of fat in the diet used for the treatment. Hanssen cited investigations of others which proved that obesity resulted from an increased transformation of carbohydrates into fat. owing to an anomaly of metabolism. His patients were given a diet that provided generous amounts of greens, some fruit, 100-125 grams of meat or fish, 100 grams of bread, 65 grams of cream, 65 grams of butter, 35 grams of cheese, 2 eggs and 25 grams of olive oil. He compared the results with those obtained in other hospitals and found that his patients lost the same amount of weight with an intake of 1,850 calories as was the case in other hospitals in which the intake of patients under treatment for obesity was only 950 calories.

[Editor's Note: Due to the length of this paper, it has been divided into two sections. Second section will appear in the April issue.]

THE ANALYSIS OF SULFONATED (SULFATED) OILS COMMITTEE REPORT*

Committee Report, No. 3 Determination of Inorganic Salts in Sulfonated Oils

By RALPH HART, Chairman

INCIDENTAL to manufacture or through admixture, commercial sulfonated oils may contain various amounts of inorganic salts, such as alkali chlorides, sulfates, and less frequently acetates, carbonates, etc. The determination of inorganic sulfate in sulfonated oils is part of the regular procedure for finding organically combined sulfuric anhydride by the barium sulfate method, but the other inorganic salts are practically always included with the "undetermined," largely because no satisfactory methods of analysis for inorganic salts have been described. For a more complete and thorough analysis of a sulfonated oil, it is desirable that the total inorganic salts be determined separately rather than grouped with the "undetermined."

Available Methods

Herbig[1] determines inorganic sulfate by dissolving the sample in ether, washing with a saturated solution of sodium chloride, and precipitating the sulfate in the wash water with barium chloride. To overcome troublesome emulsions, which highly sulfonated oils tend to form, he replaces one-third of the ether with benzene. If emulsions still persist the oil is dissolved in absolute alcohol, filtered, the residue dissolved in hot water, and the inorganic sulfate determined in the filtrate. Nishizawa[2] attributes such emulsions to the comparatively easy solubility of the normal or di-sodium salt of sulfonated oils in concentrated sodium chloride solution. On the other hand, the mono-sodium salt is practically insoluble in a saturated solution of sodium chloride. Accordingly he modifies the Herbig method by first converting the compound into the mono-sodium salt with hydrochloric acid — using methyl orange as the indicator — before washing with the salt solutions.

Burton and Robertshaw[3] determine total inorganic salts by digesting the dehydrated sample with alcohol or a mixture of alcohol and ether, filtering, dissolving the residue in hot water, and determining the solids in the filtrate by evaporation. The inorganic salts, they state, are not completely precipitated by alcohol with many sulfated oils. Some tests made in this laboratory indicate that the Burton and Robertshaw method may yield good results with sodium sulfate but it is not satisfactory with sodium chloride, using 95% alcohol and ether as the solvents. The results on a sample, in which the inorganic salts contained about 90% sodium chloride and the rest sodium sulfate, were as follows:

Solvent—	Inorganic Salts Added Per cent	Found Per cent
2 parts ethyl ether and 1 part 95% alcohol	7.84	6.32
2 parts ethyl ether and 1 part absolute alcohol	7.84	7.52

Difficulties may also be experienced with the alcohol-ether solvent in the case of dehydrated oils containing at the same time large amounts of fatty glycerides or mineral oils and neutral soap, since the former are sparsely soluble in alcohol and the latter in ether.

Wizöff or Herbig Method

In the official method of the Wissenschaftliche Zentralstelle für Öl- und Fettforschung (Wizöff),[4] a mixture of 40% of ether and 60% of amyl alcohol is used as solvent for the oil, otherwise the procedure is the same as the Herbig benzene-ether method. The procedure for this method is as follows:

Determination of Inorganically Combined Sulfuric Acid — 5 to 7 g. of oil is shaken in a separatory funnel with 10 c.c. saturated sulfate-free common salt solution, 10 c.c. ether, and 15 c.c. amyl alcohol; the clear salt solution is separated from the ether and then washed three times with 10-20 c.c. of concentrated salt solution. The combined salt solutions are made up to 25 c.c., 1 c.c. of concentrated HCl (D-15 = 1.19) is added, and the sulfuric acid estimated by precipitating at the boil with barium chloride solution in the usual way.

The A.L.C.A. Method

Another method for total inorganic salts is that of Hart.[5, 6]. This method has been used in the writer's laboratory for a number of years as a means of control during manufacture with entirely satisfactory results. According to this method, total non-volatile inorganic salts are determined indirectly from the ash, fixed alkalinity, and organically combined sulfuric anhydride in the sample. This procedure is also the official method of the American Leather Chemists Association (A.L.C.A.).[7] It yields reliable and accurate results, except in the presence of ammonium salts and sodium acetate, since the former is volatilized during the ignition and the latter changed to the carbonate. Moreover, the formulas are calculated for sodium salts and in the presence of potassium it becomes necessary to determine the metallic elements. The following are the details of this method:

Ash—Weigh accurately from 5

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*A report presented at the Fall Meeting of The American Oil Chemists' Society, at Chicago, October 8-9, 1936.

to 10 grams of the sample into a crucible or dish, ignite gently, allowing the oil to burn, and then complete the incineration at a dull red heat until all the carbon is burned off. Cool and weigh.

Calculations — The inorganic non-volatile, stable salts are given by these formulas:

Case 1—When ($A_f - 7S$) is negative,
% Inorganic Salts = % Ash — 0.1268 ($A_f + 7S$)
Case 2—When ($A_f - 7S$) is positive,
% Inorganic Salts = % Ash — 0.0946 ($A_f + 11.77S$)

Where A_f represents the fixed alkalinity, expressed in mgms. KOH per gram of sample and S, the per cent organically combined SO_3. These formulas are based on the following reactions:

(a) The sulfonated radical, in the absence of soap or other alkali, is converted upon ashing into equal parts of sodium sulfate and sulfuric anhydride, thus:

$$2\ ROSO_3Na \longrightarrow Na_2SO_4 + SO_3 + \text{etc.}$$

(b) In the presence of soap or other alkali, all of the liberated SO_3 is bound by the former, provided there is sufficient alkali present to combine with it. The reaction is as follows:

$$2\ RCOONa + SO_3 \longrightarrow Na_2SO_4 + CO_2 + \text{etc.}$$

(c) Soap or other alkali in excess of the amount necessary to combine with all the liberated SO_3 is changed upon ashing into sodium carbonate. The derivation of these formulas are given elsewhere.[5]

The A.A.T.C.C. or Direct Method for Alkali Sulfates and Chlorides

To overcome the shortcomings of the existing methods, the American Association of Textile Chemists and Colorists A.A.T.C.C. has recently published a method,[8] according to which the alkali sulfates and chlorides are quantitatively separated in the form in which they are present in the original sample. The method consists essentially of dehydrating the sample in the presence of oleic acid, dissolving the dehydrated residue in carbon tetrachloride (in which the salts are insoluble), filtering, and weighing either the dried or the ignited residue. Details of the A.A.T.C.C. procedure with some changes suggested by this committee are given at the end of this report.

Discussion of the A.A.T.C.C. Method

According to McBain and McClutchie[9] sodium oleate or stearate exhibit no sign of gelation in chloroform. In carbon tetrachloride, however, the A.A.T.C.C. Committee has found that gelation does occur unless a considerable quantity of free oleic acid is present. The ordinary sulfonated oil was found to be stable when heated as outlined in the method as long as there was some soap present, even as little as 2.0% on the weight of the sample. There was also no reaction between the sodium sulfate and the free fatty acids upon heating. This was shown by the fact that the alkalinities of a sample containing sodium sulfate were identical before and after heating. If necessary alkali may be added to stabilize the oil, which addition will have no effect on the result, provided it is not in excess of the free fatty acids.

The dried as well as the ignited residue is determined in order to detect the presence of insoluble fatty matter, ammonium salts, or incomplete washing. Ignition of the well-washed residue, even in the absence of ammonium salts, invariably causes slight charring and shows a loss of 0.1 to 0.27% based on the weight of the original sample. This loss probably represents fatty matter adsorbed by the salts which is difficult to remove even upon prolonged washing. Where the loss upon ignition is greater than 0.3% it is probably due, in the absence of ammonium salts, to incomplete washing of the residue; in which case it is necessary to repeat the analysis with more thorough washing. Evidently the error due to adsorbed fatty matter is reduced to a minimum by ignition. For this reason and the fact that the "ignited" rather than the "dried" results check more closely with the ash and barium sulfate methods, it is recommended that in the absence of ammonium salts, the "ignited" residue be reported as the final result. The residue, of course, may be analyzed for its constituents by the usual methods of inorganic analysis.

This committee has investigated the A.A.T.C.C. method and finds that the results are sufficiently reliable for commercial purposes. It was found, however, that ammonium salts during the de-hydration are partly converted into the corresponding sodium salts by reacting with the alkali of the soap. However, due to the small difference in molecular weights between ammonium and sodium salts, the maximum theoretical error in weight (in the absence of potassium compounds) is 7% to 9% above the correct values. Actually the loss is less since only partial conversion takes place. It was also found that sodium acetate in the presence of excess of free oleic acid is practically all converted into soap during the dehydration, as shown in the following test. The salts obtained from samples containing sodium acetate were ignited and the ash tested for carbonates. The amount of carbonates found by the committee was negligible—the average being well under 0.1%. Hence sodium acetate does not interfere with the determination.

Results by the A.A.T.C.C. and the Barium Sulfate Methods

Table 1, taken from the A.A.T.C.C. Committee report[8], shows the amount of inorganic salts found in various samples of sulfonated oils analyzed by the A.A.T.C.C. and by the barium sulfate or Herbig methods. All the results in this Table represent the ignited residue with the exception of Sample No. 3, which contained ammonium sulfate and hence was dried only. It will be observed that in the presence of sodium sulfate, the difference between the two methods was less than 0.1%; in the presence of ammonium sulfate, about 0.2%; and with Sample No. 4, containing both sodium chloride and sulfate, the result by the A.A.T.C.C. method was practically identical with the theoretical value.

TABLE 1

Inorganic Sulfates and Chlorides in Sulfonated Oils by the A.A.T.C.C. and the Barium Sulfate Methods

Sample No.	Description	Kind of Salt	A.A.T.C.C. Method	Barium Sulfate Method	Difference
1	Sulfonated castor oil..........	Na_2SO_4	0.83	0.79	0.04
2	Sulfonated castor oil..........	Na_2SO_4	6.64	6.56	0.08
3	Sulfonated castor oil..........	$(NH_4)_2SO_4$	8.52*	8.73	0.21
4	Sulfonated castor oil..........	NaCl Na_2SO_4	4.93‡	....	....

*Dried only. ‡Added 4.91%.

Cooperative Samples

Samples 3A to 3F inclusive were analyzed by the committee for inorganic salts by the A.A.T.C.C. method. These samples were prepared by Mr. Ralph Wechsler of the National Oil Products Company and consisted of the following:

Sample 3A:
Sulfonated castor oil, soda neutralized, and containing approximately 30% water.
Sample 3B:
1,000 grams Sample 3A
1,000 grams water
50 grams NaCl anhydrous
50 grams Na_2SO_4 anhydrous
Sample 3C:
1,000 grams Sample 3B
20 grams Sodium Acetate

Sample 3D:
  Similar to Sample 3A
Sample 3E
  2,000 grams Sample 3D
  2,000 grams water
  150 grams $(NH_4)_2SO_4$ anhydrous
Sample 3F:
  2,000 grams Sample 3E
  40 grams sodium acetate

Committee Results

The cooperative samples were analyzed by the committee in accordance with the procedures given at the end of this report and the results are listed in Table 11. Since the check results of each analysis were very good, only the averages are given in the Table. The calculated values for samples 3B and 3C were obtained from the result for Sample 3A, namely, 1.61%; and for Samples 3E and 3F, from Sample 3D, namely, 1.75%.

TABLE II

Committee Results* of Inorganic Salts in Sulfonated Oils by the A.A.T.C.C. Method.

	Inorganic Chlorides and Sulfates, Per Cent					
	In the Absence of Ammonium Salts (Residue Ignited)			In the Presence of Ammonium Salts (Residue Dried)		
Sample No.	3A	3B	3C	3D	3E	3F
Clark	2.00	5.37	5.25	....	4.53	4.57
Hart	1.41	5.42	5.36	1.59	4.58	4.52
Irwin	1.41	5.57	5.41	1.67	4.69	4.64
Long	1.51	5.38	5.50	1.9	4.8	4.6
Sheely	2.05	5.63	5.66	2.01	4.75	4.76
Tiffaney	1.53	5.45	5.37	1.64	4.59	4.52
Wechsler	1.35	5.18	5.19	1.70	4.58	4.62
Grand average	1.61	5.43	5.39	1.75	4.65	4.60
By calculation	....	5.53	5.42	....	4.45	4.36
Difference	....	0.10	0.03	....	0.20	0.24
Greatest difference	0.70	0.45	0.47	0.42	0.27	0.24
Average deviation from mean	0.24	0.10	0.11	0.13	0.09	0.06

*Averages only are given.

It will be observed that the "found" values for Samples 3B and 3C were about 0.06% below the "calculated" results—evidently well within the experimental error. On the other hand, Samples 3E and 3F were about 0.2% above the "calculated" values. This corresponds to about 5% on the weight of the residue and is undoubtedly due to the conversion of the ammonium salt into sodium salt, as already explained. The magnitude of the per cent error on the residue would be insignificant in the analysis of an ordinary sulfonated oil since the actual error would be within the experimental error of the other determinations. It will also be noted that the average of the average deviations from the mean for all of the six samples was about 0.1%—indicating a satisfactory agreement between the different analysts.

Comments and Suggestions by the Committee

Ralph Hart: We also experienced, as noted by Mr. Sheely, the slow filtration of Samples D and F, but not of the remaining samples. The former contained the original salt whereas salt was added to the others. It was noticed with the slow-filtering samples, that most of the salt was deposited in the pores of the filter paper as a continuous sheet which could be peeled off after drying; with the others the dried salt seemed to be crystaline. Apparently in the slow-filtering samples the salt is precipitated more or less in a colloidal form.

W. H. Irwin: The method as outlined seems to yield results which are quite easily duplicable. The correction for sodium carbonate, found upon ashing the residue, is very small and not of much significance in the final result.

C. P. Long: In running these determinations 10 gram samples were used, which were felt to be more desirable than the smaller sample indicated. The checks appear to be fairly good except for Sample 3A. An Argand burner with a 4-in. glass chimney was used in igniting the residues.

M. L. Sheely: In the case of Sample 3A, considerable difficulty was experienced due to the extremely slow rate of filtration. No trouble was encountered in Samples 3B and 3C.

W. H. Tiffany: We had little difficulty in the manipulation of this method and have no suggestions to offer provided our findings agree with the members of the committee. The work was conducted by Mr. Schroeder who has never practiced this method before and if the results agree with the committee samples it would speak very well for the method.

X. Wechsler-Segessemann: We would like to suggest that the procedure be made more specific in regard to the temperature of ignition. We originally ignited the residues in an electric muffle furnace at a temperature of about 930° C. At this temperature, we were unable to ignite the residues to constant weight and the results appeared to be consistently low. For the foregoing reported determinations, ignition was carried out with an ordinary Bunsen burner to a dull red heat at the bottom part of the crucible. In this manner we had no difficulty in igniting to constant weight.

While the results for the dried residues come fairly close to the calculated values, the figures for the ignited residues of 3E and 3F are several times higher than the expected value, if we go on the assumption that all the ammonium sulfate added to the sample is volatized on ignition. We have rechecked these ignited residues under varied conditions of ignition. These variations included:

1. Ignition with Bunsen burner
2. Ignition with Bunsen burner leaving the crucible uncovered
3. Ignition with Meeker burner
4. Ignition in electric muffle furnace in closed and open crucible.

In all cases the results were high, approaching closely those reported. On qualitative examination the ignited residues were found to be free from ammonium salts and consisted of Na_2SO_4 and $NaCl$. It would seem, therefore, that during the dehydrating operation a partial conversion of the $(NH_4)_2SO_4$ into Na_2SO_4 must have taken place, the Na having been supplied by the soap.

We have checked the method for the detection of triethanolamine as outlined in your letter, using samples of sulfonated oil containing as little as .5% triethanolamine. We are satisfied with the reliability of the procedure and believe it to be a valuable addition to committee report.

Summary and Recommendations

Besides corroborating the results obtain by the A.A.T.C.C. Committee for inorganic salts in sulfonated oils by the direct method, this committee has also observed that ammonium salts during the dehydration may be converted into the corresponding sodium salts and that sodium acetate during the dehydration is converted into soap. In the samples analyzed, the error due to the conversion of the ammonium salts was found to be about 5% of the weight of the salt, which quantity would be negligible in the analysis of the ordinary sulfonated oil. Moreover, since both the soap and the acetic acid, formed during the heating of the oil in the presence of sodium acetate, are soluble in the carbon tetrachloride-oleic acid solution, this salt does not interfere with the method.

The A.A.T.C.C. method determines the inorganic chlorides and sulfates in the form that they are present in the original sample. The manipulations are simple and check results as well as agreement by both committees were entirely satisfactory. In view of these findings this committee recommends the A.A.T.C.C. or direct method as a standard method for the determination of total inorganic sulfates and/or chlorides in sulfonated oils.

PROPOSED STANDARD METHOD OF TEST FOR INORGANIC SULFATES AND CHLORIDES IN SULFONATED OILS

A.O.C.S. Method

Scope:

1. This method of test determines the inorganic sulfates, chlorides, and all other salts that are insoluble in a mixture of oleic acid and carbon tetrachloride, existing in a sample of sulfonated oil by dehydrating the sample, dissolving in a solvent, filtering, and weighing the residue. The presence of sodium acetate does not interfere with this method.

Apparatus

General:

2. Where a Gooch crucible is used, it shall be ignited in a larger crucible in which the Gooch crucible is supported by an asbestos ring as shown in Figure 1.

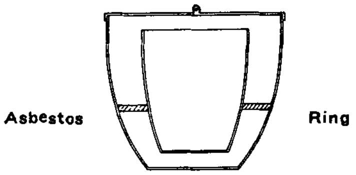

FIGURE I.

Filter Paper:

3. 9 ml. Whatman No. 40 or similar grade ashless filter paper shall be used.

Reagents

Oleic Acid:

4. Oleic acid shall be the U.S.P. grade.

Carbon Tetrachloride:

5. Carbon tetrachloride U.S.P. shall be used as the solvent.

Ether:

6. Ethyl ether shall be U.S.P. grade.

7. Asbestos fiber shall be acid and alkali washed.

Procedure

General:

8. The procedure shall depend upon whether or not the sample contains ammonium salts.

In the Absence of Ammonium Salts:

9. Three to five grams of the sample shall be weighed into a 250 ml. glass beaker, an approximately equal amount of oleic acid added, and the mixture heated in an oil bath, with constant stirring with a thermometer, at a temperature of 105° to 110° C. until practically water-free. The temperature shall now be raised to 118°-120° C. and kept there for about five minutes. The dehydrated sample upon cooling shall remain liquid otherwise more oleic acid shall be added. The dehydrated sample shall be dissolved in 100 ml. of carbon tetrachloride, warmed to 50°-55° C., and filtered through a counterpoised filter paper, using suction if necessary, or a Gooch crucible.

The Gooch crucible shall be prepared by passing through the crucible a thin emulsion of asbestos fiber in water until a pad of about 2 mms. in thickness is formed. The asbestos pad shall then be washed with water until no fibers run through. The crucible shall then be dried and ignited for 30 minutes, at first gently and finally more strongly; 75 ml. of carbon tetrachloride shall now be passed through the crucible, again ignited, cooled in a desiccator, and weighed. The process of washing with carbon tetrachloride shall be repeated until there is no further loss in weight.

The residue shall be washed with three 15-ml. portions of a 2% solution of oleic acid in carbon tetrachloride, then with six 15-ml. portions of hot carbon tetrachloride and finally with two 15-ml. portions of ether or until the residue is free from oil. Care shall be taken that the top of the filter is thoroughly washed. The last traces of the residue may be transferred to the filter by allowing the solvent to evaporate when the salts will become free flowing. The residue shall be dried at 125° to 130° C. for about 45 minutes, cooled in a desiccator, and weighed. The heating shall be repeated until constant weight is reached. The residue shall then be ignited for 15 minutes in a porcelain crucible under a Bunsen burner to a dull red heat, weighed, and the ignition repeated until the weight is constant.

In the Presence of Ammonium Salts:

10. The procedure shall be identical with the procedure in the "Absence of Ammonium Salts" with the following exceptions: (a) in preparing the Gooch crucible, it shall not be ignited but heated at 125°-130° C. for 45 minutes and the heating repeated until constant weight is reached, and (b) the residue, whether in a Gooch crucible or on filter paper, shall not be ignited but heated as in (a).

Calculations

Method:

11. The method of calculation shall depend upon whether or not ammonium salts are present in the sample.

Nonvolatile Inorganic Sulfates and Chlorides (in the Absence of Ammonium Salts):

12. The weight of the "dried" residue divided by the weight of the sample multiplied by 100 shall be the per cent of the "dried" residue. The weight of the "ignited" residue divided by the weight of the sample multiplied by 100 shall be the per cent of the "ignited" residue. The difference between the per cent "dried" residue and the per cent "ignited" residue (in the absence of ammonium salts) shall not be greater than 0.25%.

The per cent nonvolatile inorganic sulfate and chlorides shall be the per cent of the "ignited" residue, or

Per cent nonvolatile, inorganic sulfates and chlorides

$$= \frac{\text{W't ``ignited'' residue} \times 100}{\text{W't Sample}}$$

and shall be reported as ".......% nonvolatile, inorganic sulfates and chlorides by weight, A.O.C.S. Ignition Method."

Inorganic Sulfates and Chlorides, Including Ammonium Salts:

13. The per cent inorganic sulfates and chlorides, including ammonium salts, shall be the weight of the "dried" residue divided by the weight of the sample multiplied by 100, or

Per cent inorganic sulfates and chlorides, including ammonium salts

$$= \frac{\text{W't ``dried'' residue} \times 100}{\text{W't Sample}}$$

and shall be reported as ".......% inorganic sulfates and chlorides including ammonium salts, by weight, A.O.C.S. Non-Ignition Method."

Accuracy

Accuracy:

14. Results by the committee indicate that the accuracy of the method seems to be within 0.1% on the weight of the sample for a

sulfonated oil containing fixed alkali salts, and, in the presence of ammonium salts, about 5% on the weight of the dry ammonium salt.

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(Editor's Note: No official action taken by the Society.)

ABSTRACTS

Oils and Fats

Edited by
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The Chemical Causes for Spoilage of Edible Fats. K. Taufel. *Z. Untersuch. Lebensm.* 72, 287-99 (1936). A review.

The Deterioration of Fat and Fat Structure by Heat and Light in Regard to Economy and Life. II. H. Schmalfuss, H. Werner and A. Gehrke. *Fette u. Seifen* **43**, 243-7 (1936). This is the second section of a paper reviewing deterioration of fats. Major part of the review summarizes the numerous publications of the authors.

Extraction Practice for Fat Containing Residues. Ilona Taussky. *Öle, Fette, Wachse, Seife, Kosmetik* 1936, No. 14, 1-2. In the extn. of oil from used bleaching earth or hydrogenated catalyst an optimum ratio of solvent to residue may be calcd. by experimenting. In tests 5000 l. of benzine were used in a rotary extractor for extn. of 2000, 1200 and 1000 kg., resp. of bleaching earth contg. 30% oil. Each 1000 kg. of original residue retained 500 l. of solvent. The solvent free residues after the first extn. contained resp., 7.9, 5.1 and 4.1% oil. A second extn. with the same amt. of solvent yielded residue, which when freed of solvent contained 1 to 1.4% oil. (*Chem. Abs.*)

Deacidifying Oils with Dilute Lyes. F. Wittka. *Allgem. Oel- u. Fett-Ztg.* 33, 563-9 (1936). Wittka recommends using 2-7° Be. caustic in the caustic refining process. Oils which in the treatment form an emulsion that will not break with the usual salt treatment must be treated (after emulsion is formed) with a small amount of sulfuric acid so that the foots can be separated and require a second deacidifying treatment. Fat acids are recovered from the dilute foots contg. also the washing, by acidifying with H_2SO_4. This process is claimed to be economical for valuable slightly acid oils. It is admitted that the use of more concd. lyes is simpler and cheaper and should be used for the poorer grades and cheaper oils. (*Chem. Abs.*)

Hazelnut Oil. S. H. Bertram. *Öle, Fette, Wachse, Seife, Kosmetik* 1936, No. 14, 2-4. Hazelnut (*Corylus avellana* L.) kernels yielded 54% oil on petroleum ether extn. The characteristics of the oil are d^{20} 0.9144 n_{28} 1.4691, I no. (Hanus) 86.8, sapon. 192.0 and unsaponifiable 0.35%. The oil contained 8.0% satd. acids (per Bertram oxidation method). The unsatd. acids were oleic 78.2 and linoleic 9.1. Efforts to det. solid acids by the Twitchell method yielded solid acids having high I nos. The unsatd. acids were sepd. from the residue and were proven by phys. characteristics of acid and the elaidinized product to be pure 9:10 oleic acid. There was no explanation for the abnormal behavior of the Twitchell detn. for solid acids. The m. p. of the linoleic tetrabromide indicated that the linoleic acid present was the ordinary 9:10, 12:13 linoleic acid. (*Chem. Abs.*)

Rancidity in Fats. I. The Effect of Low Temperatures, Sodium Chloride, and Fish Muscle on the Oxidation of Herring Oil. A. Bansk. *J. Soc. Chem. Ind.* 56 13-15T (1937). The atmospheric oxidation of herring oil at —5° is fairly rapid, but at —20° and —28° it is very slow. Sodium chloride has no effect on the atmospheric oxidation of herring oil. Herring muscle seems to catalyse the oxidation of herring oil. This effect is destroyed by heat. The presence of sodium chloride increases this catalytic effect. It is suggested that this catalytic effect is due to the presence of an oxidative system in herring muscle.

Studies on the Nature of Antioxygens Present in Natural Fats. III. The Occurrence of Antioxygenic Compounds in Extracted Soya-Bean Oilcake. T. G. Green and T. P. Hilditch. *J. Soc. Chem. Ind.* **56**, 23-26T (1937). Extd. soya-bean meal, after digestion with dil. solns. of organic acids (e.g., 2% acetic acid in H_2O or acetone) yields about 10% of material when heated with methyl alcohol. The portion of this ext. soluble in cold acetone (about 2% of the original meal) is a viscous gum which possesses marked antioxygenic properties. In 0.2% concn. in distilled unsaturated fatty esters, the cons. exert marked retarding action on the oxidation of the esters by air at 97.5°. The yield of the conc. is many times greater from the extd. oilcake than from the fatty oils themselves; the antioxygenic compd. removed from oilseeds with the oil appears to be not more than 2 or 3% of the total amt. present in the seed cake. The description of the properties of the antioxygenic concs. previously reported by Olcott and Mattill is generally confirmed.

Oleyl Alcohol. L. A. Hamilton and H. S. Olcott. *Ind. & Eng. Chem.* **29**, 217-223 (1937). The course of oxidation of oleic acid, methyl oleate, and oleyl alco-

Antioxidants and the Autoxidation of Fats. VIII. Autoxidation of Oleic Acid, Methyl Oleate, and