

cases, only the broad claim and the claim for the alcohol found to be inoperative would be invalidated, but the specific claims for the other alcohols would still stand.

Claims for a new product must define the product by its properties, and not by the process of making it.

The inventor must file an oath with his application, stating that he believes himself to be the first inventor, etc.

While the law does not require an inventor to apply for a patent within any specified time, it is for his own interest to file as promptly as possible. Delay will afford opportunity for a second inventor to file ahead of the first inventor, and if publication or public use occurs two years before filing he cannot secure a patent.

The Patent Office then makes an examination of the application and searches their files of U. S. patents and foreign patents and also their library for publications bearing on the subject. These are cited to the applicant with the examiner's reasons for rejecting the claims. The first action by the Patent Office is almost invariably a rejection.

The applicant or his attorney then files amendments to put the claims in condition to avoid the references cited and to meet the ideas of the examiner as far as is consistent with the applicant's idea of the invention. No new matter, however, can be inserted by amendment after an application is once filed.

After one or more rejections and amendments the application will either be allowed or finally rejected. In this action the examiner is virtually the opposing attorney, acting in the interest of the public, and also the judge and jury.

If the applicant does not respond

to an action of the Patent Office within six months, his application becomes automatically abandoned. It may, however, be revived under certain conditions.

If after allowable claims are found the examiner finds another application or patent with allowable claims covering the same matter, an interference may be declared, after which it is necessary to determine which inventor first conceived the idea and reduced it to practice. This involves intricate legal procedure, taking of testimony, often in several different cities, etc., and may cause great delay in issuing a patent, and also great expense to the applicant.

Appeals may be taken from the action of the Primary Examiner or the Examiner of Interferences, first to the Board of Appeals and then to the courts.

If, after issue, a patent is found defective, through inadvertence, accident, or mistake, it may be corrected by a so-called "reissue" which then replaces the original patent but expires on the same date as the original patent would have expired.

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A patentee may assign his whole interest or a part of same to an assignee, and the owner of the patent may issue licenses of various kinds. These may be exclusive or non-exclusive, may be limited to a certain territory, or a certain period, or for use in a certain field, or in other ways. They may or may not involve the payment of royalty which may be in a lump sum or proportionate to the use of the patent.

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the use, or they might cover the sale but not the manufacture, etc. The license may or may not include the right to sue infringers and retain any recoveries. These matters are all covered in the license contract.

Infringement

When the owner of a patent learns or suspects that his patent is being infringed, the usual procedure is first to serve formal notice on the infringer, telling him to cease infringing. Failure to do so will be followed by suing the infringer in court. The suit must be in a United States District court and will be for an injunction to restrain the infringer from continuing his practice and usually for an accounting of the profits derived from the infringements, and sometimes for triple this amount in damages. Such litigation, especially if carried through the Circuit Court of Appeals, may be extremely costly and for this reason a patentee of small financial means is seriously handicapped in fighting a powerful infringer unless his patent is of great importance and value so that he can secure financial assistance.

If the patented article is marked with the number and date of the patent, damages are collectible from the beginning of the infringement, but if not so marked damages can usually be collected only from the date of serving notice.

This discussion is not intended to be complete, but only to state some of the more important matters of interest to a chemist, and it should be borne in mind that exceptions occur to all rules, and this is especially true in patent matters. The advice of an experienced attorney is indispensable to a would-be patentee.

ELECTROLYSIS OF SOAP SOLUTIONS

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Introduction

IN the search for raw materials for preparation of useful sulfated products, an effort was made to obtain high molecular alcohols and olefines by electrolysis of soap solutions.

Upon passage of an electric current through a solution of a salt of a carboxylic acid, hydrogen ions are reduced to hydrogen at the

cathode, while anions of the salt are oxidized at the anode, yielding carbon dioxide and a mixture of organic oxidation products, including alcohols, olefines and saturated hydrocarbons. Various theories for the mechanisms of the reactions at the anode have been proposed.¹ Since a definite decision between these suggested mechanisms is not possible, the simplest

equations which represent the total cell reactions are used as bases for calculations in this investigation.

- (1) $2 RCH_2CH_2COOK + 2 H_2O + 2 F = RCH_2CH_2 \cdot CH_2CH_2R + 2 KHCO_3 + H_2$
- (2) $RCH_2CH_2COOK + H_2O + 2 F = RCH_2CH_2OH + KHCO_3 + H_2$
- (3) $RCH_2CH_2COOK + H_2O + 2 F = RCH = CH_2 + KHCO_3 + H_2$

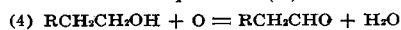
Equations (1), (2) and (3) show the main cell reactions. Equation (1) represents the familiar

Kolbe Reaction, i.e., formation of hydrocarbons of the type R-R, by union of residues from two molecules of the salt, RCOOK. (In the equations RCH_2CH_2 is used instead of the usual R in order that R shall have the same significance in all equations as in equation (3)). H. Kolbe² discovered this reaction in 1849 when he attempted to isolate the free methyl radical in the gases liberated during electrolysis of alkali acetates, but found instead that the chief constituents of the anode gases were ethane and carbon dioxide. Methyl acetate, ethylene, formaldehyde and other compounds were identified as minor constituents in the products. Salts of other low or high molecular carboxylic acids gave similar results.

Equation (2) represents formation of the alcohol, containing one less carbon atom than the original salt. These alcohols are formed by oxidation, not reduction, of the soap anions. Hofer and Moest are usually credited with the discovery that alcohols can be prepared by electrolysis of salts of carboxylic acids. Some earlier investigators had found small amounts of alcohols in products of electrolysis of such salts,³ others had sought but failed to detect alcohols.⁴ Hofer and Moest⁵ in 1902 reported that methyl alcohol was obtained as the main product from electrolysis of alkali acetates if appreciable amounts of inorganic salts, particularly bicarbonates or perchlorates, were present in the solutions. They pointed out that previous investigators must have obtained appreciable amounts of alcohols since bicarbonates formed during the electrolyses would lead to formation of the alcohols. Alcohols were obtained by Hofer and co-workers from salts of other low molecular carboxylic acids, but based on results of their work, it has been stated that in the presence of these inorganic salts, the lower aliphatic acids yield as the main product the primary alcohol, while as the molecular weight increases the per cent of alcohol formed decreases till the reaction practically stops at valeric acid.⁶

Equation (3) shows formation of the olefines, which like the alcohols contain one less carbon atom than the original salt. Thus both the alcohols and the olefines from ordinary soaps contain an odd number of carbon atoms in the molecule. Small amounts of aldehydes obtained are assumed to be

formed by oxidation of the alcohol as shown in equation (4).



Since only about 1 to 5 per cent aldehydes were usually obtained in this study, it is unimportant for present purposes whether or not this assumption is true. It will be noted that in each reaction shown in equations (1) to (3) inclusive, upon passage of two Faradays of electricity, one mole of oxidation product and one of hydrogen are formed. The $KHCO_3$ shown in the equations is formed by union of potassium hydroxide formed at the cathode with the carbon dioxide liberated at the anode, unless acid is added periodically during the electrolyses.

With the exception of the patent based on the present investigation,⁹ no attempt to prepare high molecular alcohols by electrolysis of soap solutions has been reported. Most investigators have electrolyzed soap solutions only in order to obtain high molecular hydrocarbons by the Kolbe Reaction, and have merely mentioned other products as troublesome impurities. In the most thorough study of electrolysis of soap solutions reported, J. Petersen⁷ obtained mainly hydrocarbons of the type R-R, formed by union of residues from two molecules of soap by the Kolbe Reaction. No olefine was obtained from saturated soaps and alcohols were not mentioned, although this work was reported four years after Hofer and Moest reported good yields of alcohols from electrolysis of salts of low molecular carboxylic acids.

The most definite evidence for formation of high molecular alcohols in the early literature was the identification by Petersen⁸ of unsaturated alcohols in products of electrolysis of potassium undecylate or of potassium oleate. However, it was stated definitely that these alcohols were minor constituents, and that the main products were diolefines of the type R-R, formed by the Kolbe Reaction. Olefines of the type shown in equation (3), i.e., those containing one less carbon atom than the original soap, were obtained in small amounts also.

Experimental Method

In the present work, soaps were electrolyzed under various conditions. Unless otherwise stated, the following conditions were used: (a) potassium soaps of hydrogenated

coconut oil were used, (b) temperatures of solutions were maintained at 50° C., (c) reflux condensers were used to prevent evaporation of low molecular alcohols, (d) the solutions were stirred, (e) diaphragms were not used, and (f) a copper coulometer was used for measuring the quantity of electricity passed through the cells.

Upon completion of the electrolyses, most of the organic oxidation products were on the surface of the soap solutions and could be separated easily. In an effort to recover all of the products, they were extracted from the aqueous-alcoholic soap solutions by means of petroleum ether. After it was found that the products had saponification values varying from about 0 to 80, the esters present in subsequent products were saponified by heating with alkali before the recovery by extraction with petroleum ether. There was some loss of the more volatile alcohols and olefines, particularly the latter, during evaporation of petroleum ether from the extracted materials.

Instead of trying to isolate the constituents from the products, the compositions were usually calculated from results of chemical analyses of the mixtures. Using equations (1) to (4) inclusive and a few simple assumptions, it is possible to calculate, to the right order of magnitude, the amounts of alcohols, olefines and aldehydes in a product from the hydroxyl, iodine and carbonyl values of the product and the characteristics of the original fatty acids.*

*The amount of olefines was calculated by the following method:

$$\begin{aligned} & \text{If } S = \text{weight of product,} \\ & I_s = \text{iodine value of product,} \\ & A = \text{grams alcohols in product, calculated from hydroxyl value,} \\ & I_A = \text{calculated iodine value of the alcohols,} \\ & D = \text{grams aldehydes in product, calculated from carbonyl value,} \\ & I_D = \text{calculated iodine value of the aldehydes,} \\ & O = \text{grams olefines in product,} \\ & I_o = \text{iodine value of the olefines,} \\ & H = \text{grams hydrocarbons of the type R-R,} \\ & I_H = \text{iodine value of these hydrocarbons.} \\ & I_s S = I_A A + I_D D + I_o O + I_H H \\ & \text{Substituting } S = (A + O + D) \text{ for } H \\ & \text{and solving for } O, \\ & O = \frac{(I_s - I_H)S + (I_H - I_A)A + (I_H - I_D)D}{(I_o - I_H)} \end{aligned}$$

Since I_A , I_o , I_D and I_H were calculated from the iodine value of the fatty acids, I_s and S were determined, and A and D were calculated from hydroxyl and carbonyl values, respectively, the amount of olefines could be estimated. While this calculation is based on the assumption that the products consist entirely of alcohols, olefines, aldehydes and hydrocarbons of the type, R-R, and that soaps of all molecular weight are oxidized at the same rate, the calculated amount of olefines are of the right order of magnitude even if the assumptions are not strictly true.

Effect of Anode Materials

The first experiments in the present work gave the key to the explanation for the failure of previous investigators to obtain appreciable amounts of alcohols from electrolyses of soap solutions. Soap solutions containing potassium bicarbonate were electrolyzed with smooth platinum and with graphite anodes under only moderately well controlled conditions. The products from platinum anodes were solid and those from graphite anodes liquid at room temperature. Yields were too small for accurate analyses, but approximate analyses showed that the products from graphite anodes contained much higher percentages of alcohols and olefines than those from platinum anodes. Table I shows the results of similar experiments under more accurately controlled conditions.

Electrolyses of Table I were carried out in cells that were similar except for the material used as anode. The first two columns are

are obtained with graphite anodes and in some cases even higher amounts. The failure of previous investigators to obtain appreciable amounts of alcohols and olefines is at least partially due to the fact that they used only smooth platinum anodes.

A number of other anode materials were tried but none gave as good results as graphite. Platinized platinum anodes gave low yields of high alcohol and olefine content, but the main anode reaction was evolution of oxygen, suggesting that the difference between results with various electrodes are related to the character of the electrode surface and are influenced by the same factors that influence oxygen overvoltage.

Effect of Inorganic Salts

Another important factor is the presence of inorganic salts in the soap solutions. Inorganic salts in general aid in two ways: (a) they lead to a higher content of alcohols

Hofer and Moest reported that it was essential to have inorganic salts present to obtain a good yield of alcohols from electrolysis of salts of carboxylic acids. In the present work, however, the presence of these salts was definitely of secondary importance compared to the composition of the anode. Even in the absence of inorganic salt, electrolysis of a 15 per cent soap solution containing 15 per cent formula No. 30 alcohol yielded a product calculated to contain 39 per cent alcohols and 18 per cent olefines. As this was the lowest yield of alcohols and olefines obtained in any experiment with graphite anodes, every inorganic salt tried was beneficial in increasing the content of alcohols + olefines in the products. Differences between salts were often small and probably not significant in some cases. However, perchlorates and chlorates were among the most efficacious while bicarbonates and chlorides were among the least. Alkalies often led to good products, but yields were low due to evolution of oxygen.

Effect of Low Molecular Alcohols

Another factor was even more influential than inorganic salt in leading to good yields, namely, the presence of low molecular alcohol in the soap solutions. Petersen⁷ used ethyl alcohol to prevent foaming of soap solutions during electrolysis. It is even more important because it leads to a higher current with a given potential and thus to greater yields based on electric energy. For example, in electrolysis of solutions containing 14 per cent soap and 5 per cent KClO₃ with graphite elec-

TABLE I.

Comparison of Graphite and Platinum Anodes in Electrolysis of Coconut Oil Soap Solution contained 14% soap*, 5% KClO₃ and 20% formula No. 30 alcohol**

Anode Material	Graphite	Smooth Platinum	Smooth Platinum
Area of face of anode in cm ²	16.5	16.5	16.5
Potential in volts	5.0	5.0	7.0
Average current in amperes	2.44	1.46	3.05
Current density, amperes/dcm. ²	14.8	8.85	18.5
Grams product per Faraday	54.7	45.0	40.8
Hydroxyl value of product	94.8	77.4	86.9
Iodine value of product	72.6	34.7	25.9
% Alcohols in product	30.4	24.8	27.9
% Olefines in product	38.5	14.3	8.7
% Alcohols + Olefines	68.9	39.1	36.6

*Saponification and iodine values of the fatty acids were 269.3 and 9.9, respectively.

**U. S. formula No. 30 alcohol is 100 gallons of 95% ethyl alcohol plus 10 gallons pure methyl alcohol.

results from two cells run in parallel; the data of the third column were obtained from another electrolysis in the cell used in obtaining the data of column two, and are included to show that the differences in the first two experiments are not due to difference in current density. While the per cent of alcohols in the product obtained from the graphite anode was not appreciably higher than percentages obtained with platinum anodes in these particular experiments, the per cent alcohols + olefines was. Under some conditions lower percentages of alcohols and olefines than those obtained in these experiments have been obtained from platinum anodes, but under no conditions tried has a product containing less than 57 per cent alcohols + olefines been obtained from graphite anodes. Under most conditions, between 70 and 80 per cent alcohols + olefines

and olefines in the products, and (b) they lead to much higher electric current with a given potential and thus to greater yields from a given amount of electric energy.

TABLE II.

Effect of Varying Concentration of Ethanol in Soap Solutions Subjected to Electrolysis.

% No. 30 Alcohol	(a) Solutions containing 2% KClO ₃			Grams Product per Faraday
	% Alcohols in Product	% Olefines in Product	% Alcohols + Olefines	
4	49	32	81	13
6	47	33	80	16
8	34	40	74	31
10	40	36	76	29
15	36	39	75	47
20	28	43	71	40
25	33	41	74	56
30	33	41	74	43
35	29	38	67	29
40	35	37	72	40
45	35	34	69	24
50	29	39	68	35
55	28	40	68	35
60	25	42	67	22
65	24	37	61	24
(b) Solutions containing 4.4% KClO ₃				
4.4	57	18	75	10
6.5	55	22	77	24
8.7	46	32	78	28
(c) Solutions containing 2% KClO ₃				
10	50	30	80	14
15	38	37	75	38
20	34	39	73	32
25	37	37	74	27
30	35	38	73	20
35	39	34	73	17

trodes at a potential of 5 volts, using the same cell successively for solutions containing 0 and 20 per cent formula No. 30 alcohol, the use of 20 per cent alcohol in the soap solution led to a 5 fold increase in current with a given potential and to a yield 26 times as great as that obtained in absence of alcohol from the same quantity of electricity. However, the solution containing no ethyl alcohol did yield a small amount of product containing 53 per cent alcohols and 23 per cent olefines.

The effect of varying the concentration of ethanol is demonstrated by the data of Table II, which were obtained by electrolyzing solutions containing 12 per cent soap and the indicated amount of formula No. 30 alcohol at 50° C., using graphite electrodes.

These data show that up to about 20 per cent ethanol, yields based on quantity of electricity increased, the per cent alcohols in the product decreased and the per cent olefines increased. As the per cent alcohol increased from 4 to 65 per cent, the per cent alcohols + olefines gradually decreased, but the yield per Faraday first increased and then decreased, with a maximum yield at about 20 per cent ethanol.

Results of electrolysis of solutions containing 15 per cent soap, 5 per cent KClO₃ and 10 per cent low molecular alcohol given in Table III show that other low molecular, monohydric alcohols gave re-

TABLE III
Electrolyses of Soap Solutions Containing Various Low Molecular Alcohols. Graphite Electrodes—Temperature 50° C.

Alcohol in Soap Solution	% Alcohols in Product	% Olefines in Product	% Alcohols + Olefines	Grams Product per Faraday
Methanol	42	37	79	8
Ethanol (No. 30)	41	36	77	32
n-Propanol	37	34	71	33
Isopropanol	35	39	74	38
n-Butanol	29	41	70	79
Monomethyl ether of ethylene glycol	45	34	79	19
Monoethyl ether of diethylene glycol	47	35	82	12

sults similar to those obtained with ethanol.

Each value in Table III is the average of two electrolyses except in case of methanol. There is a trend indicating that up to butanol, the yield increases but the per cent alcohols in the product decreases with increasing molecular weight of the alcohol used. Results of a more extensive investigation of some of these alcohols are given in Table IV.

These data show that: (a) isopropyl alcohol gives results similar to those obtained with ethanol, (b)n-butanol gives high yields of products containing only moderately high content of alcohols + ole-

TABLE IV.
Electrolyses of Soap Solutions Containing Various Alcohols with Graphite Electrodes. Solutions contained 12% soap and 2% KClO₃.

% Alcohol in Soap Solution	Temperature of Soap Solution	(a) Using isopropanol.			Grams Product per Faraday
		% Alcohols in Product	% Olefines in Product	% Alcohols + Olefines	
5	50	47	34	81	20
10	50	54	39	73	38
15	50	36	42	78	69
20	50	38	38	76	45
10	30	36	43	79	31
10	40	32	39	71	47
10	40	37	38	75	46
10	50	34	39	73	38
10	60	32	39	71	26
(b) Using n-butyl alcohol.					
2.5	50	43	37	80	19
5	50	32	42	74	83
10	50	27	43	70	79
15	50	24	44	68	61
20	50	25	46	71	51
10	30	29	40	70	60
10	40	29	40	69	61
10	40	32	39	71	52
10	50	27	43	70	79
10	60	30	39	69	78
(c) Using methyl ether of ethylene glycol.					
5	50	57	30	87	63
10	50	45	34	79	23
10	50	46	34	80	15
15	50	46	34	80	13
20	50	36	40	76	37
10	30	50	38	88	4
10	40	47	35	82	12
10	50	45	34	79	23
10	50	46	34	80	15
10	60	45	34	79	16

finer (about 70 per cent), and (c) monomethyl ether of ethylene glycol gives low yields of high content of alcohols + olefines (about

Effect of Other Conditions

Data of Table V show that the composition of the products of electrolysis is not affected greatly by

TABLE V.
Electrolysis of Soap Solutions at Various Temperatures. Each value is the average of two electrolyses of solutions containing 12% soap, 2% KClO₃ and 20% formula No. 30 alcohol with graphite electrodes.

Temperature (° C.)	Potential (volts)	Current (amperes)	% Alcohols in Product	% Olefines in Product	% Alcohols + Olefines	Grams Products per Faraday
0	11.5	0.71	29*	47*	76*	13*
10	11.5	1.00				
20	10.0	2.88	36	37	73	19
30	10.0	2.60	32	40	72	65
40	7.0	2.49	31	42	73	63
50	7.0	2.30	32	39	71	69
60	6.0	2.73	33	38	71	36
70	6.0	3.12	30	37	67	28

*Products obtained at 0° and 10° C. were combined in order to obtain enough product for accurate analysis.

changes in temperature but that higher electric currents are obtained from a given potential at the higher temperatures, and that greater yields from a given amount of electricity are obtained between 30 and 50° C. than at either higher or lower temperatures.

At 0° or 10° C. a white organic precipitate collected on the anodes and even at 20° C. some of the potassium chlorate precipitated from the solutions.

In studying the above and similar factors, six cells were used simultaneously. In order to determine

TABLE VI.
Reproducibility of Results in Different Electrolytic Cells. Solutions contained 12% soap, 2% KClO₃ and 20% formula No. 30 alcohol.

Cell No.	% Aldehydes in Product	% Alcohols in Product	% Olefines in Product	Grams Product per Faraday
1	2.4	36.8	36.8	74.8
2	2.4	38.8	35.4	47.2
3	2.9	38.8	34.7	47.5
4	3.1	39.2	35.0	70.8
5	3.5	38.2	35.2	43.1
6	3.5	35.9	35.3	38.7
Average	3.0	38.0	35.5	53.7

whether differences found were attributable largely to differences in behavior of the cells, identical solutions were electrolyzed with graphite electrodes at 7 volts and 50° C. with the results given in Table VI.

Obviously, the differences in composition of products from the various cells were small; in fact, the agreement was better than sometimes obtained in successive electrolyses of similar solutions in the same cell. Differences in yield were large, but yields were of secondary importance to composition of the products at the time this work was done. It is known that better yields are obtainable in a cell of better design.

Efforts to determine more than one decomposition potential were unsuccessful. When the applied potential was gradually increased from zero and the current noted 30 seconds after each successive increase in potential, there was a sharp increase in current with a small increase in potential at a little above 2 volts. If the solutions contained ethyl alcohol, the current-potential curve was practically a straight line above this decomposition potential, the slope of the curve being greater the higher the temperature. In absence of low molecular alcohol, the current continued to rise only until a potential of 3 or 3.5 volts was reached and then decreased slightly while the

potential increased to high values, perhaps due to collection of fatty acids on the anodes. Analysis of products from electrolyses at various potentials gave no indication that decomposition potentials could be obtained by this method or that composition could be varied appreciably by choice of potentials.

When a diaphragm was used to separate anode and cathode compartments, no organic products of electrolyses were found in the cathode compartment and the products from the anode compartment were practically the same as those obtained from cells without diaphragms. The material used as cathode had no effect on the composition of the products. The following factors had only minor effect on the composition of the products compared to the effect of the material used as anode: current density, concentration of soap, concentration and kind of inorganic salt, mixtures of salts or salts and alkali, ratio of soap to salt, periodic addition of fatty acids, soap, mineral acid or other material, maintaining a constant pH at various values between 6.5 and 11.0, design of cell, etc. Soaps other than coconut oil soap, such as those from pure lauric acid, pure myristic acid, commercial stearic acid, commercial red oil, etc., also yielded products calculated to contain 70 to 80 per cent alcohols + olefines of one less car-

bon atom than the original soap. (Unsaponifiable material was extracted from the red oil soap before the electrolyses.)

The fact that soaps from individual fatty acids gave about the same results as the soaps from commercial oils indicates that the assumption made in the calculations is not greatly in error.

Summary

It is not thought that the electrolytic reactions are simple, nor is it thought that the types of compounds discussed are the only ones in the products of electrolysis. Although the calculated compositions are not precise, they are of the right order of magnitude, and good yields of products consisting mainly of alcohols and olefines are obtained by electrolyzing with graphite anodes a soap solution, preferably containing appreciable amounts of an inorganic salt and a low molecular alcohol.

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REPORT OF THE GLYCERIN ANALYSIS COMMITTEE

FOR some time the Glycerin Analysis Committee has been inactive. It is now proposed to reopen the subject with a survey of existing methods of glycerin analysis, comprehensive enough to include:

1. Determination of the glycerin yield of fatty oils.
2. Analysis of lyes, sweet waters, etc.
3. Analysis of crudes and intermediates.
4. Analysis of finished glycerins,

including detection and estimation of impurities.

Such a program obviously will require several years for its completion. Fortunately we have as a basis the excellent work of the International Committee whose methods have stood the test of daily use for nearly a quarter century. Most of these methods can be adopted with little or no change whatever.

During the coming year it is proposed to confine our work to a study of the dichromate process and the

determination of the glycerin yield of fatty oils. Suggestions and assistance from anyone interested will be very welcome.

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