control sample rather than by diluting the color of the unknown to match the color of the control sample.

The preparation of tubes containing the color developed by known amounts of free sulphur in cottonseed oil (control samples) is relatively simple and can be carried out along with the sample being tested. Usually this is done by dissolving a known amount of sulphur in cottonseed oil to approximately the free sulphur concentration in the earth, using 10 c.c. of this solution, adding 2 c.c. carbon bisulphide and 2 drops of pyridine in the 25 x 300 m.m. tube and heating in the water bath.

After heating, the solutions are made up to 50 c.c. in Nessler tubes with carbon tetrachloride for the color comparison. A blank is run on the reagents using the same procedure as on the sample under test but eliminating the earth. Since the color developed in this test is affected by light and heat it is advisable to make up known color standards at the time the sample is being tested and also to avoid exposure to sunlight. This test will detect 0.001 to 0.0005 per cent free sulphur in a bleaching earth. A similar procedure may be used in determining the amount of free sulphur in bleaching carbons.

In carrying out this test it is necessary to prevent loss of material during evaporation or boiling. Should a tube ignite during the manipulation the test must be rejected because carbon bisulphide forms sulphur on ignition in the air. The extracted earth or carbon containing carbon bisulphide can spontaneously ignite in coming in contact with the air. Due care should be exercised in the disposal of these residues. It is not advisable to boil for over one hour in the steam bath. Prolonged heating has a tendency to break down the carbon bisulphide and give positive tests.

TABLE I

Some Typical Results Obtained on the Free Sulphur Content of Several Bleaching Earths and Carbons

Description	Coloration
1. Control sample	Straw
2. Standard containing 5 parts per million of free sulphur	Slight wine-red
3. Standard containing 10 parts per million of free sulphur	Wine-red
4. Standard containing 20 parts per million of free sulphur	Increasing in intensity to a strong wine-red color.
5. Standard containing 50 parts per million of free sulphur	
6. Standard containing 80 parts per million of free sulphur	
7. Standard containing 100 parts per million of free sulphur	
8. A.O.C.S. standard bleaching earth (Official Fullers Earth 1940-41)	Straw (less than 5 parts per million of free sulphur)
9. Domestic bleaching earth "A"	Straw (less than 5 parts per million of free sulphur)
10. Domestic bleaching earth "B"	Very strong wine—red (200 parts per million of free sulphur)
11. Domestic bleaching carbon "A"	Straw (less than 5 parts per million of free sulphur)
12. Domestic bleaching carbon "B"	Very strong wine—red (400 parts per million of free sulphur)

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Report of the Cooperative Work of the International Fat Commission, 1938-1939—Part II*

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SOAP ANALYSIS

(Part B of the work program)

I. Determination of Rosin Acids

The method of the *French Delegation* whose procedure follows was to be tested:

Necessary reagents: 98-99% methyl alcohol, containing about 10 g. per 1000 cc. of sulfuric acid of 66° Be'.

$\frac{N}{5}$ alcoholic potash.

Procedure: For the investigation about 2 g. (designated as "p") of fatty acid, containing rosin acids is accurately weighed into a 150 cc. ground joint flask. Exactly 20 cc. of the methyl alcohol solution of sulfuric acid is added and the solution refluxed for about 30 minutes. The solution is allowed to cool and the free acid titrated with standard $\frac{N}{5}$ alcoholic potash in the presence of phenolphthalein. Let "N" be the volume of potash used. As a blank, also accurately determined, exactly 20 cc. of the methyl alcohol solution of sulfuric acid is heated as in the test.

Let "N'" be the volume of potash used in the blank. The per cent of rosin is given by:

$$\frac{(N - N') \times 6.6}{p} - 1.6$$

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* This is the second section of this report, the first part of which was published in OIL & SOAP, February, 1942. In Part II, the names of the investigators and their comments have been omitted, except for the remarks on the Determination of Free Alkali, as adding little or no value to the article.

The results obtained by the separate investigators are compiled in the following table:

TABLE I

PERCENTAGE CONTENT OF ROSIN ACID OBTAINED

	The distributed test (Theoretical: 22.4%)	An independent test		
A. Denmark				
1.	22.8			
2.	23.2			
B. Germany				
1.	22.3			
2.	......	9.33 (Theoretical 10%)		
3.	22.7			
4.	21.8			
C. France				
1.	22.5			
2.	22.45			
D. Italy		D. McNicoll's Test		
1.	22.85	23.00		
2.	23.45	22.37		
3.	21.95	....		
		Theory	Found	Found ×1.08
E. Norway	21.6	12.3	11.2	12.1
		19.25	17.9	19.3
		18.1	16.7	18.0
		D. McNicoll's Test		
F. Switzerland	25.75	22.4		

II. Determination of Chloride

To be checked were

a) The International Commission method.

b) One Swiss method.

c) Two English methods (1 & 2).

The procedures of the methods are given below:

a) International Commission Method:

Weigh about 10 g. of test soap in a porcelain dish and carefully ash it. The ash is taken up in warm water and the chloride content determined according to one of the following described analytical methods of inorganic chemistry: Titration in neutral or slightly alkaline medium with $\frac{N}{10}$ silver nitrate solution in presence of neutral potassium chromate until the development of the red color of silver chromate (Mohr's method) or precipitation of the chloride with silver nitrate solution in an acid medium (nitric acid) and titration with a potassium thiocyanate solution (Ferric ammonium alum as an indicator). The nitric acid used must be chloride free and should be heated for removal of nitrous acid (Volhard's method).

If "p" is the weight of the soap sample and "n" the number of cc. of $\frac{N}{10}$ silver nitrate solution, one may calculate

$$\%\ \text{chloride} = \frac{0.585\text{n}}{\text{p}}\ \text{as sodium chloride}$$

$$\%\ \text{chloride} = \frac{0.745\text{n}}{\text{p}}\ \text{as potassium chloride.}$$

b) Swiss Method:

Five g. of soap are dissolved in about 50 cc. of hot water, transferred to a 200 cc. graduated flask, and treated with 5 cc. concentrated nitric acid to separate fatty acids. Twenty-five cc. of $\frac{N}{10}$ silver nitrate solution is added immediately. By this means the fatty acids are well separated and also the precipitated silver chloride is floculated after warming a short time on a water bath. After cooling, the flask is filled to the mark and filtered; the first 40 or 50 cc. is rejected. One hundred cc. of the filtrate is backtitrated after addition of 2 to 3 cc. of ferric ammonium alum solution with $\frac{N}{10}$ thiocyanate solution.

$$1\ \text{cc.}\ \frac{N}{10}\ AgNO_3 = 0.00585\ \text{g. NaCl or}$$
$$= 0.00745\ \text{g. KCl.}$$

c) English Method (1):

Since ashing the soap gives low values, the method of the "British Soap Makers" for determination of chloride is proposed: Dissolve 10 g. of soap in hot water and transfer to a 250 cc. volumetric flask. Add 10 cc. of a 20% calcium nitrate solution with vigorous shaking, and allow to cool. Dilute to the 250 cc. mark, filter and titrate 100 cc. of the filtrate with standard silver nitrate solution, using potassium chromate as an indicator.

c) English Method (2):

The proposal of *Dr. A. B. Shepherd:* Dissolve 5 g. of soap in hot water, add an excess of chlorine free calcium nitrate solution (about 25 cc. of a 20% solution of crystaline calcium nitrate in water). Titrate, without filtering, with $\frac{N}{10}$ silver solution, using 5% potassium chromate as an indicator. If "p" is the weight of the soap sample, and "n" the number of cc. of $\frac{N}{10}$ silver nitrate solution used in the titration, the chloride content is obtained:

$$\text{Chloride} = \frac{0.585\text{n}}{\text{p}},\ \text{calculated as sodium chloride}$$

or

$$\text{Chloride} = \frac{0.745\text{n}}{\text{p}},\ \text{calculated as potassium chloride.}$$

The results of the various investigators, average value, are collected in Table II.

III. Determination of Total Fatty Acids

To be tested were

a) Dutch Method

b) I. C. Method.

The description of both methods is given below:

a) Dutch Method:

Ten g. of soap was dissolved in a porcelain dish in 100 cc. of warm distilled water and transferred to a separatory funnel. The porcelain dish is rinsed several times with a little water which is poured into the separatory funnel. About 10 cc. of sulfuric acid (1:5) is added to the soap solution in order to separate the fatty acids. After cooling the solution to room temperature, about 100 cc. of ethyl ether is added to the separatory funnel, the funnel strongly shaken for one minute and allowed to stand. The acid layer is allowed to drain into a second separatory funnel where it is again shaken with 50 cc. of ethyl ether. The combined ethereal extracts are washed twice with 50 cc. of 10 per cent sodium chloride each

TABLE II

	International Commission Method	Swiss Method	English Method 1	English Method 2 (Shepherd)
A. Germany				
1. Theory	91.60	45.80	92.55	46.28 mg. NaCl
Found	91.31	45.55	95.35	47.99 mg. NaCl
2. Theory	0.174	0.230	0.357	0.450% NaCl
Found	....	....	....	....
3. Theory	....	....	....	....
Found	0.257	0.246	0.261	0.295% Cl
4. Theory	1.0	1.0	1.0	1.0 % NaCl
Found	0.98	0.99	1.03	1.06 % NaCl
B. France				
1. Theory	0.795	0.795	0.795	........% NaCl
Found	0.787	0.805	0.809	........% NaCl
2. Theory	1.00	1.00	1.00	........% NaCl
Found	1.01	0.96	1.07	........% NaCl
D. Italy				
1. Theory	0.94	0.94	0.94	0.94 % NaCl
Found	0.88	0.89	........[*]	0.99 % NaCl
2. Theory	....	....	....	....
Found	0.386	0.387	0.477	0.622% NaCl
E. Norway				
1. Theory	0.797	0.797	0.797	0.797% NaCl
Found	0.798	0.782	0.819	0.816% NaCl
F. Switzerland				
Soap I	0.60	0.61 and 0.58	....	0.47 and 0.62 % NaCl
Soap II	0.40[+]	0.73 and 0.70	....	0.88 and 0.90 % NaCl

[+] Tests on Soap II were *intentionally* done without taking usual care consequently the results are low.

[*] Not determinable.

time to remove mineral acid and filtered through a dense filter into a tared flask. The ether is evaporated almost completely at low temperatures and the residue dissolved in about 20 cc. neutralized alcohol. The alcoholic solution is then neutralized with N/2 alcoholic potassium hydroxide in presence of phenolphthalein, the alcohol driven off and the residual soap dried in a drying oven to constant weight. The weight of potassium (K-1) is subtracted from the weight obtained and thus the weight of fatty acids in the soap sample is obtained.

b) I. C. Method:

About 10 g. of soap are placed in a 250 cc. flask; 100-150 cc. of distilled water and 10 cc. of sulfuric acid (diluted 1:5) are added and the whole heated with occasional shaking on a sand bath (methyl orange as an indicator) until the fatty acids have separated on the surface of the liquid. Care must be taken that no soap adheres to the wall of the flask. After cooling the solution is transferred to a separatory funnel, the flask is washed out once with a little alcohol or acetone, then with 100 cc. of ethyl ether in small portions, all of which is carefully added to the separatory funnel. The funnel is shaken strongly for one minute and allowed to stand. The water layer is transferred to a second separatory funnel and shaken again with 50 cc. of ethyl ether. The combined ethereal extracts are washed twice with 10 per cent sodium chloride solution to remove mineral acids (the maximum amount which shall be used for washing shall not exceed 100 cc.), and filtered through a dense filter into a tared flask (after preliminary drying with sodium sulfate if necessary). The ether is evaporated and the flask dried to constant weight in the usual manner and weighed. The weight obtained is referred to 100 g. of substance.

The results (average values) obtained by the various experimenters are compiled in the following table:

TABLE III

FOUND PER CENT FATTY ACIDS BY THE

	I. C. Method	Dutch Method
A. Germany	Coconut Oil Soap	
1.	38.21 (1 hr. at 100°) 38.33 (30 min.+15 min. at 60°)	38.45 (2nd[+] hr. at 100°)
	Olein Soap	
	27.26 (1 hr. at 100°)	27.20 (5 hr. at 100°)
	Soap with 35 Per Cent Coconut Oil Fatty Acids in Total Fats	
	38.43 (1 hr. at 100°)	38.45 (3 hrs. at 100°)
2.	64.7	64.4
3.	65.17	65.06
4.	49.76	49.78
B. France		
1. Theory	....	6.520 and 7.183
Found	....	6.467 and 7.152
C. Italy		
1.	75.28	77.56
2.	64.13	68.47
D. Norway		
Theory	64.28	64.28
Found	64.29 (after eight 15-min. drying periods)	64.19 (after 20 min. at 120°)
E. Switzerland	69.1	69.1

[+] Translators' note: "s Std." is here translated "2nd hr." It is possible that this "s" is a typographical error and should have been some number.

IV. Determination of Free Alkali

To be tested were:

a) A French Method

b) An Italian Method.

The text of both methods is given below:

a) French Method:

Ten g. of soap is dissolved on a boiling water bath or over a small flame with constant stirring in 100 cc. of 80 per cent neutralized alcohol to which a known amount[1] of fatty acids of known molecular weight has been added.

For highly refined soap (72 per cent) 1 g. of fatty acid is generally sufficient, corresponding to about 3.5 cc. of a normal solution. After solution takes place, the uncombined fatty acid is back titrated. If n' is the number of cc. used and n the number of cc. needed to titrate the added amount of fatty acid, the total free alkali is calculated as $(n-n')$. The determination can also be made on soaps containing sodium hyposulfite and calcium carbonate.

b) Italian Method:

Qualitative Test: Let a drop of alcoholic phenolphthalein solution fall on a freshly cut surface of the soap to be tested; a more or less distinct red color shows the presence of free alkali. The test can also be made by dissolving a small piece of soap in absolute alcohol, warming the solution and adding one drop of phenolphthalein solution.

Quantitative Determination: Ten to twelve g. of soap is dissolved in a 400 cc. beaker in 150 cc. of neutralized 95 per cent alcohol by heating on a sand

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[1] For a series of experiments a freshly prepared alcoholic fatty acid solution of known titer can be used.

bath. The solution is filtered at 60° through a hot-water filter and the filter washed with 95 per cent alcohol. The solution is allowed to cool to 50° and then titrated with standard $\frac{n}{2}$ acetic acid or any other appropriate organic acid (using phenolphthalein as an indicator). If n is the number of cc. of $\frac{n}{2}$ acetic acid used, and p the weight of the sample then:

$$\frac{(n)(0.0155)(100)}{p} = \text{percent; calculated as } Na_2O.$$

and

$$\frac{(n)(0.02335)(100)}{p} = \text{percent; calculated as } K_2O.$$

Remarks

A. Germany

The French method gives satisfactory results. It will consequently be adopted. The title should be completed by the note which is made here, that the determination includes the caustics and alkali carbonates.

Research has shown that for reaction of the free alkali with the fatty acid, a four hour heating is needed. The text is correspondingly changed.

The Italian method of determination of caustic alkali gives very inaccurate values, since a varied amount of alkali is retained by the filter. The method should therefore be rejected.

B. France

Concerning the *Italian method* it is noticed: Since a part of the alkali carbonate remains on the filter, only a part of the true value is obtained. However, the method is useful, especially if only a small amount of alkali carbonate (about 0.1 per cent) is contained in the soap, as is the case, for example, in well refined soap. Moreover, it is emphasized that sodium carbonate is by no means insoluble in alcohol which has been diluted by the water in the soap.

Inasmuch as free caustic alkali is to be determined, sufficiently accurate values are obtained by the help of this method, with the assumption that the soap to be tested contains no unsaponified fat, which is usually the case.

Consequently, it follows that on the instant when the soap is dissolved in alcohol a part of the free caustic alkali is used up in saponifying the unsaponified fat and so escapes determination. Summarizing, it must be said that there is no method by which the free caustic alkali can be determined with absolute accuracy. Apparently the best one can accomplish is the determination of caustic alkali as the difference between total alkali and free alkali carbonates.

C. Holland

Research has shown that the *French method* gives excellent values by addition of a known amount of stearic acid. The *Italian method,* on the other hand, gives no consistent results. Apparently a part of the free alkali, because of the proportionately long lapse of time which is used by solution and filtration, is converted into carbonate. The *French method* is preferred.

D. Italy

It is determined by all our research that the values obtained by the *French method* are too high, while the *Italian method* fails to work only at a content of free alkali under 0.01 per cent; in all other cases its applicability is very good in comparison.

E. Norway

The following values were obtained in the comparative tests:

TABLE IV

French Method			Italian Method		
Theory	Found	Difference	Theory	Found	Difference
1.50	1.43	—0.07	3.49	3.19	—0.30
3.64	3.68	+0.04	1.83	1.67	—0.16
5.28	5.21	—0.07	1.30	1.15	—0.15

The deviation in the *French method* appears to be due to pure errors in determination. The *Italian method* appears to be less reliable in contrast with the *French method* and gives not entirely satisfactory results.

F. Switzerland

The *French method* gives excellent results when it is a question of measuring the sum of sodium hydroxide and sodium carbonate. However, when it is applied to differentiation it fails to work. The *Italian method* is uncertain and gives too low results, which is especially unpleasant when working with low amounts of free alkali. The filter retains the alkali: a soap solution reddened with phenolphthalein can be decolorized by filtration!

TABLE V

	Calculated	French Method	Italian Method
Soap alone	0.00%	0.00% Na_2O	0.00% Na_2O
Soap + KOH	0.16%	0.18% Na_2O	0.09% Na_2O
Soap + KOH	0.48%	0.51% Na_2O	0.34% Na_2O
Soap + KOH + Na_2CO_3	0.48% 1.09% respectively	1.10% Na_2O	0.36% Na_2O

We give below the methods for determination of free alkali which can be used with advantage and which have shown good use as controls with the *French method.*

Determination of Free Alkali in Sodium Soaps (Solid and Liquid Soaps)

Free NaOH: Two g. of soap is weighed in a 250 cc. flat bottom flask equipped with a reflux condenser and dissolved by heating with 100 cc. neutral 95 per cent alcohol. (The alcohol must be previously neutralized with $\frac{N}{2}$ KOH towards phenolphthalein until a strong pink color is developed.) After complete solution, the soap solution is rapidly cooled and titrated with $\frac{N}{10}$ alcoholic hydrochloric acid. (The end point must be that of the alcohol originally used.)

Per cent NaOH = (0.2)(n); where n = cc. $\frac{N}{10}$ HCl

Free Sodium Carbonate: Sixty cc. of neutral (phenolphthalein) distilled water is added to the above titrated solution, and the whole titrated again with $\frac{N}{10}$ alcoholic HCl to the same end point as the distilled water.

Per cent Na_2CO_3=(0.53)(n); where n=cc. $\frac{N}{10}$ HCl.

Determination of Free Alkali in Potash Soaps (Soft Soaps)

Free KOH: Two g. of soap is weighed into a 500 cc. wide mouth Erlenmeyer, 200 cc. of 60 volume per cent neutralized alcohol is added, and the whole warmed on a steam bath until completely dissolved. Ten cc. of a neutral 10 per cent barium chloride solution is then added and the whole shaken well and rapidly cooled by immersion in cold water. The solution is then titrated with $\frac{N}{10}$ HCl in presence of phenolphthalein.

Per cent KOH = (0.28)(n); where n = cc. $\frac{N}{10}$ HCl.

Free Potassium Carbonate: The free potash is not directly titrated. On the contrary, the total free alkali is determined and the free potassium carbonate calculated by difference. Two g. of soap is weighed into a 500 cc. Erlenmeyer flask; 200 cc. of 60 volume per cent alcohol, neutralized as before, is added and the flask and contents are heated on the steam bath until complete solution. After rapid cooling, the solution is titrated with $\frac{N}{10}$ HCl, with phenolphthalein as an indicator to the same light pink color as the alcohol originally used. (One drop of $\frac{N}{10}$ HCl in excess should cause the light pink color to disappear.)

Total cc. $\frac{n}{10}$ HCl used = N

cc. $\frac{n}{10}$ HCl used for KOH = n

cc. $\frac{n}{10}$ HCl used for K_2CO_3 = N-n

Per cent K_2CO_3 = 0.69 (N-n).

Abstracts

Oils and Fats

Edited by
M. M. PISKUR and SARAH HICKS

THE NUTRITIONAL VALUES OF SHORTENING. *Bakers Digest 16*, 163-4 (1942). Generally speaking, both animal and vegetable fats and oils are of equal value in the human diet. This value, which is of the highest order, is determined by the following five major factors: (1) digestibility of the fats; (2) their content of essential fatty acids; (3) their vitamin and protein sparing action; (4) their melting points in their relation to fat utilization; and (5) their content of fat-soluble vitamins.

STUDIES ON HIGH MOLECULAR WT. ALIPHATIC AMINES AND THEIR SALTS. V. SOLUBLE AND INSOLUBLE FILMS OF THE AMINE HYDROCHLORIDES. E. J. Hoffman, G. E. Boyd, and A. W. Ralston. *J. Am. Chem. Soc. 64*, 498-503 (1942). Insoluble monolayers of octadecylamine hydrochloride spread on various sub-solns. have been investigated by means of the film balance. An increase in temp. causes octadecylamine hydrochloride films to become more expanded. The heat of spreading at an area of 35 A^2 per molecule was estimated to be 320 ergs cm.—^{2}. This value was compared with values for other insoluble films possessing different polar groups. The behavior of oxygen acid salts of octadecylamine has been studied.

MILK FATS FROM COWS FED ON FRESH PASTURE AND ENSILED GREEN FODDER. I. OBSERVATIONS ON THE COMPONENT FATTY ACIDS. T. P. Hilditch and H. Jasperson. *J. Soc. Chem. Ind. 60*, 305-10 (1941). The object of work reported in the present communication was to compare the component fatty acids of milk fat from cows fed solely on silage and hay during the winter season with that of milk fat from cows fed on fresh summer pasture grass. It has been found that the milk fat from the silage-fed cows closely resembles in composition that reported in earlier studies of cow milk fats from animals fed on winter diets of the usual type constg. of hay, roots, and farm-grown concentrates. The characteristic differences observed in the fat of milk from cows on summer and winter diets can not therefore be directly connected with change in the components of their feed. The possible mode of production of milk fat is further discussed in the light of the present results. Some further information has been sought as to the nature of the at present somewhat indefinitely characterized polyethenoid C_{18} acids of cow milk fats.

THE LIPIDS OF THE DUODENAL MUCOSA OF SWINE DURING THE ABSORPTION OF FAT. Raymond Reiser. *J. Biol. Chem. 143*, 109-14 (1942). There is no change in the phospholipid or cholesterol content of the duodenal mucosa of swine during absorption. There are no triglycerides in the fasting duodenal mucosa of swine and only small amts., if any, during absorption. About 2.5% of the dried wt. of fasting mucosa is free fatty acid and this amt. is approx. doubled 5 hrs. after the ingestion of oil. A theory is presented to explain the apparent differences presented by the histological and chemical studies of the absorbing mucosa.

THE INFLUENCE OF DIETARY FAT ON LACTATION PERFORMANCE IN RATS. L. A. Maynard and Edith Rasmussen. *J. Nutrition 23*, 385-98 (1942). Paired-feeding studies, involving equalized calorie intakes, are reported in which lactation performance was measured by the growth of standardized litters and by their composition. In one experiment a diet of natural foods containing approximately 4.5% fat was compared with a similar diet containing approximately 9% fat. The young from the mothers on the high-fat diet made better growth and contained more dry matter and fat in thirteen out of fifteen paired comparisons. Similar results were obtained for protein and calorie content. In a second experiment in which purified diets containing 0.3% and 18% fat were compared the data for the high-fat diet indicated a superiority in growth and in dry matter content in ten out of twelve comparisons, in fat and calorie content in all cases, and in protein in six out of nine cases. With the limited feeding practiced, the better lactation performance on the high fat diet