2. an unknown component which may be a lamellar liquid crystal; and 3. the free organic compound.

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International Chemical Union

Minutes of Meeting of International Commission on Fats and Oils. Part I.

London, England, July 1947

Translated by C. L. Hoffpauir of Southern Regional Research Laboratory,* New Orleans, Louisiana.

THIS translation gives the minutes of the meeting in London during July 1947, incorporating the decisions made on the unified international methods and the plans for collaborative work to be done before the next meeting of the Commission. Methods under discussion included those for the sampling and analysis for moisture and oil specified for several oil-bearing seeds. They also included those for alkalis and rosin in soaps and those for soluble and insoluble volatile acids and sterols in fats and oil, also for the determination of thiocyanogen and peroxide numbers in fats and oil. The organization of and the representation on the present International Commission on Fats and Oils are also given.

INTERNATIONAL COMMISSION FOR THE STUDY OF FATTY MATERIALS

In accordance with the attached minutes of the London meeting, the following notes are offered:

SUPPLEMENT I_1—METHOD OF ANALYSIS OF OLEAGINOUS SEEDS

Having edited the text in accordance with the decisions taken by the Commission, we have taken the liberty, without submitting it to you for examination, to transmit it to the International Union of Chemistry so that it might be published in the booklet of unified methods, 1947.

SUPPLEMENT I_2—METHOD FOR DETERMINATION OF ROSIN

This method was adopted by the Commission and should be inserted in the published book.

The program of work you proposed is described as follows:

I—*Determination of Free Caustic Alkali in Soaps.* The text is given in supplement II_1.

We propose that the method be tried under the following conditions:

Weigh approximately 5 grams of fatty acid containing no trace of neutral oil into a tared flask. Add alcohol and exactly neutralize with an aqueous solution of sodium hydroxide, completing the neutralization with N/10 alkali. Remove all the alcohol and the major part of the water by heating and then placing in an oven. Add 5 ml. of aqueous N/10 sodium hydroxide containing in solution 20 mg. of anhydrous sodium carbonate. An analytical sample of a soap with a known content of free caustic alkali and free carbonate alkali is thus obtained. Add immediately the necessary quantity of absolute alcohol, previously boiled and neutralized at a temperature of 70°C. Finish the analysis according to the method and compare the free caustic alkali titrated to that which was effectively introduced.

II—*Determination of Free Carbonate Alkali.* The text of the method is given in Supplement II_2.

First Method: This method should be applied to the above analysis after titration of the free caustic alkali.

Second Method: Prepare neutral soap as described. Incorporate in it calcium carbonate and a known quantity of caustic alkali and of sodium or potassium carbonate (of the order of 1%). Apply the method to the soap so obtained.

III—*Qualitative Research on Rosin.* The text is given in Supplement II_3.

The technique should be tried on different products capable of producing the colors and on the same products mixed with 0.50% of rosin: lanolin, sulforicinoleate, degras, oxidized linseed oil, oil of grape seed, chinawood oil, or fish oil.

IV—*Soluble and Insoluble Volatile Acids.* The text is given in Supplement II_4.

The method should be tried on a sample of fatty material (a mixture of cow's butter and coconut oil) which will be distributed by the Swiss Commission.

V—*Phytosterols and Cholesterols.* The text is given in Supplements II_5 (Netherlands Commission) and II_{5A} (Danish Commission).

The tests should be made on the sample mentioned in paragraph IV above.

VI—*Thiocyanogen Number.* The lead thiocyanate should be prepared by the two methods described in Supplements II_6 and II_7.

The thiocyanates so prepared will be compared by use of them as reagents in the methods of Supplements II_{6A} and II_{7A} as applied to a sample of semi-drying oil which will be distributed by the Swiss Commission.

Then comparisons will be made of methods II_{6A} and II_8 as applied to the sample of tallow which

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* One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

will be distributed by the Swiss Commission in order to note the influence of the addition of carbon tetrachloride.

The text of the method for absolute iodine number, mentioned in the minutes, will be sent later. It will be tried on the sample of semi-drying oil.

VII—*Peroxide Number.* The same semi-drying oil will be used to compare results of methods II$_9$, II$_{9A}$, and II$_{10}$, with analyses being made as near as possible on the first of February, 1948, and the samples kept in the dark until that date. It is important that the different Commissions work at the same time on the samples kept under similar conditions.

VIII—*Determination of Neutral Oil in Strongly Acid Fatty Materials.* The four methods (Supplements II$_{11}$, II$_{12}$, II$_{13}$, and II$_{14}$) should be tested comparatively on a sample of olein and on a sample of acid oil which will be distributed by the Swiss Commission.

It will be necessary that the results of the work of the National Commissions be returned not later than the first day of May, 1948.

Please accept, Mr. President, the expression of my best wishes.

Jean Vizern, *Secretary.*

MEETING, ROYAL SOCIETY, LONDON, 1947

First Meeting, July 18

Present:

Denmark: Helholt.
United States: Dr. Snell.
France: Prof. Margaillan, Vizern, Wolff, Melle Francois, Dr. Loury, Dr. Sisley.
Great Britain: K. A. Williams, Melle E. Lewkowitsch, Dr. Lee, G. J. Robertshaw.
Italy: Prof. Anselmi, Dr. Balestrini.
Netherlands: Dr. Voerman, Dr. Bertram, Dr. Boekenoogen.
Switzerland: Dr. Sturm, Weder,
Czechoslovakia: Prof. Vesely.

The meeting opened at 5 p. m. President: Dr. Voerman; Secretary: Dr. Vizern.

The president welcomed the delegates and expressed regrets for the absence of Prof. Fachini because of his health. Denmark and the United States were admitted unanimously.

The president again saluted the new members and expressed his satisfaction. After approval of the minutes of the meeting in London, 1946, the technical discussion began on the basis of the report presented by the secretary general.

I. Analysis of Oleaginous Seeds.

a) *Preparation of the Sample:* Though international unification of procedures are desirable, it is not the duty of the Commission to fix the rules of sampling. However, the Commission is responsible for indicating the minimum weights of samples permitting the analyst to perform correct determinations.

It is decided that the sample sent to the laboratory should weigh approximately 2 to 5 kilos for seeds of moderate dimensions such as cabbage, palm, peanuts, and soya and for large seeds such as shea and coconut, and a minimum of 200 grams for small grains such as flax, rape, and sesame.

The chemical characteristics (content of moisture and oil) should be expressed always in per cent of the seed as it is, that is to say containing its impurities. Eventually, in order to satisfy a demand, the characteristics of the pure seed can be indicated.

The analyst is allowed freedom to proceed with the determinations according to the circumstances, on the seed as is or on the pure seed, after separation of the dust by sifting and of mineral and organic foreign bodies by sorting.

In this latter case the contents in per cent of the seed as is will be calculated by taking into account the percentage of impurities. Further, it is necessary eventually to determine moisture and oil in the impurities and take into account the percentages so obtained in calculating the results on the original seed sample. Certain seed may contain certain foreign oleaginous seed. Some parties may demand an oil determination on such foreign seed.

When the acidity of the oil is requested, the analyst who extracts the oil from the different fractions (pure seed, impurities, and foreign seed) should take into account the different acidities obtained on the fractions in making the final calculations on the original sample.

On the intervention of the Italian and Swiss Commissions the discussion was directed to the determination of impurities required by certain contracts. It was decided that this determination should be made by the usual methods of sieving and sorting. When the dusts contain oil, the percentage obtained should be lowered by the weight of seed corresponding to the weight of the oil. Therefore in seed containing 50% oil from which are separated 2% of dust containing 20% oil, that is 0.4% of oil, the seed are said to contain $\frac{0.4\times100}{50} = 0.8\%$ of particles of seed. Hence, the percentage of non-seed particle or inert dust present as impurities is $2.00\text{-}0.80 = 1.20$. The question of foreign oleaginous seeds, in the determination of impurities, should be settled according to the specifications of the contracts.

The Commission fixed also the minimum weights of reduced sample (ground to a meal with or without previous separation of impurities) as 1 kilo for such materials as copra and olives, 500 grams for all seeds except small seeds, and 100 grams for small seeds such as flaxseed, sesame, poppy, and tomato. For the very small seed, after sifting and separation of large foreign materials, it is optional to estimate the other foreign materials on an aliquot which must not be less than 10 grams.

All these operations should be performed with sufficient rapidity and under such conditions that there are no appreciable changes in the moisture content. The same precaution should be observed in grinding. The method for the preparation of copra was discussed. All the delegates were in accord with the fact that grinding inevitably results in loss of oil. Grating appears to be necessary. The Netherlands and Italian Commissions recommend a mechanical grater of German manufacture, capable of grating the entire sample in a short time. The Netherlands Commission will look into the actual possibility of buying such equipment and will advise the International Commission.

The meeting was adjourned at 7 p. m.

Second Meeting, July 19, at 10 a. m.

b) *Determination of Moisture:* All the members were in accord as to the necessity of making the determination on the ground material for all large and medium size seed. Opinions differed for small seed. The French Commission maintained that determinations made on the ground material lead to low results because of the drying nature of most of the oils contained in small seeds. Recognizing that the determination on the whole seed is more exact, though more lengthy, a factor which should not enter into the choice of an arbitration method, and that the coarsely ground material recommended by the British Commission is impossible to standardize, the Commissions came to the view of the French Commission. It was therefore decided that for small seeds the moisture should be determined on the whole seed.

The Commission recommends further the use of drying ovens which can be obtained in sufficient quantity.

The use of an analytical sample of approximately 5 grams (± 0.5 gram), weighed to 1 mg., was adopted. Metal crystallizing pans, preferably of aluminum, 7 cm. in diameter and 3 to 4 cm. high, are to be used.

Inasmuch as vacuum ovens cannot be used generally, ovens which can be regulated at 103°C. to ± 2°C. should be used. Since there are important variations of temperature between different points in actual air ovens, the Commission requests the Commission on Laboratory Material to perfect an oven giving a temperature as uniform as possible. The first weighing should be made after three hours heating in the oven. The samples are then weighed each hour until differences in weight between two successive heatings and weighings of 5 mg. at most are obtained.

c) *Determination of Fatty Material:* As specified in its decision in London in 1946, the Commission adopts petroleum ether distilling between 40 and 60°C. and having a bromine number less than one as the solvent for extractions. It feels that as soon as production permits it will adopt pure pentane or normal hexane instead as the solvent.

After some discussion the Commission adopted an analytical sample of approximately 10 grams within 1 gram, weighed to a milligram.

The proposition of the Netherlands Commission relative to preliminary drying of the analytical sample for three-fourths hour at 103-105°C. in order to inactivate the enzymes, opposed by the British and French Commissions who consider the operation useless compared to submitting the analytical sample to immediate extraction, was rejected by a vote of four against three. It was decided that the unified text of 1947 will not include this requirement but that the Commissions make tests to establish whether it should be given consideration again. The Commission recommends that the extractions be undertaken as soon as the grinding of the sample is complete.

Very damp seed, such as olive, tomato, and grape, should be partially dried to a moisture content of about 10%. The Commission abandons the recommendation made previously of adding anhydrous sodium sulfate as the French Commission had objected that the temperature of extraction was almost always higher than that of transformation of sodium sulfate from the hydrated to the anhydrous form.

The analytical sample should be extracted for four hours in an appropriate extractor. The use of a continuous extractor was recommended. A second extraction for two hours duration is made after grinding the meal in a mortar as fine as possible, using a metallic mortar and silicious sand. The same extraction flask and solvent as for the first extraction are used.

A third control extraction for two hours duration, the results of which are collected and weighed separately, is done after grinding the meal in a mortar again.

The weight of the oil so obtained should not exceed 10 mg. In case the amount obtained on the third extraction exceeds 10 mg., new extractions, preceded by new grinding, should be made until satisfactory results are obtained.

The major part of the solvent should be removed by distillation and the last traces by heating at about 100°C., but not above 105°C., with the aid of either a current of air or a vacuum. The time of heating should be brief and should not exceed 20 minutes. A second heating should be done in the same manner. The difference in weight obtained between the two weighings should not exceed 10 mg. If greater it will be necessary to continue the removal of the solvent.

Third Meeting, July 21, at 9:30 a. m.

II. DETERMINATION OF FREE CAUSTIC ALKALI IN SOAP

The Netherlands Commission suggested the barium chloride method. This proposal was opposed by the French Commission, who consider this method unreliable, in particular when it is used to determine small quantities of free caustic alkali. The British Commission favored the alcohol method for soaps containing small quantities of free caustic alkali and the barium chloride method for others. It appears that the objections of the British Commission to the alcohol method in the case of soaps containing larger amounts of free alkali were due to the fact that the final titration is done with an aqueous acid solution. Too large a dilution of the alcohol is obtained which causes the carbonate alkali to be titrated as caustic alkali.

Bringing together their points of view and considering that it is desirable to adopt a single general method to be used on an analytical sample sufficiently large, because of heterogeneity in certain soaps, the delegates agreed on the following points:

The analytical sample will be 10 grams.

Absolute alcohol should be used in sufficient quantity that the alcohol should be at least 95% after dissolving the analytical sample.

The necessary quantity of alcohol will be boiled under reflux for 10 minutes in order to eliminate dissolved gases.

After addition of phenolphthalein the alcohol will be neutralized at a temperature of about 70°C. with N/10 alcoholic potassium hydroxide solution.

The analytical sample is then added rapidly into the alcohol. It is boiled under reflux until the soap is dissolved. The alkali is titrated at a temperature of approximately 70°C. with N/10 alcoholic solution of sulfuric acid for samples containing up to 0.10% free caustic alkali. A N/2 solution is used for higher amounts of alkali.

However, in order to agree with the desires of certain nations, the Commission decided that the method, so established, will be submitted to study during the current year before being definitely adopted.

III. DETERMINATION OF FREE CARBONATE ALKALI

The divergences of viewpoints, which were manifested, arose from the fact that in certain countries the industries manufacture mostly soaps to which are added silicates, borates, phosphates, and the like, and in other countries the soaps are essentially of the unfortified type of "Marseille Soap." It seems clear that for these last, which contain only very small quantities of sodium carbonate, the method of determination of carbon dioxide which will be expressed finally as sodium carbonate is of doubtful value.

As a consequence the Commission decided to recommend a preliminary qualitative method consisting of dissolving the soap in alcohol, with final strength of the alcohol in the solution at least 95%. When the solution involves but little trouble, one applies the method which consists of dissolving in alcohol, titrating the free caustic alkali, then lowering the strength of the alcohol to 50% by the addition of water and titrating the carbonate which one dissolved in this medium.

When the solution leaves appreciable insoluble material the test is reproduced quantitatively, filtered, and the insoluble portion washed with alcohol and submitted to the techniques of mineral analysis. The text will be submitted to a new experimental examination by the different commissions before its definite adoption.

Fourth Meeting, July 21, at 4:30 p. m.

IV. DETERMINATION OF ROSIN IN SOAPS

The Commissions stated that the two methods compared on the same sample of soap prepared by the Swiss Commission (Mac Nickol—Vizern and Guillot) gave comparable results. The differences with relation to theoretical content result not from the method but from the uncertainty of the molecular weights of the rosin used and on the limit of esterification of the fatty acids, which varies somewhat with their nature.

The Commission decided to adopt the method of Vizern and Guillot which requires only usual reagents. It will be specified that the determination of rosin can only give approximate results because of signal uncertainties.

As a result of some discussion the Halphen test was considered as more specific than the Liebermann-Storch reaction for characterizing and detecting small quantities of rosin.

The National Commissions will apply the Halphen test to the following fatty materials, making the test before and after the addition of 0.50% of rosin: lanolin, sulfonated products, degras, oxidized linseed oil, grape seed oil, and chinawood oil.

V. SOLUBLE AND INSOLUBLE VOLATILE ACIDS

The discussion opened on the eventuality of replacing the expression of Reichert and Polenske numbers by an expression of milligrams of potassium hydroxide per gram as in the expression of the saponification and acid numbers. The Italian Commission rose against this modification, pointing out that the reference values contained in the chemical literature will be modified and that the methods do not furnish true results but only results dependent on a stated method of operation. The French Commission supported the proposal, indicating that the conventional character of the method has no bearing on the essential obligation of the Commission, which is terminated in homogenous definitions of the different indices. The Czechoslovakian Commission supported this point of view by showing interest in adopting definitions permitting easy comparison between different indices. As a consequence, the International Commission decided to adopt indices of soluble and insoluble volatile acids which will be determined only by a specific method and expressed in milligrams of potassium hydroxide per gram of sample.

The technique was studied. All the Commissions were in accord on the apparatus and its dimensions. It is specified that the duration of the distillation should be between 19 and 21 minutes. Further, the distillate, before filtration, should be brought to a temperature of 15°C. for 15 minutes. The walls of the condenser and the filter will be washed three times successively with 15 ml. of recently boiled water cooled to a temperature of 15°C. before dissolving the insoluble fatty acids in alcohol. The method with these specifications will be tried on a mixture of one-third cow's butter and two-thirds coconut oil, which will be distributed to the several National Commissions by the Swiss Commission.

VI. PHYTOSTEROLS AND CHOLESTEROLS

Since the industry is apt to abandon the digitonin standard, it was decided that the study be reinstituted on the use of the method previously proposed by the Netherlands Commission, which differs from the Danish method only in several details. The National Commissions will try these methods on the sample intended for the determination of volatile acids.

VII. THIOCYANOGEN NUMBER

The Commissions are in accord on the necessity of specifying that the lead thiocyanate should be of recent preparation and that it should be prepared by the analyst himself. The different texts on hand were compared. In order to determine the most simple and dependable technique the National Commissions will make the investigations on a sample of semi-drying oil and a sample of tallow which will be distributed by the Swiss Commission.

These investigations will include:

1. The comparison between the two proposed techniques for drying the lead thiocyanate: a) washing the precipitate with water and drying in a vacuum bell jar with phosphoric anhydride as a desiccant; and b) washing the precipitate with water, acetic acid, and acetic anhydride and suspending in acetic anhydride for at least 8 days (proposal of the Netherlands Commission, which feels that the preservation is thus assured for a very long time).
2. The comparison between the method utilizing only acetic acid as the vehicle and the method using a mixture of acetic acid and carbon tetrachloride. The tests prescribed will be tried on a tallow to see if solution of the solid fatty material is not too difficult in the absence of carbon tetrachloride.

The reagents prepared for the tests of the different techniques should have concentrations of approximately N/10.

The French Commission proposed to study the comparison between the determinations of the content of linoleic acid by the thiocyanogen number and by the method of absolute iodine number, which it considered easier to use.

Fifth Meeting, July 22, 9:30 a. m.

VIII. LEA NUMBER (PEROXIDE NUMBER)

The peroxide number is expressed by some as the ml. of N/500 solution per gram, and by others as ml. of N/100 solution per kilo. In view of these differences and with the desire to have uniform definitions for the different indices the French Commission proposed to unify the peroxide number and to define it as the number of milligrams of peroxide oxygen contained in a gram of sample. The Commission agreed to this proposal but because of the small quantity of oxygen decided to express it in micrograms. Hot and cold methods with and without the use of CO_2 were considered. It was decided that they will be tried on the semi-drying oil used for the study of the thiocyanogen number.

IX. SOAPSTOCK (PATES DE NEUTRALISATION)

The Commission decided not to take any position on the analysis of these products as they are of interest only to certain National Commissions, **are not** actually objects of international trade, and there is a tendency in the industry to transform them to fatty acids or true soaps. However, it is interested in the determination of neutral oil in strongly acid fatty materials. It retains four methods which will be tried on a sample of olein and on a sample of acids from the decomposition of soapstocks which will be distributed by the Swiss Commission.

Composition of the Commission for 1947-48

President—Dr. G. L. Voerman
Vice President—Foster D. Snell and Dr. Sturm
Secretary—J. Vizern
Members:
- Denmark—K. Helholt
- United States—Foster D. Snell, H. E. Longenecker, and V. C. Mehlenbacher
- France—Prof. L. Margaillan, J. Vizern, G. Wolff
- Great Britain—K. A. Williams, Dr. Lee, G. J. Robertshaw (observer for the leather chemists)
- Italy—Prof. S. Fachini, Prof. S. Anselmi, Dr. G. Balestrini
- Netherlands—Dr. G. L. Voerman, Dr. I. S. H. Bertsam, Dr. H. A. Boekenoogen
- Switzerland—Dr. Sturm, Weder
- Czechoslovakia—Prof. Vesely, Dr. Herites, Dr. Metzl

Meeting 1948

Considering that the success of the work is due in a large part to the frequent contacts between its members, the Commission decided to meet in 1948 in Paris with preference for the month of July.

PUBLICATION OF THE UNIFIED METHODS, 1947

The view is expressed that the International Union of Chemistry will publish the unified methods as adopted to date.

(To be continued in Part II and III)

Minor Oil-Producing Crops of the United States

E. B. KESTER, Western Regional Research Laboratory,[1] Albany, Calif.

IN the six years since the author and G. R. Van Atta published an article under the present title,[2] new developments in the processing of minor oil-bearing materials, derivable principally from agricultural waste, have made it desirable to review the subject and include revisions in the data, descriptive matter on present practices and trends, and reports of technological investigations in the more recent literature.

The third year after V-J day finds the United States in a period of inflated prices and costs. Vegetable oil and meal prices in 1940 and 1947 are listed in Table I. Alhough 1940 and 1947 prices differ widely, showing a two- to three-fold increase, with few exceptions, for the period, the trend has been, by no means, consistently upward. During the spring of 1947 there was a general marked drop in oil prices, followed by a rapid rise to a maximum in January, 1948, and then a sharp decline along with other commodities. These broad fluctuations, of course, may continue until the postwar turbulence, which affects all phases of our national life, has subsided.

TABLE I

Oil Prices (Cents per Pound)

(From *Oil, Paint, and Drug Reporter* and from *Feedstuffs*)

	June 1940	Nov. 1947
Corn	8⅜	......
Cottonseed	9	32
Almond (sweet)	75	180
Soybean	7	31
Linseed	9.8	31.8
Tung	22	28
Apricot kernel	38	65-85

Feed Prices (Dollars per Ton)

	May 1940	Nov. 1947
Cottonseed (43%)	32.50	93.00
Coconut	21.00	78.50
Linseed	24.00	83.00
Soybean	38.50	103.00

However unstable may be our present scale of prices, the existence of enormous tonnages of agricultural wastes that pollute streams, create health hazards, and are a liability to the food manufacturer, suggests with some insistence that something should be done to reclaim at least a part of their values. The observer, speculating on what he can recover or make from such wastes, would probably consider one or more items in the following list:

Feed
Substrates for microbiological processes
Syrups
Vegetable oil and meal
Citric, tartaric, or other organic acid
Vitamins
Plastic filler
Tannin
Mucilage
Pectin
Furfural
Charcoal
Fertilizer

He may then attempt to sample a waste and have assays made to learn how much useful material he might recover and, with the data, estimate his gross returns per unit of weight. At this stage disillusion often comes, as he multiplies a number of conversion factors and ends with a pitifully small percentage of starting material in the form of salable goods. The vegetable oils in waste often hold more promise than some others in the list, as they usually involve mere segregation of the oil-bearing part, such as seeds, followed by expression or extraction, but even in this case the various economic factors bearing on a proposed enterprise should be carefully considered

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[1] Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

[2] Oil and Soap *19* (7), 119-125, July, 1942.