21°-100°C. and 0°-100°C. probably included a small amount of energy due to a modification in the solid state, this is not necessarily so. For example, their temperatures were increased so rapidly to 100°C. (only two or three minutes), they may not have been converted to the alpha or beta form. Going on this assumption and also that the stearines were originally in the gamma form, the latent heat of fusion of cottonseed stearines agrees remarkably well with the 44.2 calories per gram value reported by Rao and Jatkar for the crystallization heat of tristearine in the gamma form.

Attention should be called to the agreement between the latent heat of fusion values for the different stearines in the range 21°-100°C. and 0°-100°C. This agreement is of especial interest when it is considered that detailed calculations were necessary to arrive at these results. It is also of interest to observe that the specific heats of the liquid oils and the completely solid stearines, as well as the latent heat of fusion, are a little more than one-half that of the corresponding states of water.

Summary

The specific heats of several vegetable oils have been determined by a more exact procedure than those which seem to have been used heretofore. Values slightly higher than the literature figures were obtained, that is, an average value of 0.53 was determined as compared to reported results of 0.50. The specific heat of cottonseed oil with respect to temperature increases slightly with elevating temperatures. The specific heats at different temperatures were not determined on the other oils, but likewise, these probably show no significant variation in specific heat with respect to temperature in the range 21° to 100°C.

The specific heats of completely solid vegetable fats are believed to be reported for the first time. These values are about the same for such different stocks as completely hardened palm and sunflower oils, and their values are approximately one-half that of the liquid, that is, 0.28 as compared to 0.53.

The latent heats of fusion of a number of fats have also been determined by what is thought to be a satisfactory procedure. Theoretical aspects and empirical results are given to show that the latent heat of fusion varies with the temperature when two phases are present, and that for this reason, any references to the latent heat of fusion should be accompanied by temperature limitations. This is not so important, however, when the specimen is completely solid at the temperature in question.

The data and graphs presented show the general variation in latent heat of hardened cottonseed oil at one temperature with respect to iodine value.

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Purification of Oleic Acid, Methyl Oleate, and Oleyl Alcohol for Use As Chemical Intermediates[1]

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Introduction

During research on the chemical modification of animal fats and oils, it was desirable to have readily available large quantities of oleic acid, methyl oleate, and oleyl alcohol of a relatively high degree of purity (90% or higher). None of the commercial samples examined contained more than 75% of the oleic constituent. As a rule, the oleic content was between 60 and 70%. It was impossible to purchase pure materials, and in the case of oleic acid high-priced preparations labelled to indicate a high degree of purity invariably contained high percentages of linoleic and saturated acids. For many commercial uses of these materials a high degree of purity was not essential, but it was felt that if purified products were available commercially new uses would develop.

The impurities present usually made reaction products more difficult to purify and necessitated handling considerably more material for a smaller return of the desired product. Also, in many reactions the impurities were so altered that it was not possible to isolate any useful products from this portion of the starting material except with great difficulty. This was particularly true of the linoleic constituent, which in many reactions behaves essentially the same as the oleic constituent. As a result larger quantities of reagents were required to react with the impure starting materials in order to insure complete utilization of the oleic constituent, thereby increasing the cost of any reaction.

Laboratory procedures for the preparation of highly purified (96-100%) oleic acid (1) (6), methyl oleate (9), and oleyl alcohol (2) (4) are fairly well known, but these procedures are lengthy and tedious and often result in low yields. Low-temperature crystallization methods have been employed to prepare pure oleic acid and methyl oleate, but as far as we can ascertain, purified oleyl alcohol, other than its possible

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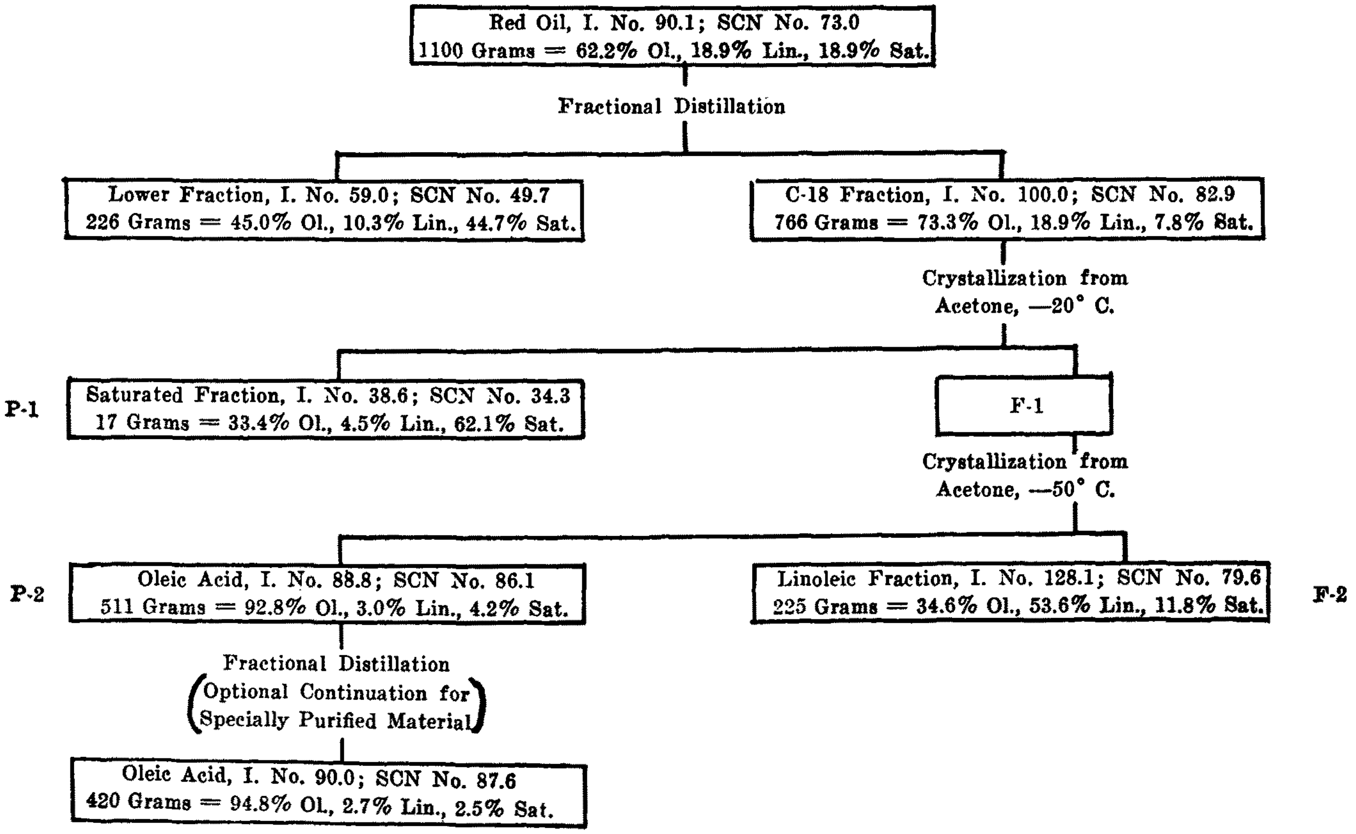
[1] This paper was presented at the meeting of the American Oil Chemists Society, held in Chicago, on October 7, 1943.

CHART I

SUMMARY OF PURIFICATION OF RED OIL BY PROCEDURE I



Red Oil, I. No. 90.1; SCN No. 73.0
1100 Grams = 62.2% Ol., 18.9% Lin., 18.9% Sat.

Lower Fraction, I. No. 59.0; SCN No. 49.7
226 Grams = 45.0% Ol., 10.3% Lin., 44.7% Sat.

C-18 Fraction, I. No. 100.0; SCN No. 82.9
766 Grams = 73.3% Ol., 18.9% Lin., 7.8% Sat.

Saturated Fraction, I. No. 38.6; SCN No. 34.3
17 Grams = 33.4% Ol., 4.5% Lin., 62.1% Sat.

Oleic Acid, I. No. 88.8; SCN No. 86.1
511 Grams = 92.8% Ol., 3.0% Lin., 4.2% Sat.

Linoleic Fraction, I. No. 128.1; SCN No. 79.6
225 Grams = 34.6% Ol., 53.6% Lin., 11.8% Sat.

Oleic Acid, I. No. 90.0; SCN No. 87.6
420 Grams = 94.8% Ol., 2.7% Lin., 2.5% Sat.

isolation from natural sources, has been prepared only by the reduction of purified esters of oleic acid. In most of the procedures described in the literature, the parent material is olive oil. This oil has a high oleic content, but for the commercial production of oleic acid, methyl oleate, and oleyl alcohol, olive oil is not the most satisfactory starting material because it is fairly expensive, and, at present, difficult to obtain. There is little doubt that if purified oleic acid, methyl oleate, and oleyl alcohol were available commercially at a comparatively low price, the organic chemist would be able to produce many industrially valuable derivatives from these substances. In addition, these purified materials should find increased utility where good color stability and lack of color and odor are advantageous. Examples of such uses include pharmaceutical preparations and cosmetics.

The present investigation was undertaken for the purpose of preparing oleic acid, methyl oleate, and oleyl alcohol of high purity (above 90%) in good yields from inexpensive and readily available commercial materials by a relatively simple procedure. This was accomplished by fractional distillation and a single low-temperature fractional crystallization.

Experimental

Starting Materials: The commercial oleic acid employed was a typical sample of red oil. The methyl oleate employed was prepared from the red oil by a standard esterification method using a large excess of anhydrous methanol and a small quantity of concentrated sulfuric acid as a catalyst. The methyl oleate was not distilled prior to its use in the fractionation procedure described below but was used directly as obtained. The oleyl alcohol was of commercial origin. Thiocyanogen numbers were determined using 0.1N thiocyanogen solution and a three-hour reaction time (7) (8). Theoretical values for thiocyanogen and iodine numbers were used in calculating the composition of the starting materials and the purified products.[2]

Purification Processes: Two lines of approach were tried. In procedure I the starting material was first fractionally distilled to obtain a fraction which consisted almost entirely of C-18 compounds. This fraction was then fractionally crystallized once from acetone at low temperatures. In procedure II the starting material was first fractionally crystallized, and the oleic fraction thus obtained was fractionally distilled.

Procedure I: The starting materials invariably contained fractions both below and above C-18 in chain length. These were substantially removed by one fractional distillation through a Vigreaux column 3 feet long and 1 inch in diameter (approximately 5 theoretical plates), electrically heated, and insulated with magnesia pipe covering to reduce heat loss. A still head with automatic control of reflux ratio was employed. A reflux ratio of about 4:1 was employed during fractionation. Eleven hundred grams of material were usually distilled in each run at a pressure of about 1 to 2 mm. It was possible to fractionate

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[2] Formulas employed for calculating composition do not yield absolutely correct values when applied to crude starting materials, since they are based on the assumptions that the starting materials contain no unsaturated substances below or above C-18 in chain length and contain no unsaturated substances with more than two double bonds. The error introduced by these assumptions is not large, and is negligible when the formulas are applied to fractionally distilled or crystallized oleic fractions.

1100 grams in an average working day. The lower fractions from red oil and commercial oleyl alcohol were solids at room temperature. In no case was the distillation residue more than 5% of the total starting material.

The low-temperature crystallizations and filtrations were conducted in a room at ordinary temperature. The crystallizations were carried out in a 3½-gallon Pyrex glass bottle equipped with an efficient mechanical stirrer and a thermometer. The bottle was placed in a 6-gallon stoneware crock, which in turn was in a wooden box. The free space between the crock and the box was filled with powdered 85% magnesia pipe-covering compound (average thickness of insulation, about ¾ inch). The actual cooling of the crystallizing solution was effected by surrounding the bottle with an acetone-dry ice bath. This cooled the crystallizing solution to —70° C., if necessary, in a short time. The direct addition of dry ice to the crystallizing solution was not satisfactory because of possible contamination from the impurities in the dry ice.

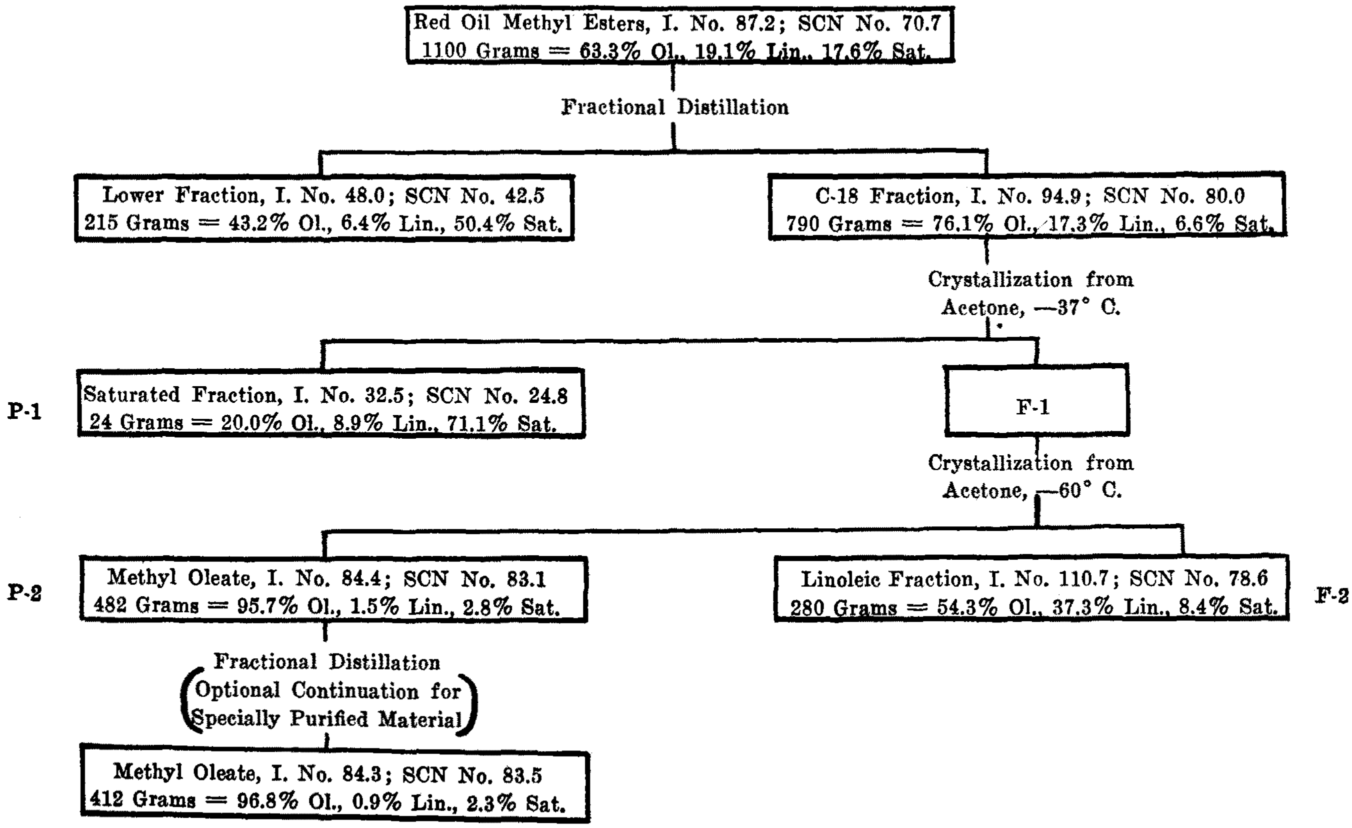
All filtrations were carried out in a 10-inch Buchner funnel which was jacketed by a galvanized iron pail wrapped with several thicknesses of asbestos cloth. The funnel was supported by a tight-fitting rubber stopper inserted through a hole in the bottom of the pail. An additional rubber stopper was attached to the protruding stem of the funnel for insertion into the filter flask. In this way it was possible to surround the funnel with acetone-dry ice mixture at any temperature down to —70° C. The liquid surrounding the funnel was maintained at the temperature at which it was desired to carry out any particular filtration.

Inasmuch as the crystallization technique was the same for the preparation of oleic acid, methyl oleate, and oleyl alcohol, only the procedure employed with oleic acid will be described in detail. Since different temperatures were required for each substance, however, when temperatures of crystallization are given, the corresponding temperatures required for methyl oleate and oleyl alcohol will be shown in parentheses.

Six hundred grams of the C-18 fraction obtained in the fractional distillation of red oil (methyl esters of red oil or commercial oleyl alcohol) were dissolved in 6 liters of acetone, and the solution was cooled to —20° C. (methyl oleate, —37° C.; oleyl alcohol, —10° C.), with stirring. The stirring was just rapid enough to keep any material from adhering to the sides of the bottle. The mixture was maintained at this temperature for one hour, with stirring, and then filtered through the jacketed funnel, which had been pre-cooled to the proper temperature. The solid material removed consisted mainly of stearic acid (methyl stearate or octadecyl alcohol). The filtrate was diluted with 3 liters of acetone and cooled to —50° C. (methyl oleate, —60° C.; oleyl alcohol, —45° C.), with stirring. After being maintained at this temperature for about one hour, the mixture was filtered through the jacketed funnel, which had been pre-cooled to the appropriate temperature. In general, all filtrations were very rapid. The white, crystalline cake of oleic acid (methyl oleate or oleyl alcohol) was sucked and pressed as dry as possible and then washed on the funnel with 1400 cc. of acetone cooled to —70° C. The crystal cake was well mixed with the wash liquid and then sucked and pressed as dry as possible. The oleic acid (methyl oleate or oleyl

CHART II

SUMMARY OF PURIFICATION OF RED OIL METHYL ESTERS BY PROCEDURE I



alcohol) was then cut out of the funnel, and the occluded acetone was recovered by distillation. The filtrate and washings (linoleic fraction) contained the bulk of the linoleic acid (methyl linoleate or linoleyl alcohol) present in the C-18 distillation fraction.

This combination of fractional distillation and a single low-temperature fractional crystallization constituted procedure I (two-stage process). It was recognized, however, that it was not possible to remove all the fractions below and above C-18 in one distillation. In some cases, therefore, the main oleic fractions were submitted to an additional fractional distillation after the low-temperature crystallization. The increase in purity of the final product by this step, however, was not sufficient to compensate for the time spent and the reduction in yield. The progress of the purifications by procedure I is illustrated in charts I, II, and III.[3] The re-fractionation step is added for completeness only.

Procedure II: The starting material was first fractionally crystallized, and the oleic fraction thus obtained was fractionally distilled. Crystallizations and distillations were carried out as previously described. It was not possible to crystallize satisfactorily the crude methyl oleate without a prior fractional distillation. Red oil did not crystallize and filter as well as the distilled material, and it was necessary to remove saturated acids at —25° C. and oleic acid at —60° C. The crystallization of commercial oleyl alcohol offered no difficulties. The progress of the purifications by procedure II is illustrated in charts IV and V. Although only 600 grams were crystallized at any one time, for purposes of comparison with the results obtained by procedure I, yields were recalculated on an 1100-gram basis.

Properties of the starting materials and final products not shown in the charts are given in Table I. Yields are based on the quantity of oleic constituent in the final product, as compared with the quantity in 1100 grams of the starting material. Yields reported for purifications by procedure I are based on the two-stage process for comparison with procedure II and do not include the final re-fractionation.

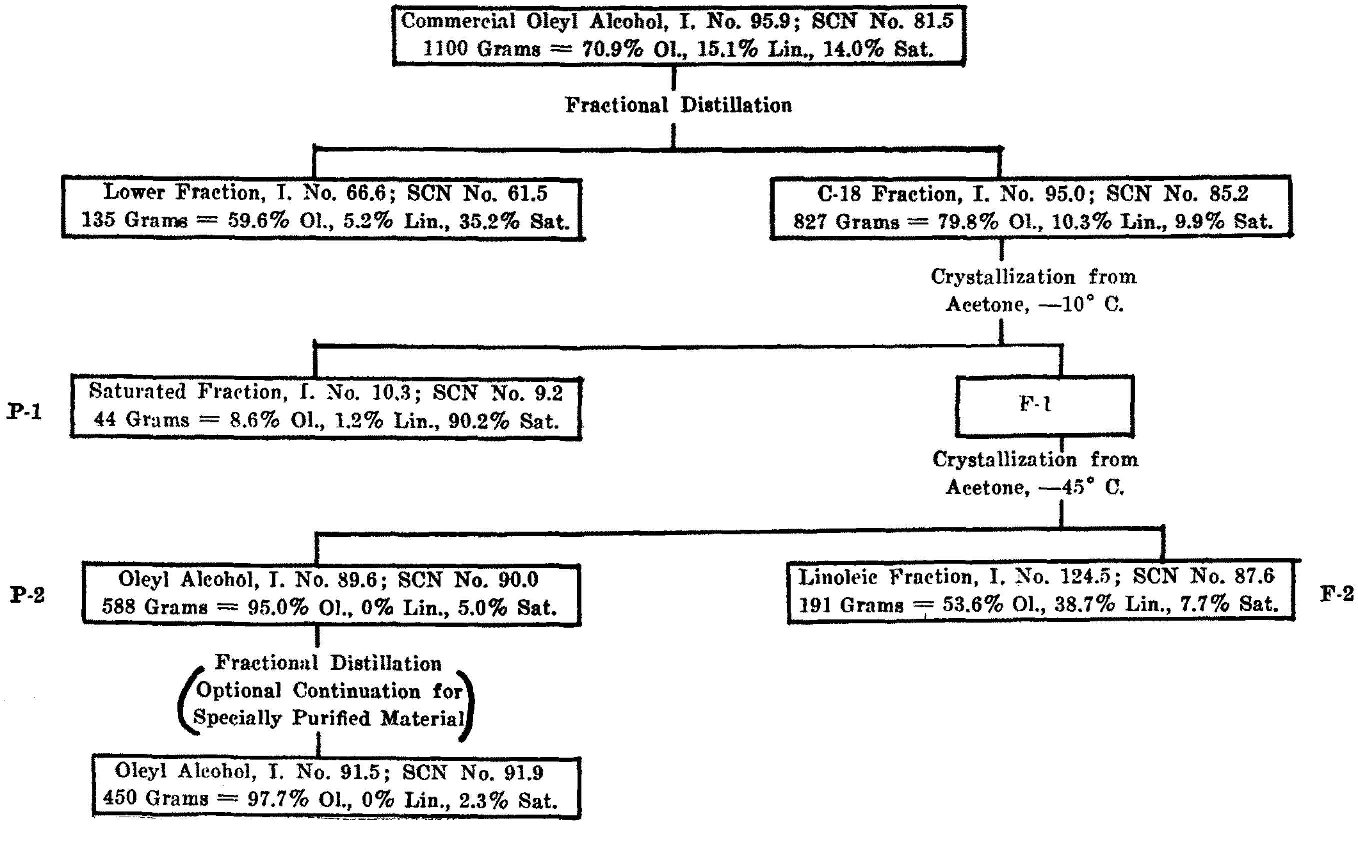
Discussion

The data presented in this report indicate that the large-scale industrial preparation of purified oleic acid, methyl oleate, and oleyl alcohol from readily available commercial sources of these materials should be easy to carry out. In fact, the procedures described parallel the purification of many common and readily available organic compounds. There is little doubt that, in general, oleic acid, methyl oleate, and oleyl alcohol having a purity of 93 to 96% are eminently suitable for commercial application in organic synthesis.

Procedure I is more satisfactory for large-scale utilization than procedure II. Most important, this method gave the higher yields. Also, by distilling the commercial material at the start, a concentrate of substances below C-18 in chain length, originally present in the starting material, was easily obtained. This forerun amounted to about 20%, by weight, of the starting material. In the oleic acid and methyl oleate purifications, this lower boiling fraction may be a source of fatty acids suitable for soap manufacture

CHART III

SUMMARY OF PURIFICATION OF COMMERCIAL OLEYL ALCOHOL BY PROCEDURE I



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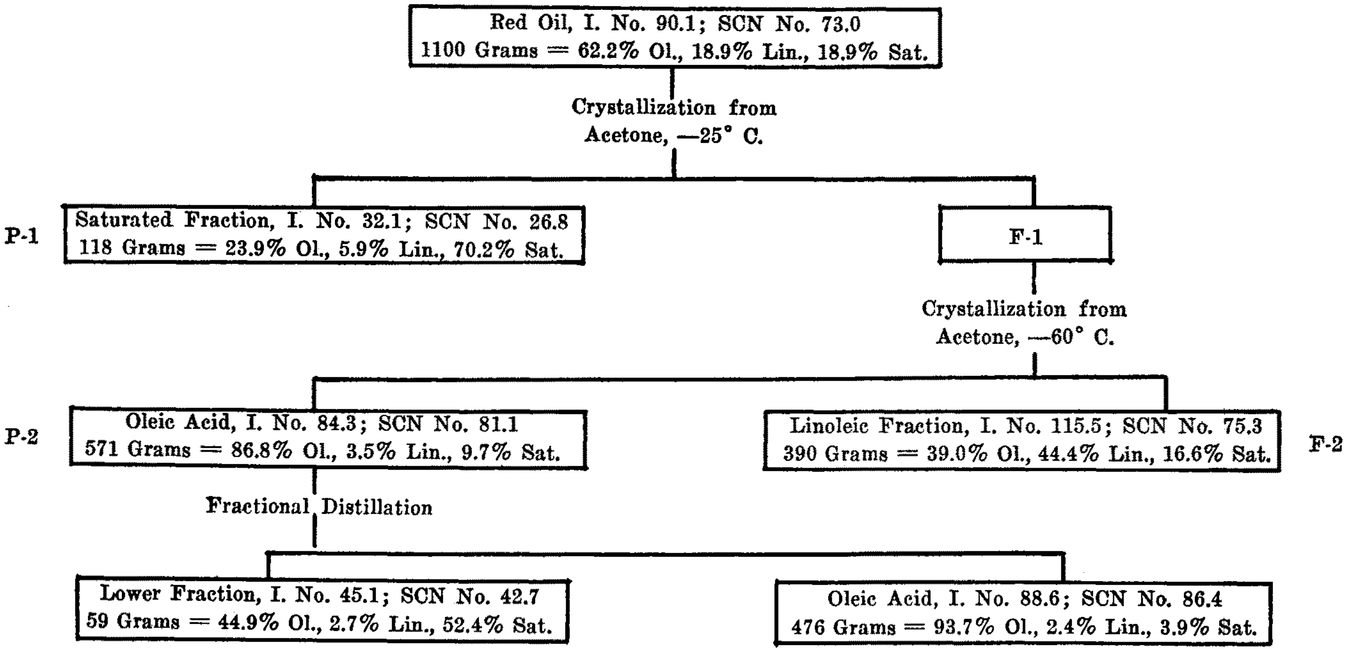
[3] The abbreviations Ol., Lin., and Sat. stand for oleic, linoleic, and saturates, respectively. P and F stand for precipitate and filtrate.

CHART IV

SUMMARY OF PURIFICATION OF RED OIL BY PROCEDURE II



(see charts I and II). When procedure II was employed with red oil, on crystallization these lower fractions were distributed in the saturated fraction, the oleic fraction, and the linoleic fraction and were, therefore, more difficult to isolate completely. In addition, by distilling first, the C-18 distillation fraction was enriched in oleic constituent, thereby permitting the crystallization of more oleic constituent per total weight of material than if the crude substance were crystallized. This also reduced the quantity of solvent to be handled and recovered per pound of purified oleic constituent produced. Also, the crystallization of distilled materials usually produced a crystalline oleic fraction easier to filter, wash, and press dry than the crude materials crystallized directly. From an industrial standpoint this is important, since there would be no need for the washings employed in the laboratory if the mother liquor could be satisfactorily removed from the crystal cake by pressing.

When the saturated and oleic constituents were removed from the C-18 distillation fraction by crystallization (procedure I), the filtrate obtained was richer in the linoleic constituent than the final filtrate obtained in procedure II (compare charts I and IV). For example, in the crystallization of the C-18 distillation fraction from red oil, the product obtained from the final filtrate consisted of about 54% linoleic acid, 34% oleic acid, and 12% saturated acids. This fraction, which represented about 20%, by weight, of the total starting material had approximately the same percentage composition of fatty acids as several important semi-drying oils, and should be very valuable. For example, it has been shown recently that polymerized vegetable oil acids, such as soybean oil acids or their derivatives, from which the monomeric acids have been substantially removed, may be reacted with polyhydric alcohols to produce rubberlike polymers (3). Also, fatty acid mixtures containing 50 to 55% linoleic acid may be used in the manufacture of alkyd resins. The utilization of this fraction, as well as the distillation forerun, should go a long way toward placing a process of this type (procedure I) on a commercial basis and should serve to reduce the cost of the purified oleic acid. It should be possible by additional crystallizations from acetone or other suitable solvent to increase considerably the linoleic acid content of this fraction (5). The linoleic fraction from red oil was light brown; most of the colored materials in the C-18 distillation fraction were concentrated here. A rapid straight-run distillation removed most of the color and yielded a pale-yellow product. When a light-colored commercial oleic acid was used as the starting material, a light-colored linoleic acid-rich fraction was obtained directly without redistilling.

The advantages of procedure II over procedure I were that (1) less time was required to prepare the purified materials (on a laboratory scale) and (2) the purity of the oleic constituent was usually slightly higher. The most time-consuming part of the purifications was the fractional distillation step. When the starting materials were first crystallized, considerably less material had to be distilled. Unfortunately, procedure II was not as generally applicable as procedure I, since its success was dependent upon the nature of the starting material. The success of procedure II required that the starting material be rich enough in oleic content to yield an oleic fraction which could be filtered satisfactorily. The percentage content of oleic constituent required varied for each substance and also was dependent upon the nature and quantity of the impurities present, especially the linoleic constituent. High concentrations of C-18 saturated materials did not seem to interfere, since they could be easily and effectively removed in the first stage of the crystallization process. For example, in this work the commercial oleyl alcohol crystallized and filtered very well. Another sample of commercial oleyl alcohol (61.3% oleyl alcohol, 9.7% linoleyl alcohol, and 29.0% saturated alcohols) also crystallized very well, even though the oleyl alcohol content was low and the per-

TABLE I

Properties of Starting Materials and Properties and Yields of Purified Products
(See Charts I to V for Properties Not Shown)

Starting Material	Color [1]	Acid Number		Saponification Number		Purification Procedure	Final Product					
		Found	Theor.[4]	Found	Theor.		Purified Material	Yield [2] %	Melting Point [3]	Color [1]	Acid Number	Saponification Number
Red oil	80Y 4R	188.5	198.6	190.5	198.6	I	Oleic acid	69.5	10.2-11.6° C.	0.5Y 0.0R	197.8	198.5
Red oil	80Y 4R	188.5	198.6	190.5	198.6	II	Oleic acid	65.0	9.8-11.6° C.	0.4Y 0.1R	198.8	200.5
Red oil methyl esters	20Y [5] 4R	2.2	0	188.4	189.2	I	Methyl oleate	66.3	About —25° C.	0.1Y 0.0R	0	189.2
Commercial oleyl alcohol	3Y 0.2R	0.5	0	2.0	0	I	Oleyl alcohol	71.6	7-8° C.	0.2Y 0.0R	0	0
Commercial oleyl alcohol	3Y 0.2R	0.5	0	2.0	0	II	Oleyl alcohol	64.6	7-8° C.	0.2Y 0.0R	0	0

[1] Lovibond Tintometer, 1-inch cell. [2] Yields are based on the quantity of oleic constituent in the final product, as compared with the quantity in 1100 grams of starting material. [3] Representative samples. [4] Theoretical values are the values for the pure oleic constitutent. [5] Lovibond Tintometer, ½-inch cell.

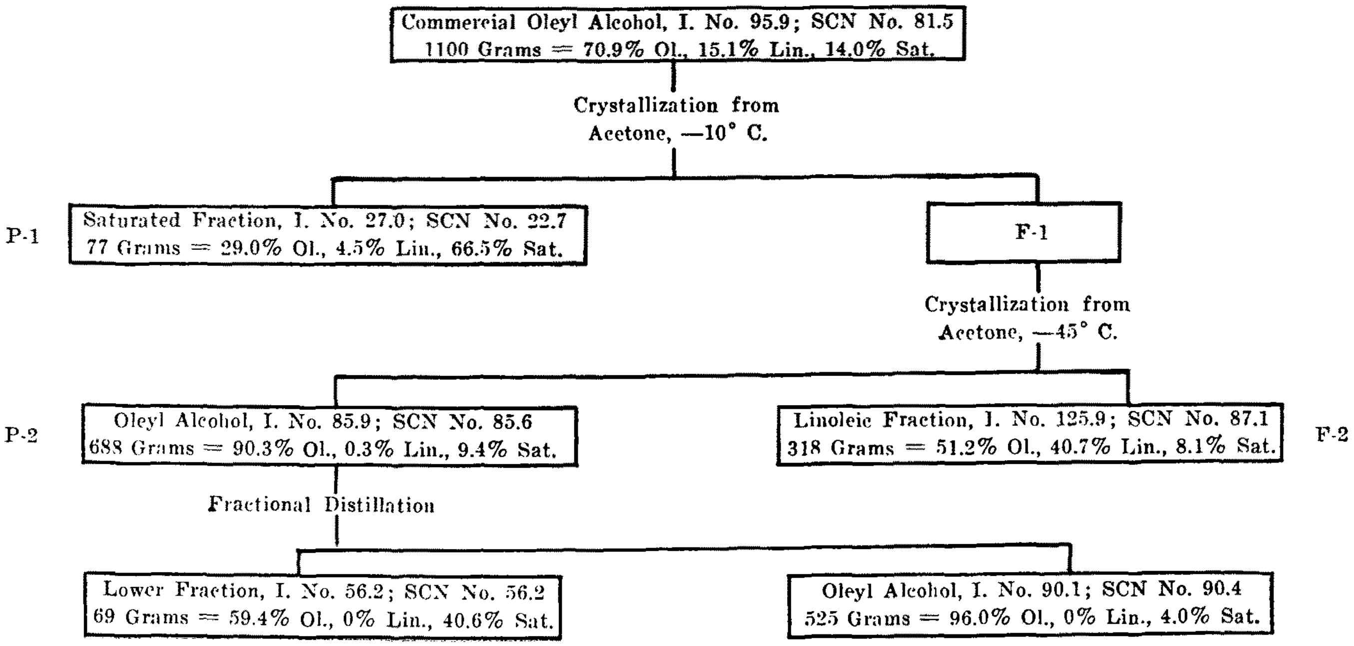
centage of saturated alcohols was high. The red oil used in this work was barely satisfactory by procedure II, whereas the methyl esters could not be processed satisfactorily by this procedure. The prior fractional distillation was very important as far as these two materials were concerned. In direct crystallizations of commercial oleic acid containing 70 to 75% oleic acid conducted in the past (procedure II), the filtrations and subsequent operations proceeded just as well as with fractionally distilled materials, and the yields were as good as those obtained by procedure I. The yields were satisfactory because large losses did not occur on crystallization, as was the case when a red oil containing 62% oleic acid was employed. An additional crystallization of the oleic acid obtained by both procedures yielded products free from linoleic acid, as determined by iodine and thiocyanogen numbers.

For certain specific uses, the procedures may be modified and certain steps eliminated. For example, when a high degree of purity is less desirable than freedom from color and obnoxious odors, it may not be necessary to remove the saturated fraction in the crystallizations, since this fraction represents a small percentage of the total material handled and contributes very little to the color and odor.

Although determinations of thiocyanogen number have been employed by oil and fat chemists for several years, it is well to emphasize that no attempted evaluation of purity of higher unsaturated fatty acids, esters, and related compounds can be considered complete without a thiocyanogen number determination under controlled conditions. Too frequently articles still appear in the chemical literature in which it is assumed that the fatty materials employed are pure because the iodine, acid, and saponification numbers

CHART V

SUMMARY OF PURIFICATION OF COMMERCIAL OLEYL ALCOHOL BY PROCEDURE II



agree closely with the theoretical values. For example, it is possible to prepare a mixture consisting entirely of linoleic acid and stearic acid which would have the theoretical iodine, acid, and saponification numbers for pure oleic acid, whereas actually it would contain no oleic acid. A thiocyanogen number determination would immediately reveal the true composition. It is suggested that specifications for oleic acid and related materials be amended to include a thiocyanogen number analysis under controlled conditions.

Acknowledgment

The authors are indebted to E. F. Jordan of the Eastern Regional Research Laboratory for the analytical data reported in this publication and for assistance in conducting the low-temperature crystallizations.

Summary

1. Oleic acid, methyl oleate, and oleyl alcohol of high purity (93 to 96%) were prepared from readily available and inexpensive commercial materials in 65 to 70% yields by fractional distillation and a single low-temperature fractional crystallization.

2. In the fractional distillation of red oil and its methyl esters, lower fractions amounting to about 20% of the starting material were obtained. These are suggested for use in soap manufacture.

3. A fraction containing more than 50% linoleic acid was obtained from the C-18 fraction of red oil by fractional crystallization. This fraction amounted to about 20% of the total starting material and had approximately the same percentage composition of fatty acids as several important semi-drying oils.

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The Isolation and Determination of Chlorophylls A and B in the California Avocado*

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Philip R. Park Research Foundation

In conjunction with the investigation of the oil soluble components of the California avocado, the identification and determination of its green pigments was undertaken. Methods for the isolation, purification, and identification of the chlorophylls have been recently developed (1-6), based largely on chromatography. However, due to its high oil content (up to 30%), the avocado presents a more difficult problem than does ordinary plant tissue. This oil rapidly inactivates the adsorbents used and thus prevents the isolation of the chlorophylls in any appreciable quantities. This difficulty was overcome by various modifications of the chromatographic method as described below. The quantitative determination of the chlorophylls was carried out spectrophotometrically on the crude avocado extract before decomposition of the very sensitive chlorophylls became appreciable (7, 8).

Experimental

Two varieties of California avocados were investigated in these experiments, the Fuerte and the Nabal. In all cases, the edible portion (whole fruit minus seed and skin) was tested. In the methods to be described the reagents used were as follows:

Methyl Alcohol, Absolute, C. P. Baker's
Calcium Carbonate, C. P. Baker's
Ether, Absolute, C. P. Baker's (diethyl)
Anhydrous Sodium Sulfate, Purified Grade
Distilled water saturated with C.P. Calcium Carbonate used in all cases
Skellysolve F, B.P. 30-60°C.
Skellysolve H, B.P. 70-100°C.
Cellite 545, obtained from John's Manville Sales Corp.

The Isolation of Chlorophyll from Avocado. All experiments were carried out in semi-darkness, the only illumination being that from a 25-watt tungsten filament lamp which was used in such a manner that the apparatus could be manipulated, yet would not receive any direct light.

Approximately 50 grams of ripe avocado (edible portion) was weighed into the micro cup of the Waring Blendor; 0.5 gram of $CaCO_3$, and 100 ml. of CH_3OH were added. The mixture was blended for approximately 30 seconds. If the avocado is not properly ripe this short blending time may not bring about a complete breakdown of the tissue. In this case the blending time should be increased to as long as five minutes. Sufficient ice was added directly to the mixture from time to time to keep the temperature at all times below 20°C.

To the completely broken down avocado mixture was added 15 grams of Celite 545, and 50 ml. of CH_3OH. This was mixed by blending for from 10 to 20 seconds and then filtered through rapid flowing qualitative filter paper on a buchner funnel with vacuum. The filter cake was removed from the funnel, separated from the filter paper and placed again in the blendor cup. The filter paper was placed in the buchner funnel for the next extraction.

One hundred ml. of CH_3OH and 50 ml. of skellysolve F were added to the cup, the mixture blended for 30 seconds, filtered as before, and the filter cake placed again in the blendor cup. These extractions were continued until the last extract came through the filter colorless. Usually a total of five extractions was sufficient.

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* Supported by a grant from Avocado Growers of California.