acid. The solution was saturated with sodium chloride and extracted with ether for 150 hours in a Kutscher-Stuedel apparatus.

After distilling off the ether, 1.7 gm. of pure fatty acids remained. Based on the total water product, this weight of fatty acids amounted to 0.23 weight percent.

For further identifications, the fatty acids were esterified with methyl alcohol in the presence of sulfuric acid, and the esters distilled in the micro column.

Only one fraction (containing 1.8 ml. of methyl acetate) was obtained since on increasing the distillation temperature the esters of the higher fatty acids decompose in the presence of sulfuric acid.

No methyl formate was found.

The amount of acetic acid obtained from the methyl acetate was 1.36 gm. Methyl acetate accounted for approximately 80 weight percent of this amount. The remaining 20 weight percent of the acetic acid was attributed to the higher fatty acids which were distinguished through their typical odors.

Formic acid was not obtained.

In the original water product, approximately 80% of the fatty acids were present in the free form as shown by the acid and saponification number determinations.

This fact is also proven in the boiling curve in Figure 2 of the fraction of the original water product boiling up to 100°C., and in the corresponding fraction of the saponified water product in Figure 1.

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Figure 1

MICRODISTILLATION OF THE SAPONIFIED WATER

PRODUCT FRACTION BOILING UP TO 100°C.

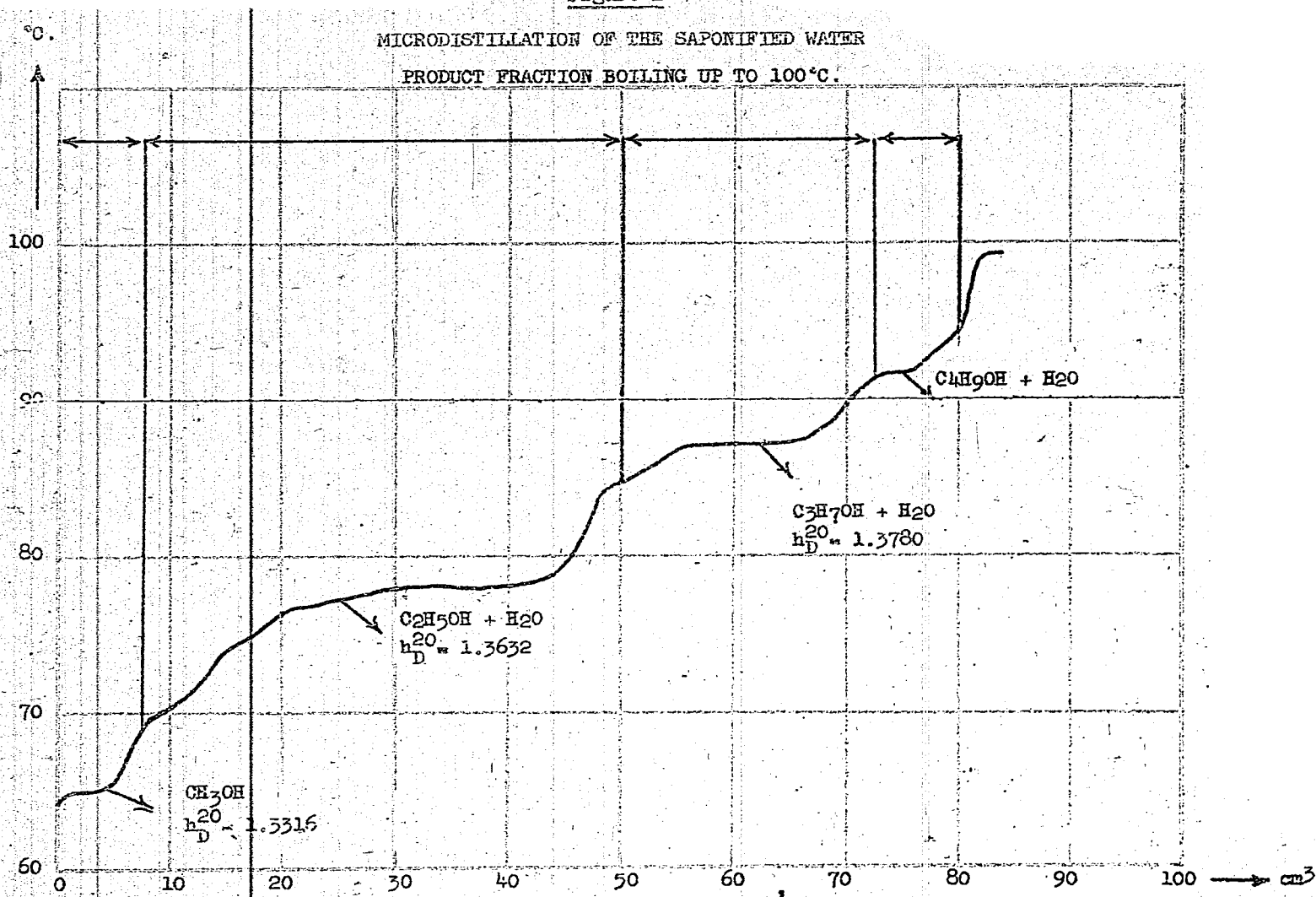
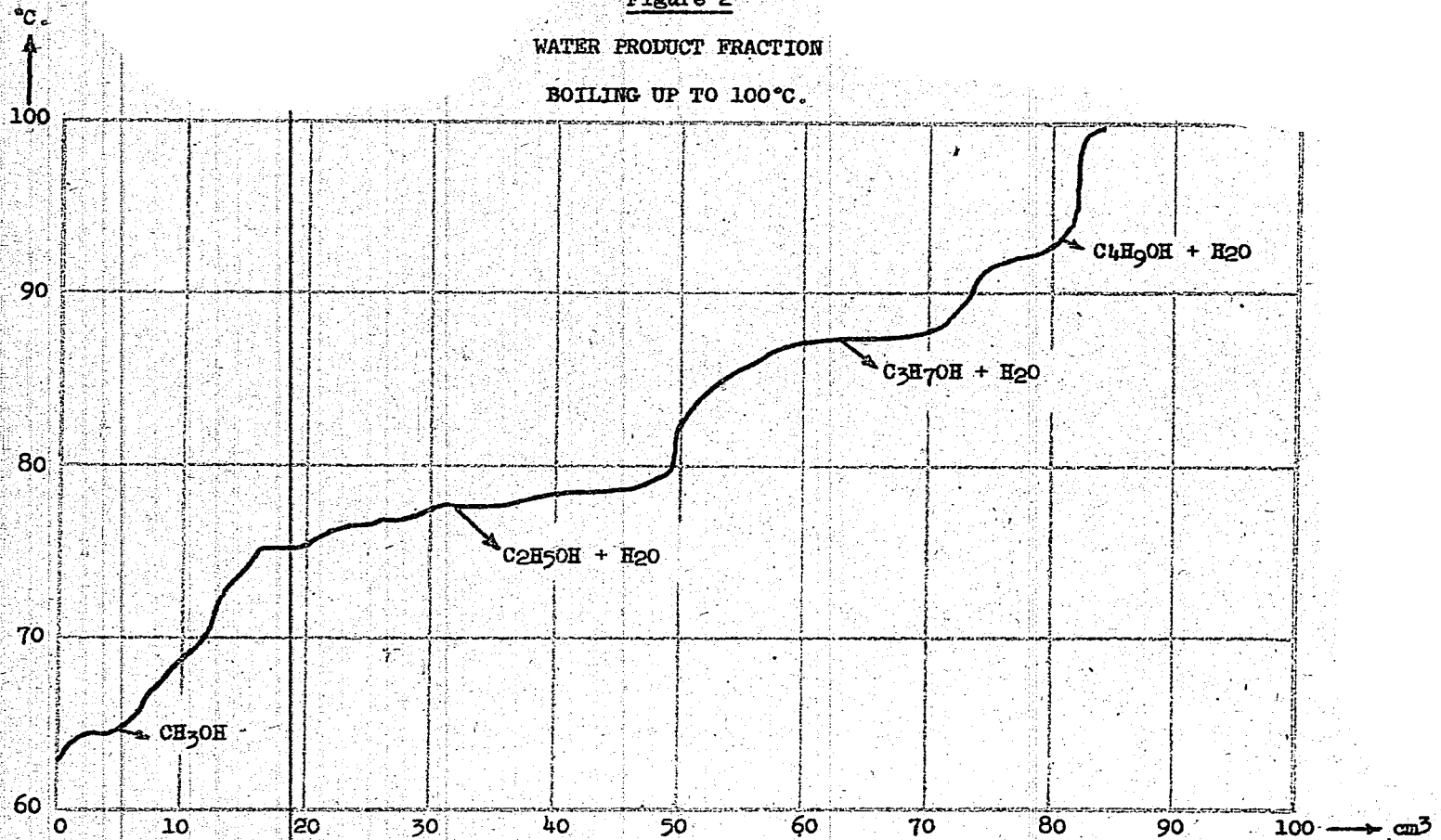


Figure 2

WATER PRODUCT FRACTION

BOILING UP TO 100°C.



REACTION TEMPERATURE AND YIELD OF LIQUID AND LIQUIFIABLE HYDROCARBONS
AND WATER PRODUCTS AGAINST THE VARIATION IN THE COMPOSITION OF THE SYNTHESIS GAS

