values by the use of suitable solvent systems. Numerous reactions of fat analysis have been carried out on paper, *e.g.*, the formation of characteristic metallic soaps, the addition of halogens and mercury acetate, the catalytic reduction, the saponification of glycerides as well as the determination of the Dien Value, the Iodine Value, using radio-active isotope iodine 131, and the Acid Value with the help of cobalt 60. Photometric and electrometric methods have been employed for the quantitative evaluation of the paper chromatograms. Thus it is now possible to carry out the qualitative and quantitative analysis of the fatty acid components of most of the oils and fats even when the amount of the sample to be analyzed is extremely small, *e.g.*, a single soy bean or a single kernel of sunflower seed. The separation glycerides and recently the surface chromatography have been tackled very satisfactorily, also the paper-chromatographic analysis of detergents with the help of the "Transparency Method." With the help of allyl-ester and urethane methods it has been possible to analyze the long-chain wax alcohols and wax acids so that a method for the exact analysis of wax has been developed for the first time. Among the optical methods infrared spectography has been taken up and will be investigated and developed through the systematic study of polyunsaturated acids.

On the preparative side, numerous compounds have been synthesized, namely, fatty aldehydes, the chlorides, and amides of polyunsaturated acids, alkine acids as well as new glycerides, *e.g.*, conjugated fatty acid glycerides and mixed glycerides of amino acids. The synthesis of long-chain fatty acids and their derivatives has been carried out through the addition of olefines to the fatty acid chlorides with the formation of halogen-alkyl ketones as the intermediates.

In the field of detergents increased attention has been paid to the study of their physico-chemical properties. The study of autoxidation and polymerization has been carried forward, especially under the influence of new types of catalysts. The adducts which are formed during the copolymerization of drying oils and cyclopentadiene have been isolated in the pure form.

In the sphere of biology of fats, conjugated oils and especially the glycerides of parinaric acid were succesfully examined in the study of fat absorption. It has been established that the fat administered through the rectum is also absorbed.

From the technical point of view the interesterification, which had been already exploited during the war and catalytic hydrogenation were studied. A new process has been developed, through which the hydrogenation is carried out economically in a continuous way and so that no iso-oleic acids are formed. In surface coatings the Chemische Werke Bayer, Leverkusen, have brought a new type of basic material, the Desmodur-Desmophen-finish into the market. It is a product of condensation of iso-cyanates with polyalcohols to form polyurethanes and is an example of successful industrial research, results of which have been often kept secret for obvious reasons.

The second edition of the "Deutsche Einheitsmethoden zur Analyse von Fetten und Fettprodukten" (standard German methods for the analysis of fats and fat products) was published in the form of a looseleaf book. The journal "Fette-Seifen-Anstrichmittel" has appeared in a substantially more extensive form. Beyond the analysis of fats and fat products a new work, "Analyse der Fette und Fettprodukte" (Springer-Verlag, Berlin) was published last year.

Fat research in Germany, in spite of all the difficulties, has endeavored to make up for the deficiencies caused by the war and its consequences. It has been desired to establish contacts with research workers in foreign countries through participating in various international conferences, *e.g.*, the Union Chimique Pure et Appliqué ("Jupak"), Association Internationale de la Chimie Industrielle, the Commité International de la Detergence," and the "International Society of Fat Research." We are especially happy over our friendly relations with the American Oil Chemists' Society, and I would like to offer you, once again, our congratulations on the Golden Jubilee Celebration of your society and best wishes for further successful work.

An Historical Survey of Fats and Oils Research in Canada

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ORGANIZED fats and oils research in Canada began at the Fisheries Experimental Station at Prince Rupert, B.C., in the late 1920's under the guidance of H. N. Brocklesby, now a consulting chemist in the United States. A year or so later the Ontario Research Foundation opened a fats and oils laboratory under the direction of A. D. Barbour, now deceased. Laboratories in other organizations were started during and since World War II.

Fats and oils research in Canada is influenced by our dependency on imports for most of our edible vegetable oil requirements. This caused a serious situation during World War II when many sources of supply were cut off. Greater self-sufficiency in fats and oils production has been an important objective for some years.

Our huge wheat surpluses also influence research. It has been realized for years that an alternative crop to wheat is desirable. Edible oil seeds are the obvious answer as they would provide much-needed oil while relieving the wheat situation. But it is not easy to make a change. Wheat is comparatively easy to grow as it matures in about 100 days. Soybean, sunflower, and safflower crops in the past have required 120–130 growing days, and, if they are to provide satisfactory alternative crops to wheat, earlier maturing varieties must be developed.

Much progress has been made by the plant breeders.

W. G. McGregor, at present senior cerealist of the Experimental Farms Service, developed an excellent rust-resistant flax. Better soybean varieties have been developed, and this crop has increased from 200,000 bu. in 1941 to 6½ million bu. in 1957. Most of the soybean crop is in Ontario, but an earlier-maturing variety is now being grown in Manitoba.

L. E. Kirk began breeding sunflowers in 1936. A crop was introduced in Manitoba during the past war. At first, rust was a serious problem, but the Experimental Farms Service distributed a rust-resistant variety in 1954, and a better hybrid will be ready for distribution in 1960. These new varieties mature early enough to be grown over a major portion of Western Canada, which is no mean achievement.

Safflower is also coming along well. Research was started in 1936. Commercial production began in 1957 and in 1958 was about 12,000 tons.

A rape crop was introduced in western Canada in 1943 as the Canadian Navy required two million pounds of the oil annually for special lubricants. This crop matures in 90–100 days, and it soon became apparent that it was well suited to Western Canada. Rapeseed acreage increased from 40,000 acres in 1945 to 679,000 acres in 1958. Most of the crop is exported.

Coincident with the great increase in oil seeds crops in the last 15 years there has been a large expansion of crushing and solvent-extraction facilities.

The quality of oils intended for edible use is important. Canadians are accustomed to the same high-quality margarine, shortening, and cooking oils as are consumed in the United States and would not welcome inferior products. Sunflower and soybean oils are satisfactory, but there is some difference of opinion as to the acceptability of rapeseed oil.

Most of the fats and oils research that has been published has been done or financed by government departments or research organizations. It has been predominantly of a practical nature but is becoming increasingly specialized and more fundamental.

Fisheries Research Board of Canada

In 1926 Dr. Brocklesby joined the staff of the Pacific Fisheries Experimental Station at Prince Rupert, B.C., one of the stations of what was then the Biological Board of Canada but later the Fisheries Research Board. An extensive study of fish oils was begun which included a) the physical and chemical characteristics of pilchard, salmon, herring, and dogfish oils; b) the possibility of using fish oils in paints and in foods, and c) their medicinal properties, particularly their vitamin content. Associated with Dr. Brocklesby were O. F. Denstedt, now professor of biochemistry, McGill University, B. S. Bailey, now deceased, F. Charnley, and later L. A. Swain, now on the staff of the University of Toronto.

Much of the early work was done with pilchard oil. It was the most important of the fish oils in Canada at that time, but in recent years the fish have not been found in commercial quantities.

Dr. Swain specialized in the chemistry of the unsaponifiables in marine oils. He developed a chromatographic method for comparing the unsaponifiable fractions of oils by successive elutions of alumina columns with light petroleum, methylene chloride, ethyl ether, and methanol. The first fraction consisted of hydrocarbons, such as squalene. The second contained cholesterol, fatty alcohols, vitamins A and D, and some pigments. The third contained such glyceryl ethers as chimyl and selachyl alcohols. The fourth fraction was small in quantity and was not identified (52).

Recently the work of the Fisheries Research Board has shifted to more fundamental studies. It is now concentrated at the Technological Station at Halifax, N. S., under the direction of F. A. Vandenheuvel. Their studies include the chemistry of marine oils, the kinetics of hydrogenation and of alkali isomerization, and the synthesis of unsaturated fatty acids. One of the projects at the Halifax station is the segregation of herring oil, which contains too high a proportion of saturated constituents to be a good drying oil and too high a proportion of highly unsaturated C_{20} and C_{22} acids to be an ideal starting-material for shortening and margarine. Vandenheuvel and Jangaard applied countercurrent solvent segregation to the methyl esters, using nitromethane. Esters with an iodine value of 126 were separated into extract and raffinate phases with iodine values of 220 and 80 (54).

In 1953 the Fisheries Research Board initiated a study of marine sterols at the Pacific Experimental Station. Idler and Fagerlund isolated a new sterol, 24-methylene cholesterol from several species of shell fish and later synthesized it (24). They isolated another new sterol, 24-dehydrocholesterol, from the barnacle *Balanus glandula* and also synthesized it (13).

Much of the work of the Fisheries Research Board as well as other information is contained in Bulletins 37, 59, and 89, published in 1933, 1941, and 1951. The last edition, "Marine Oils with Particular Reference to Those of Canada," is a volume of 400 pages and a standard reference book. Much credit is due to the Board and to Brocklesby, Denstedt, Bailey, Carter, and Swain for these publications.

The Ontario Research Foundation

The oils and fats laboratory at the Ontario Research Foundation has been concerned with vitamin assays, nutritional problems, hydrogenation of oils, infrared studies, and projects financed by industries and government organizations on a contract basis.

Dr. Barbour was one of the first to study the digestibility of iso-oleic acid. After feeding hydrogenated cottonseed oil to rats, the depot fat contained 9% iso-oleic acid. During subsequent fasting it disappeared as rapidly as the other fatty acids (2).

Because of the scarcity of fats and oils during World War II the Oils and Fats Administrator of the Wartime Prices and Trade Board requested that the utilization of linseed oil for edible purposes be investigated as it was the only vegetable oil that could be produced in bulk in Canada at that time. The National Research Council of Canada initiated studies at the Ontario Research Foundation and at Macdonald College, McGill University, seeking a solution to the flavor problem associated with the use of hydrogenated linseed oil in shortenings. Both laboratories collaborated closely with the fats and oils laboratory of the Division of Applied Biology of the National Research Council in Ottawa.

We did not find a remedy for the severe reversion problem at the Ontario Research Foundation. However during the critical period a fair amount of hydrogenated linseed oil found its way into shortenings intended for use in bread.

In the course of the work it was found that when linseed oil was hydrogenated, an isomeric linoleic acid, termed isolinoleic, was formed by the hydrogenation of the 12:13 double bond of linolenic acid. Linseed shortenings contained 15–20% of this iso-acid. Strong evidence was found that its breakdown was one of the causes of the unpleasant flavor deterioration of hydrogenated linseed oil (27, 28, 30, 37).

It was concluded from infrared studies in our laboratory that one of the double bonds of isolinoleic acid has a *trans* configuration (29).

After the war autoxidation and flavor deterioration studies were continued for a time with the help of Mrs. Ruth Knapp and Miss Elizabeth Kirby. Changes in infrared spectra accompanying polymerization and autoxidation were studied (31, 32).

Macdonald College, McGill University

During World War II W. D. McFarlane and his group at Macdonald College also studied flavor reversion in linseed-oil shortenings. Armstrong and McFarlane concluded that a derivative of linolenic acid was the cause of the trouble (1). Privett, Pringle, and McFarlane found that hydrogenation of the acetone extract of linseed oil polymerized in an atmosphere of carbon dioxide yielded a shortening that did not revert in flavor (44). But Crampton and Millar, also at Macdonald College, found that a high incidence of death occurred when this material was fed in a 28-day test to rats. Further tests by Crampton, Farmer, and Berryhill confirmed that the nutritive value of some oils was lowered by heat-polymerization at 275°C. (11).

An extensive study to determine the nature of the damage to the oils by heating was begun at Macdonald College under the direction of Common and Crampton. Ethyl esters of the heat-treated oil showed the same adverse effects as did the triglycerides. Neither the distillate nor the residue obtained by distilling the esters supported growth. On separating the distillate into adduct-forming and nonadduct-forming fractions with urea, it was found that the former, consisting of straight-chain monomers, was as nutritious as esters prepared directly from normal unheated oils. But the nonadduct-forming fraction (N.A.F.D.) was "toxic," and its harmful effect was measurable when it comprised as little as 2.5% of the diet. The effect was ascribed tentatively to "cyclized" monomeric esters.

An association between toxicity of the N.A.F.D. fraction and the presence of polyene acids in the original oil was demonstrated. The fractions from menhaden and linseed oils were the most toxic, that from soybean oil less so, and that from sunflower seed oil only slightly injurious (6, 8, 9, 10).

Wells and Common of Macdonald College (55), and Macdonald (38), and later McInnes of the National Research Council Laboratories studied the chemical nature of the N.A.F.D. fraction. The presence of C_{18} diunsaturated cyclic (six-membered) fatty acids was established.

The National Research Council Laboratories

Three of the laboratories of the National Research Council are active in the oils and fats field, one in the Division of Applied Biology, Ottawa; one in the Division of Pure Chemistry, Ottawa; and one at the Prairie Regional Laboratory in Saskatoon, Saskatchewan.

a) *Division of Applied Biology.* The fats and oil laboratory of the Division of Applied Biology was started during the past war and was involved with a variety of problems concerned with the war effort. At that time the use of animal or marine oils in edible products was mandatory as the use of vegetable oils by manufacturers was limited to a maximum of 50% of their over-all oil quota. Lips, Grant, and White were concerned with the stability of animal fats and the use of antioxidants. Dr. Lips made a survey of Canadian lard.

Later, when it became apparent that the rapeseed crop was particularly suitable for the western prairies, N. H. Grace and colleagues made a study of the utilization of the oil (14, 15, 16, 35, 36, 57).

R. P. A. Sims began a fundamental study of polymerization about 1950, following up the work of Crampton and Common at Macdonald College. A dilution-polymerization technique permitted the separation of inter- and intraglyceride reactions. The kinetic orders of the reactions were provisionally established. Analyses of the polymerized oils showed that they contained substantial amounts of trimeric and higher polymeric fatty acids (46, 47, 48, 49).

Dr. Lips studied the stability of tocopherol toward oxygen in stable solvents and in methyl oleate. The rate of tocopherol destruction varied with its concentration and with the stability of the medium (33, 34).

More recently, Morris Kates has been studying the hydrolysis of lecithin and other phosphatides by enzymes present in various plant tissues, the isolation, separation, and identification of plant phosphatides, and phosphatide metabolism in leaves by using the isotope tracer technique (12, 26). Tattrie, Bailey, and Kates investigated the action of lipase on stereoisomeric forms of oleoyl-α,β-dipalmitin and found that the enzyme has no stereospecificity towards triglycerides (53).

b) *Division of Pure Chemistry.* C. Y. Hopkins found that one of the *Cruciferae* oils contained a substantial amount of 11-eicosenoic acid in the glycerides (17). This led to a general recognition of its occurrence along with erucic in seed oils of rape, mustard, and other *Cruciferae.* Further work has demonstrated its presence in at least six plant families, also in cod liver and dogfish liver oils, and in white whale blubber (18, 19, 20).

Chisholm and Hopkins have shown the presence of epoxyoleic acid in some species of *Malvaceae.* They have succeeded in isolating 12,13-epoxyoleic acid (5, 21).

Hopkins and Chisholm studied the relation between dietary fat composition and nutritive value in collaboration with Beare, Murray, and Campbell of the Food and Drug Laboratory in Ottawa. On feeding pure methyl esters of oleic, eicosenoic, and erucic acids to rats at the 5% level, they found that the acid residues were deposited in the body fats in substantial amounts but that erucic acid was deposited in lesser amounts than the other two acids. Growth rates were not significantly different (22, 42).

They determined the optimum ratio of saturated to mono-unsaturated fatty acids in rat diets, keeping linoleic acid constant at 10% of the total fatty acids. Fat mixtures in which the remainder of the acids were 30% saturated and 60% mono-unsaturated gave the highest rate of growth with male rats (23, 41).

c) *Prairie Regional Laboratory.* In 1946 a fats and oils laboratory was opened at the new Prairie

Regional Laboratory at Saskatoon. It incorporated the Oil Seeds Laboratory of the University of Saskatchewan, established a few years earlier. H. R. Sallans, while in this laboratory, made a study of Canadian linseed.

In a study of solvent segregation Youngs and Sallans found that useful fractionations of soybean and linseed oil could be made by using as solvent 100 parts of acetone with 3 to 7 parts of water (56).

One of the important functions of the Prairie Regional Laboratory is to determine the composition of oils from prairie crops and from strains developed by the plant breeders. Fractional distillation procedures have been used, but recently B. M. Craig introduced gas-liquid chromatography as a tool for these analyses with considerable success. Craig and Murty synthesized two plasticizers for use as liquid phase by esterifying adipic and succinic acids with diethylene glycols containing 1% by weight of diglycerol to provide cross-linking. Good separation of palmitic, stearic, oleic, linoleic, and linolenic methyl esters was obtained (7).

Barry and Craig and Porck and Craig have developed methods for the synthesis of symmetrical diglycerides (3, 43). Craig has also conducted extensive studies in the field of dilatometry.

Lemieux and von Rudloff discovered that olefinic double bonds are readily oxidized in an aqueous solution of periodate. Conditions were found which permit the quantitative oxidation of unsaturated fatty acids to the expected end-products (45).

The Food and Drug Laboratories, Department of National Health and Welfare

The research done by the Food and Drugs Laboratories is chiefly related to the development of methods required for the enforcement of the Food and Drugs Act.

J. H. Mahon and R. A. Chapman developed methods for the estimation of antioxidants in lard and shortening and for detecting adulteration of butter with vegetable oils (39, 40).

The increased production of Canadian rapeseed oil and its possible incorporation into food in Canada led Joyce L. Beare, T. K. Murray, and J. A. Campbell to carry on extensive feeding tests with rats. When compared with corn oil, rapeseed oil caused a significant decrease in body weight gains and in food consumption but only when fed at the highest levels (16 and 20% of the total diet) (4). It is unlikely that human consumption of a vegetable oil would approach these levels.

The Department of Agriculture

Most of the research in lipides in this Department has been done in the Chemistry Division of Science Service in Ottawa.

In the Animal Chemistry Unit Migicovsky, Wood, and Scaefe are intensively studying the mechanism of cholesterol synthesis. One phase of this is the effect of dietary oils and fatty acids on cholesterol metabolism in the rat.

A new oils and fats laboratory was established in the Plant Chemistry Unit a few years ago and Dr. Sims, formerly of the National Research Council, is in charge of it. He is investigating lipide biosynthesis in plants.

The Universities

Little work has been done in the universities on fat chemistry as such. Quite a lot of research on the metabolism of lipides has been carried on and is under way, for example the work on brain lipides by Dr. Rossiter's group at the University of Western Ontario, Dr. Best's work at the University of Toronto on lipotropic factors, Dr. Beveridge's work at Queen's University on the relation of lipides and fats to atherosclerosis, and Dr. Carroll's work at the University of Western Ontario on the effects of erucic acid and of oils containing erucic acid on the cholesterol content of the adrenals and other tissues of rats.

R. N. Jones of the National Research Council Laboratories and R. G. Sinclair and A. F. McKay of Queen's University, Kingston, Ontario, found that the infrared spectra of saturated fatty acids show much more structure when they are in the crystalline state than when they are in solution. A progression of absorption bands of uniform spacing and intensity was observed between 1180 and 1350 cm^{-1} in the spectra of saturated fatty acids in the solid state (25, 50, 51).

The Canadian Committee on Fats and Oils

During World War II a number of us who were concerned with problems arising from the shortage of edible oils met together informally from time to time to discuss our work. In December, 1944, this group was organized as a subcommittee of the Canadian Committee on Food Preservation, a committee of the National Research Council of Canada. The growth of the subcommittee led to the establishment in March, 1952, of an independent committee, commonly known as the Canadian Committee on Fats and Oils. Business meetings are held once a year, and one-day conferences or conventions every two years.

Acknowledgment

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Progress in the Chemistry of Lipides in South Africa in the Past Fifty Years

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WITH THE UNIFICATION of South Africa in 1910 the country passed from the heroic period to one of steady progress and economic growth. Government research laboratories, concerned primarily with agriculture and mining were first set up, and during the following 10 years many fully-fledged universities were established. These new institutions made possible a slow but steady increase in scientific research and provided the scientific personnel who have played so great a role in both the economic and scientific development which has taken place in recent years.

In 1945 the Council for Scientific and Industrial Research was set up, and since then rapid progress has been made in the establishment of a large number of scientific laboratories and research institutes to serve special industries and needs.

In 1909, where this survey begins, there was very little systematic research in South Africa. Investigations in fat chemistry were chiefly concerned with the casual investigation of indigenous plants as sources of vegetable oils suitable for burning and soap-making. Many seed fats and waxes were analyzed in a rudimentary way by analysts in the Cape Colony by the Welcome Chemical Research Laboratories, England, but chiefly at the Imperial Institute, London.

Lipide research really began in South Africa with the work initiated by W. S. Rapson, then at the University of Cape Town, now vice president of the Council for Scientific and Industrial Research, Pretoria. During a period of about 10 years W. S. Rapson and his associates carried out an extensive survey of the lipides, principally of fish caught off the South African coast. These investigations, reported in some 33 papers, covered the seasonal variation in yields of fish oils and the physical and chemical properties of these oils. Extensive use was made of the Hilditch ester fractionation technique in the determination of the composition of the fatty acid fractions, and computation forms relating to this method were devised to simplify the calculation of the results (1). The difficulty of getting accurate unsaponifiable determinations with a number of marine oils was traced to the presence of large proportions of glyceryl ethers, and the effect of the presence of these substances on the accuracy of the S.P.A. method was examined. A method of determining these substances by means of periodate oxidation was also developed.

Particular importance was placed on the occurrence, distribution, and determination of vitamin A, and these investigations led to the rapid commercial exploitation of fish liver oils for the manufacture of vitamin A concentrates on a large scale (2, 3). The work also led to the manufacture of a wide range of products, particularly drying oils for paints. Commercial production of fish oils began in 1946 and four years later had reached 11,000 tons a year. At one factory a propane segregation (Solexol) plant was set up (4) to produce high vitamin A concentrates and to upgrade marine oils by removing color bodies