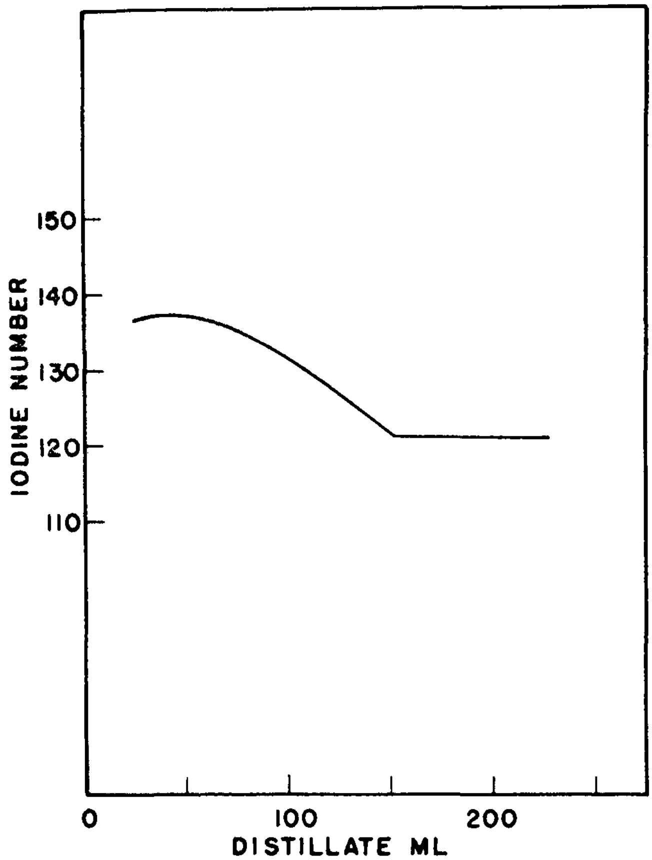

FIG. 8. Fractional distillation of a methyl linoleate-methyl oleate mixture.

chain length will ever be fractionated by a straight distillation. However, as commercial columns are improved we may look forward to unvarying chain length in commercial fatty acids wherever this is desired.

Summary

Important factors in the construction and operation of laboratory fractionating columns are discussed. These include column diameter and insulation, packing, pressure drop, effect of reflux and molecular weight on fractionating efficiency, reflux regulation, choice of operating pressure, constant pressure maintenance, and accurate measurement of pressure. Accurate boiling points in the 1-20 mm. range are reported for the first time for methyl esters of myristic, palmitic, stearic, oleic, and linoleic acids. Heats of vaporization are given.

Two types of columns, representing two stages of development in column design, are compared with respect to fractionating efficiency, and experimental results are given for the distillation of 50-50 mixtures of palmitic and stearic, stearic and oleic, and oleic and linoleic methyl esters through a Podbielniak column.

BIBLIOGRAPHY

1. Longenecker, H. E., Oil and Soap *17*, 53 (1940).
2. Podbielniak, W. J., Technical Bulletin.
3. Marshall, M. J., and Sutherland, B. P., Ind. Eng. Chem. *19*, 735 (1927).
4. Tongberg, C. O., Quiggle, D., and Fenske, M. R., Ind. Eng. Chem. *26*, 1213 (1934).
5. Norris, F. A., Russoff, I., Miller, E. S., and Burr, G. O., J. Biol. Chem. *139*, 199 (1941).
6. *Ibid.* *147*, 273 (1943).
7. Morton, A. A., "Laboratory Technique in Organic Chemistry," (1938), (McGraw-Hill Company).
8. Ralston, A. W., Selby, W. M., Pool, W. O., and Potts, R. H., Ind. Eng. Chem. *32*, 1093 (1940).
9. Pool, W. O., and Ralston, A. W., Ind. Eng. Chem. *34*, 1104 (1942).
10. Whitmore, F. C., and Lux, A. R., J. Am. Chem. Soc. *54*, 3448-56 (1932).
11. Longenecker, H. E., J. Soc. Chem. Ind. *56*, 199 (1937).
12. Klem, A., Nature, *142*, 616 (1938).
13. Weitkamp, A. W., and Brunstrum, L. C., *Oil and Soap 18*, 47 (1941).
14. Robinson, C. S., and Gilliland, E. R., "Elements of Fractional Distillation," (1939), (McGraw-Hill Company).
15. Baker, R. H., Barkenbus, C., and Roswell, C. A., Ind. Eng. Chem. (Anal. Ed.) *12*, 468 (1940).
16. Podbielniak, W. J., Ind. Eng. Chem. (Anal. Ed.) *13*, 639 (1941).
17. Hall, S. A., and Palkin, S., Ind. Eng. Chem. (Anal. Ed.) *14*, 807 (1942).
18. Selker, M. S., Burk, R. E., and Lankelina, H. P., Ind. Eng. Chem. (Anal. Ed.) *12*, 352 (1940).
19. Lecky, H. S., and Ewell, R. H., Ind. Eng. Chem. (Anal. Ed.) *12*, 544 (1940).

The Fatty Oil Industry in Canada[1]

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THE function of the Oils and Fats Administration in Canada is to organize, control, and be responsible for supply, distribution, and price control of all fatty oils and products manufactured therefrom including soaps, detergents, protective coatings, fatty food products, and a variety of finished goods and materials too numerous to mention. It is, therefore, analogous to a combination of the functions of the sections of the WFA, OPA, and the WPB dealing with fats and their products.

Before I attempt to deal in detail with my subject, I should remind this audience that in order intelligently to convert the Canadian statistics to the like United States position, you must multiply our returns by 12 in order to arrive at a comparable figure.

When it is considered that Canada is the fourth largest producer of war supplies and equipment among the United Nations and that the energies of half of the working population are absorbed by the war effort, the implications of the tremendous change which has taken place in the Canadian economy due to the impact of war can be better appreciated. This gathering will understand that this enormous industrialization program must inevitably be paralleled by an increase in fatty oil consumption both in industry and in the dietary. Industrial demands of fats for non-food purposes are estimated to have increased at least 60% above the pre-war level (1935-1939) and while the dietary requirements are strictly controlled, there is a constant pressure induced by increased purchasing power, longer working hours, and greater employment.

Under normal circumstances Canada imported over 50% of her oils and fats requirements, exclusive of butter. This is a considerable import requirement, and in a global conflict it is a serious threat to a balanced economy and to the war effort. Unlike our enemies, Canada and the United States were not pre-

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pared with large stockpiles and internal adjustments to meet the velocity and scope of the conflict. Considerable difficulty has been experienced in meeting the essential requirements of the country geared to the production of materials of war on a large scale, and these circumstances have necessitated the development of new sources of off-shore supplies, the adjustment and control of our imports, and the stimulation of domestic production.

NOT least among the outstanding wartime developments in Canada has been the rapid and pronounced expansion of the oils and fats industry. This expansion is directly the result of a long-time necessity for greater self-dependence, the impetus being given when the former Far Eastern sources of supply were no longer available and other off-shore supplies prohibited due to lack of shipping. In Canada the measures to establish an equilibrium in the oils and fats program were undertaken in 1941, and the co-ordinated efforts of government and industry have unquestionably obviated a fat famine in Canada.

The pattern taken to stabilize our position involved:

1. Expansion of production
2. Control of imports and exports
3. Bulk purchasing
4. Allocation to industry
5. Co-ordinated subsidy and price control.

I shall endeavour to deal with these phases briefly in this paper.

Prior to the war Canada was oblivious to the fact that notwithstanding that she was an agricultural nation, she was unable to meet, even partially, her fatty oil requirements. True, we have always produced a small quantity of flaxseed, but even this crop fell into disrepute with a producer who is wholly wheat-minded. Therefore, in conjunction with the Dominion Department of Agriculture, it became expedient to promote the production of vegetable and animal fats in the country. Flaxseed production was increased from the 1935-1939 pre-war average of 1.5 million bushels to approximately 17½ million bushels in 1943. Unfortunately the 1944 crop of flaxseed will not be much in excess of 11 million bushels. This is particularly disappointing as the linseed oil requirements of the United Nations are in excess of supply, and the Argentine flaxseed prospects are not favourable due to drought.

The other oil-bearing crops promoted in Canada during the war are soya beans, sunflowerseed, and rapeseed. Commercial marketings of soya beans are small, and production is quite insufficient for our domestic crushing requirements with the result that we remain potential importers of United States beans. Rapeseed and sunflowerseed are new crops which were first tried out under commercial conditions in 1943. The rapeseed appears to be behaving quite satisfactorily in the Prairie Provinces, and a preliminary estimate indicates a yield per acre of approximately 700 pounds. While the 1944 acreage goals have not been accomplished, we still hope to crush at least 7 million pounds of seed. The introductory prices being paid for these crops are f.o.b. delivery point 5 and 6c per pound for sunflowerseed and rapeseed, respectively.

LIVESTOCK production in Canada has been significantly increased with the result that animal fat production is now more in line with consumption. A few figures will quickly illustrate something of the unprecedented livestock production: since 1939 hog production has increased from 4.3 million population to 8.15 million in 1943. Cattle show an increase of 15%, and sheep and lambs of 20%. Poultry has been increased by 30%.

To ensure that the fat situation derived the maximum benefit from the increase in slaughtering, it was required by government order that no sales of beef carcasses would be permitted unless all internal fats, including crotch, kidney, brisket, and cod fats, had previously been removed. As a direct result of this order, it is estimated that at least 25 million pounds of tallow have been recovered for edible purposes.

To promote further the recovery of inedible fats a national fat salvage campaign was initiated in 1942 with reasonably satisfactory results although this method of procurement involves considerable difficulties and makes heavy administrative demands. In line with government policy to ease restrictions the compulsory order requiring butchers to accept fats delivered by housewives was cancelled a few weeks ago. The salvaging of household fats has not been discouraged, but mandatory regulations are considered unnecessary.

There has been a striking growth in the inedible tallow and grease production in Canada, and it might be mentioned that the rendering industry has made definite contributions. The change in the distribution of population has benefited this industry for the concentration of population in urban centres, the development of local slaughtering coupled with the relatively good ceiling prices have resulted in profitable rendering operations. The limiting factor, however, has been the availability of suitable labour.

As a result of a variety of methods designed to promote fat supplies, the over-all production of animal fats and lard during 1943 was in excess of 200 million pounds, or more than 25% greater than the pre-war average.

Producers of marine animal oils increased their production from an average of 23 million pounds for the years 1935-1939 to 36 million pounds in 1943. The marine mammals processed in Canada include porpoise, dolphin, and seal; however, the primary sources of marine oils are derived from the pilchard and herring fisheries located on the Pacific Coast, which supply over 60% of our total marine animal oil production. In addition to the body oils there has been developed a prosperous liver oil industry. The high-vitamin oil industry is located on the Pacific Coast where the livers of the ling, halibut, and dogfish yield oils of very high "A" potency. These oils have been exported to Great Britain for food fortification purposes.

On the Eastern Seaboard we have developed a cod liver oil industry. Canada was faced with a serious cod liver oil problem when imports from the United Kingdom and Norway were largely cut off as a result of the war. The Wartime Prices and Trade Board recognized the cod liver oil requirements and directed a program which resulted in the opening of a considerable number of new plants for the production of crude cod liver oil in eastern Canada. The results of the program have been such that Canada has today satisfied her own requirements and become an exporter of cod liver oil.

IN ORDER to appreciate better the cod liver oil developments it is interesting to note that nearly 70% of the oil produced in Canada prior to the war was common cod oil suitable for use in the tanning trade and produced by sun rotting livers. Today, less than 20% of our production is as cod oil, and cod liver oil production has increased from about 500,000 pounds in pre-war years to 3,000,000 pounds at the present.

In spite of a notable increase in domestic production Canada has been obliged to draw upon Newfoundland production for additional supplies. The use of fish oils in Canada has been, to a large degree, interchangeable with vegetable oils, but they are still primarily used in the manufacture of soap, lard substitutes, in the processing of leather, and to a lesser degree in the protective coatings of industry.

The increased production of fats and oils has taxed the pre-war processing facilities to a maximum. In particular, the production of oil-bearing material has required increased facilities, and this is reflected in a substantial growth of the industry. It should be pointed out that the pre-war facilities were inadequate to meet requirements, and all mills have been working at full capacity throughout the war.

The pre-war vegetable oil crushing capacity, in terms of bushels of flaxseed, was approximately 3.9 million bushels. With the increased processing of domestic crops of flax, rape, soya, and sunflower coupled with the importation of government bulk-purchased oil-bearing materials such as copra, peanuts, palm kernels, etc., crushing capacity has been increased to approximately 8½ million bushels.

There are various trends of thought concerning the development of an oil crushing industry in Canada. Naturally, some producers of animal fats will tend to look askance at this development assuming it to be competitive in the edible fat market, but this may be offset by the fact that the industry will complement any post-war livestock program since its major function is to supply low-priced oil cake meal for feeding cattle. There is no question of the industry being competitive with the dairy industry for the manufacture and import of margarine are prohibited by law. Broadly speaking, the concept whereby the fats of animal origin are considered on the economic defensive is no longer quite true for, by simple chemical procedure, the competition between fats of all origins and types is being equated.

Just prior to the war the Canadian importations of vegetable oils varied between 200 and 248 million pounds annually, the principal imports being cottonseed, peanut, cocoanut, and palm oils. In response to the stoppage of the major part of these imports the Wartime Prices and Trade Board, empowered by the War Measures Act, established full control of imports, exports, and intra-country transactions and proceeded to enforce a system of complete allocation of fats and oils, whether processed or unprocessed, to all civilian and war uses. By specific allocation and conservation orders manufacturers were required to restrict their usage of fats and oils to specified percentages of the basic period, i.e., either the calendar year 1941 or the mean of the years 1940-1941, and to employ certain fats for specific purposes only. The striking characteristic of the Canadian controls has been that there are no unwritten exemptions, and no transaction, sale, or delivery is permitted without prior approval from the Ottawa office.

The original civilian quotas established for the more important industry groups were:

	Pct.
Shortening	80
Cooking oils, mayonnaise, etc.	80
Soap	100
Paints, varnishes	70
Linseed oil through distributive trades	70
Linoleum, etc.	70
Pharmaceutical	100
Printing inks	90

These regulations have avoided any direct consumer rationing of fats, exclusive of butter. The conservation and allocation program has had the effect of freezing the per capita consumption of fats. The requirements of the armed forces are allocated on an extra-quota basis, but proof of actual contract and details of product, etc., must be furnished before release of any fatty materials is made.

THE method of allocation and restriction is complemented by price stabilization controls which consist of placing a ceiling over the prices of virtually all goods and services. All products, including the fatty oils and products manufactured therefrom, are required to be sold at prices prevailing during the so-called "basic period" in late 1941. This price ceiling is rigorously enforced notwithstanding the fact that costs of production may have risen. The government has sometimes found it necessary to pay subsidies to enable producers to meet an unavoidable but serious increase in costs. Such a situation exists in the soap and shortening industries which are subsidized to an amount approximating 25% of the raw material cost in the case of soap and over 50% in the case of shortening. The payment of subsidies as a means of preventing price increases, or of bringing about lower prices is a very real weapon in the anti-inflation program, but it must be used very carefully as the actual cost reverts to the tax-payer, the benefits being partially offset by higher taxes.

Notwithstanding the exacting nature of the regulations and the stringency with which they have been enforced, all classes of industry have given the most loyal co-operation. It should be emphasized that there has not been a single case of infraction of the fatty oil regulations which necessitated court action. As the supply position and shortage became more acute and the squeeze due to price control tighter, it became apparent that industry would have to make the utmost use of the possibilities of substitution and interchangeability among the fatty oils. In consequence, more than normal latitude was given to scientific planning and research with the result that considerable technical advances have occurred. Among the many noteworthy achievements has been the adaptation and ingenuity of the protective coatings industry. All military equipment and materiel must be protected by a suitable protective coating, and it is the oil vehicle which imparts the real protection to the surface. These films must be tough, hard, flexible, and waterproof, and in order to conform with assembly line production methods the films must dry rapidly.

The shortage of tung oil, which was frozen for special uses for war materials, led to the development of tung oil substitutes. The annual imports of hard drying oils amounted to 5½ million pounds of tung oil and 4½ million pounds of perilla oil and, since this total represents more than 25% of the oil usage of the pre-war paint industry, the loss of these imports was keenly felt by the industry in the early stages of the war. The manufacture of a segregated, highly unsaturated polymerized type drying oil produced from linseed oil was undertaken in 1940. The by-product of this process is a non-drying oil possessing an iodine value of about 120, which is being adapted to many industrial uses and has shown possibilities in the edible trade. The use of maleic anhydride treated linseed oil has also filled the gap for fast-drying hard finishes. The manufacture in Canada of dehydrated castor oil was also commenced during the war, and this product is generally regarded by the trade as one of the most satisfactory tung oil alternatives. It is safe to say that for most general purposes the loss of tung oil is no longer a serious handicap to the industry as a whole. Approximately 88% of the drying oil used by the protective coatings industry is represented by linseed oil.

The Canadian paint industry is still heavily engaged in war work for there is no decrease in demands for protective coatings for heavy ordnance, ammunition, food containers, and other essential uses. The industry is progressive and quality-minded, and a considerable amount of thought is being given to the post-war period. It is felt that neglected repair and maintenance requirements will doubtless help the industry to find outlets for its production before the residential construction program gets under way.

THE edible fat industry is limited to the manufacture of shortening and confectionery fats since, as previously mentioned, the manufacture of margarine is prohibited. Prior to the war these products were manufactured essentially by the hydrogenation of vegetable oils. The industry was highly specialized, having a variety of hardened blended fats which were formulated to meet the requirements of specific types of trade. Today the use of vegetable oil is limited to a maximum of 50% of the over-all oil quota, and the types of oils available vary almost from month to month. Consequently, the shortening manufacturer has to show a considerable degree of versatility in his formulation.

Many shortenings contain but a small percentage of vegetable oils while certain types or brands continue to be manufactured wholly from vegetable oil depending on the method by which the manufacturer wishes to use his vegetable oil allotment. The change has required the usage of edible tallow and lard blended with hydrogenated fish oils. Notwithstanding all the difficulties experienced, the manufacturer of shortening fats has maintained an excellent standard of quality. True, difficulty has been experienced in meeting the requirements of the trades in the habit of using tropical nut fats. These confectionery fats or vegetable butters were previously manufactured from refined, deodorized cocoanut oil or palm kernel oil, both of which possess a sharp melting point of about 23 to 28 degrees C. Lauric acid oils have been in extremely short supply, and for some considerable time there was no edible cocoanut oil available. At the present time the quota for edible cocoanut oil is limited to 50% of 1941. Satisfactory substitutes for cocoanut oil shortenings have been produced by the controlled hydrogenation and special treatment of such oils as peanut and sunflower.

During the most critical period of supply when we were threatened with an edible oil famine, work was begun by the National Research Council and the Ontario Research Foundation to explore the use of linseed oil for edible purposes. This problem was also tackled in the United States, and I believe your experience parallels our own which showed that the greatest obstacle in using hydrogenated linseed oil shortening is the development of a reversion of flavour which is carried over into the cooked product and is particularly noticeable when the product is heated.

We have recently seen fit to relax our regulations respecting the use of anti-oxidants which are now permitted in lard for domestic consumption, and we are interested in the excellent work which is being conducted on this side of the line in connection with the newer and more efficient anti-oxidants.

The soap industry is the largest individual consumer of fatty oils using over 140 million pounds per annum, of which approximately 60% is animal fats. Fish oils and vegetable oils such as cocoanut, palm, and linseed and miscellaneous foots, comprise the other major fatty raw materials. In line with the over-all policy the usage of cocoanut oil has been drastically reduced.

The Canadian soap industry, which comprises 110 plants, has been fortunate in being able to maintain production at the level of 100% of the mean of the years 1940-1941. True, the industry has been obliged to conserve the lauric acid fats, but there has been a fairly constant supply of inedible tallows and greases made possible by dint of government intervention. The industry has been more fortunate than most fatty oil users because it enjoyed a high priority position during the period of glycerine shortage. The use of rosin is not quite so general in Canada as it is in the States; the primary reason being the high laid-down cost in Canada of rosin today. For example: "Nancy" grade gum rosin costs over 8 cents per pound in Canada as compared with 7½ cents per pound for No. 2 tallow.

SOAP usage for industrial purposes has increased, and among the new outlets the synthetic rubber industry must be considered the most important. Several million pounds of tallow chip soap are required in butadiene manufacture for use as wetting, dispersing, stabilizing, and lubrication agents. Canadian soapers have just received orders from the United Nations Relief and Rehabilitation Administration to supply a total of 60 million pounds of soap. This is no small order for the existing supplies and facilities in Canada, but the industry realizes that it has a role to play in meeting the obligations of the United Nations.

Technically, there has not been any particular development in the processing and engineering phase of the industry. This is, I believe, a characteristic of the industry the world over for the saponification of fatty oils is essentially the same as it was at the beginning of the century. The post-war period will doubtless see an expansion of the industry which, to remain competitive, will have to reduce production costs, and I would look towards the pressure saponification procedures as being rapid, economical, and worthy of serious review.

The manufacturer of liquid soaps has been seriously embarrassed by the glycerine recovery regulations, and by the shortage of cocoanut oil and those edible vegetable oils which are no longer permitted for soap manufacture.

The glycerine recovery requirements have now been relaxed. To meet the dearth of oil the use of linseed, and to some extent of castor oil has become apparent although neither of these oils was used for this purpose in the past. The characteristic odour of linseed oil in soaps is minimized by appropriate production measures which consist of the use of refined oil, inhibitors, and blending agents. The price of linseed oil is subsidized to the consumer and therefore does not afford an economic barrier to its use. This is not the case with castor oil which is quite costly and can only be used in products having high ceilings.

The use of tall oil for soap manufacturing has not achieved much acceptance by the trade. Two reasons may be given, namely: that the refined product is too high in price to permit its usage and the lower priced crude product is too dark and odiferous to be acceptable.

The extensive use of sulphonated oils has necessitated considerable substitution by the oil processor. During the period of castor oil shortage we have seen trade acceptance of sulphonated products from non-hydroxylic fatty oils which were not previously acceptable in place of castor. In particular, I would mention the use of sulphonated soya, sperm, and fish oils. Even the textile industry has been required to make certain adjustments and substitutions; for example, the use of olive and peanut oils as wool lubricants has given way to the use of lard and neatsfoot oils. In the production of cloth oils there has been a trend towards the use of mineral oils to which a saponifying agent has been added. Some of the specialty products may even be stabilized emulsions of fatty oil in soap solutions. The net result has been that the specialty oil manufacturer has effected considerable savings.

Substitutions have been made in many industries. In some cases temporary hardship has been felt as in the case of the stuffing grease requirements of the leather industry. As you know, ''stuffing'' grease invariably contains a high percentage of wool grease which, on account of its flat viscosity-temperature curve, is unexcelled for leather exposed to high and low temperatures, assisting the leather to retain reasonably normal flexibility. While it was not possible to duplicate the properties of wool grease, such products as hog degras and blown fish oils overcame the initial difficulty. Today we obtain our limited requirements from United States production and, in addition, we have commenced the production of a technical wool grease in one of our own plants.

In broad outline I have endeavoured to sketch for you something of our position and problems. My presentation is a failure if I have not made clear that there has been a growth of commercial and economic interest in the oils and fats industry, a field which was heretofore not regarded as particularly important to the Canadian economy. However, due to the war emergency Canada has embarked on a series of industrial developments, and it remains to be seen how much of this development we shall be able to maintain and nourish to economic maturity.

A Spectrophotometric Method for Differentiating Between Lard and Hydrogenated Vegetable Oils

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ON SEVERAL occasions this laboratory has experienced a need for a simple and direct means of determining whether a given sample of fat was lard or a hydrogenated vegetable oil. The ordinary chemical tests are of little value in this regard although certain specific or semi-specific tests have been developed which are of some assistance as supporting evidence. The Halphen test has been devised for the identification of cottonseed oil; but lard from animals fed on cottonseed meal will give a positive reaction (14, 15) and hydrogenated oil reacts only slightly or not at all (14). The Villavecchia test for sesame oil has been used successfully as a specific test (14, 15), but it is of value in regard to this one oil only. The cholesterol and phytosterol identification tests (15) seem to be the nearest chemical approaches to the solution of the general problem of distinguishing vegetable fat from lard; but the methods are tedious and time-consuming.

The presence of small amounts of tetra-ethenoid acids (principally arachidonic) in swine depot fat has been reported by various workers (4, 6, 7). These acids containing four double bonds are not found in vegetable oils except in the oil from *Parinarium laurinum,* which is not a commercially important oil. Hilditch stated that this is the only known instance of a vegetable fat containing a tetra-ethenoid acid (6), but Brice *et al.* have recently reported the presence of a tetra-ethenoid acid in perilla oil (3). Arachidonic acid has also been reported as present in the fat from man (5), rat (16), and ox (8), as well as in human milk fat (1). A simple method, then, for the identification of such components would be of considerable value in establishing the source of the fat.

The chemical detection of small amounts of the unsaturated acids containing 3 or 4 double bonds on the basis of their bromination products is difficult, and the results are frequently open to question. The separation of small amounts of these ''polybromides'' into their hexabromide and octabromide components is still more difficult and unreliable. The solution of this type of problem has been greatly facilitated through the use of ultraviolet spectrophotometric studies of the alkali-isomerized soaps of the fatty acids in question. It has been known for several years that treatment of fats with strong alkali at high temperatures produces a conjugated system of double bonds in the polyethenoid components and that these conjugated systems exhibit characteristic absorption spectra in the ultraviolet region (2, 9, 10, 12, 13).