Robert Ackman: 1980 Kaufmann Memorial Lecturer

As a young boy, Robert Ackman used to help shine the buttons on his father's Royal Canadian Mounted Police uniform. Maybe that's why he developed the attention to detail that has characterized his career as a lipids researcher. Personally, he thinks that the cod liver oil to which he became addicted as a toddler played a latent role in shaping his later interests in marine oils and lipids.

In the Kaufmann Memorial Lecture that he delivered during the ISF/AOCS World Congress, Bob called for less costly analytical methods and more precise research reported in more concise publications (see pages 821A-829A of this issue of *JAOCS*). Instrumental analysis of fatty acid composition has been the primary focus of his research career.

Bob was born in 1927 in Dorchester, New Brunswick. Natives of New Brunswick are called "Herringchokers," he notes, because of the local seafood, whereas natives of neighboring Nova Scotia, where he now resides, are referred to as "Bluenoses." Bob says no one is absolutely sure of the origins of the latter term. His father was an officer in the New Brunswick Provincial Police until the depression, when the provincial police were incorporated into the federal Royal Canadian Mounted Police to ease the financial drain on hard-pressed local governments. Until about 1930, Bob held dual citizenship in Canada and United States (in the U.S. because maternal relatives had been U.S. citizens), but at that time, new rules required his parents to choose one or the other, and naturally they chose the land of his birth.

During his elementary school years, Bob was in a new school every two years as his father was reassigned to a new town within New Brunswick—"That was to keep the police from getting too friendly with the locals, you see," he says, "Especially those with a penchant for then currently popular types of free enterprise." When he was in the fourth grade, Bob received the gift of a chemistry set. The immediate results were some spots on the ceiling, some pungent odors and a few other minor calamities. The long-term result was that Bob's interest in science and chemistry had been piqued, an interest later nurtured by a good high school science teacher in Ontario.

After high school, Bob faced a choice of going to university and taking science, or of pursuing a promising career in pharmacy. He had been working for a pharmacist who had visions of his talented protege winning the College of Pharmacy gold medal, thus reflecting great honor on the pharmacist. To his employer's disappointment, Bob decided to go into science, enrolling in the honors course of math, physics and chemistry at the University of Toronto.

Bob began college in the fall of 1946 along with about 15,000 other students on a campus equipped to serve about 5,000 students. World War II had ended and Canadian veterans were flooding back to universities under government sponsorship. The older age level meant there was less interest in socializing and more interest in learning. "The class as a whole was more mature, more diligent," he says, "and that may have been a stabilizing influence."

Cod liver oil came into his life again in a summer job in a tannery. He admits to nostalgia at the aroma of fine leathers dressed with this oil, but not many people know they are smelling oxidized sulfonated cod liver oil.

When he graduated in 1950, Ackman began what became a nearly career-long affiliation with the Fisheries Research Board of Canada. "It was quite an elite group," he says, adding that when the National Research Council of Canada was celebrating its 50th anniversary in 1966, the FRBC sent congratulations that pointedly mentioned it was then FRBC's 54th anniversary. Bob completed parttime studies for a Master of Science degree from Dalhousie University in Halifax in 1952. The FRBC promoted him from junior research assistant to research assistant, turning his attention full time to marine oils. Fish oils at that time were major industrial oils, used in the linoleum and paint industries, as well as for vitamins, and for hydrogenation into edible oils.

Within a year, Bob decided he wanted to study for a doctorate. One reason was that his co-workers included holders of European doctorates and he was impressed not only by their work, but also the respect their titles commanded. With a bit of library sleuthing, he discovered a little-known but prestigious two-year scholarship in Britain for which qualified graduate students in physics and chemistry from Dalhousie University were almost invariably successful. Thus, by coincidence, the profits of the famous

1851 exhibition in London funded a future scholar near the exhibition's original location in South Kensington, at the Imperial College of Science and Technology, where fatty acid research flourished under R.B. Linstead and his colleagues.

"In Toronto, it was frowned upon if you took off Saturday afternoons for a football game," he recalls. Graduate study at the Imperial College was less intensive than in the Canadian universities. He had time for championship bridge and toured Europe as part of the University of London team.

Bob met his future wife in London. She was there visiting with another young lady and he was recruited as an escort. Actually, it's a sign that maybe his attention to detail is not omniscient, in that the young Catherine McKinnon and Bob soon discovered that for a while they had lived across the street from each other in Toronto. They were married in Toronto on September 11, 1957.

After completing his doctorate in 1956, Bob returned to the FRBC as a research chemist at its Halifax Laboratory. He has been in Halifax ever since, and is currently professor of marine lipids in the Fisheries Research and Technology Laboratory at the Technical University of Nova Scotia.

During 1969, Bob was among the FRBC staffers who were working 16 hours a day, seven days a week to try to determine what was causing millions of herring to die in Newfoundland. New instrumentation, a flame photometric detector, was grafted onto his favorite instrument, the gas chromatograph, and helped Bob determine that elemental phosphorus waste from a new chemical plant was responsible. Holding lagoons were constructed to permit sludge to settle, so that the elemental phosphorus could be recycled through the plant. The new methodology Bob developed is now the method of choice for determining elemental phosphorus.

In the early 1970s, allegations were first made that cardiac necrotic lesions found in rats fed rapeseed or marine oils might also be important in man. Bob was among the many Canadian fats and oils researchers assigned to do further study. He stresses that, as research progressed, a spirit of cooperation, rather than disputation, was normal among the scientists, irrespective of policy differences deliberately engendered by others. The work rapidly accelerated the introduction of new varieties of rapeseed, now being promoted under the registered name "Canola," that are low in both erucic acid and glucosinolate content.

Bob Ackman's expertise has been available for many special projects. He has been a Canadian delegate to the Codex Alimentarius fats and oils committee; he is chairman of the National Research Council of Canada Associate Committee on Fats and Oils, an editor or editorial board member for two journals, an associate referee on fats and oils for the Association of Official Analytical Chemists, and was one of the specialists invited to the WHO/FAO's expert consultation on fats and oils in human nutrition held in Rome during 1977.

Despite his numerous activities, Bob says he is out of his laboratory no more than approximately a month each year. He chooses trips carefully, he explains, because Halifax's location on the southern side of Nova Scotia, approximately 600 miles northeast of New York City, turns almost any one-day meeting into a three-day trip.

A self-admitted "workaholic," Bob does enjoy theater, particularly Gilbert & Sullivan productions, in Halifax. He and Catherine have two daughters, 18-year-old Elizabeth and 16-year-old Margaret. Bob also is somewhat of a military history buff, and recently took a detour on one trip to Philadelphia to tour the U.S. Civil War battlefield at Gettysburg, Pennsylvania.

Bob Ackman brought his pursuit of precision and excellence to the expanding field of lipids research at a time when such characteristics were needed. The result has been a career with approximately 300 scientific publications, and an impact far beyond the realms of the Herring-chokers and the Bluenoses.

Kaufmann Memorial Lecture

Presented April 29, 1980, during the ISF/AOCS World Congress in New York City.

Potential for More Efficient Methods for Lipid Analysis

R.G. ACKMAN, Nova Scotia Technical College, Fisheries Research and Technology Laboratory, PO Box 1000 Halifax B3J 2X4, Nova Scotia, Canada

It is a particular pleasure to be invited to present the H.P. Kaufmann Memorial Lecture on the occasion of this joint congress of the International Society for Fat Research and of the American Oil Chemists' Society. I can thus look out on this audience and, as speakers presumably usually do under these circumstances, speculate on the proportion of those present who actually knew personally the distinguished scientist, Dr. H.P. Kaufmann, whose memory is preserved and honored by this memorial lecture. Today, I am sure that there are a large number present who did indeed know Dr. Kaufmann personally, and many others whose work has been influenced by the career of this outstanding scientist are no doubt in the audience.

It may seem odd or unlikely to some of you that a young scientist in a remote part of the world such as Halifax, Nova Scotia, working in the somewhat unromantic

field of fish oils, did have a personal contact, albeit a slight one, with Dr. Kaufmann. In 1956, the Halifax Laboratory of the Fisheries Research Board of Canada did not have a very large library, although fortunately Dr. F.A. Vandenheuvel, the head of the marine oils section, had seen to it that this library included *Oil and Soap*. The other chief source of information in the library was Chemical Abstracts, and in the latter were many tantalizing references to work on fats and oils published in *Fette. Seifen. Anstrichmittel.* For example, in 1956 there appeared "H.P. Kaufmann and E. Gottschalk, Über die Körperöle der Fisches Hoplostethus islandicus. II. A411."

For the benefit of some younger members of the audience, I should explain that there was indeed life before the invention of the photocopier. At that time a great deal of scientific information was disseminated by reprints, or occasionally as strips of 35 mm negatives or as photographs made from negatives. One of the advantages of a good, old-fashioned chemical education was the necessity, sadly no longer required, of some ability to read scientific German, so with confidence I bombarded H.P. Kaufmann with reprint request cards.

At that time I did not know much about the Deutschen Gesellschaft für Fettwissenschaft and was unaware that Dr. Kaufmann was also the editor of *Fette. Seifen. Anstrichmittel.* Although for a time, reprints were sent, I eventually received a somewhat short and brusque letter pointing out that if we were so interested in the work of the DGF then we had better take out a subscription to *Fette. Seifen. Anstrichmittel*! Fortunately, in 1957, an increased budget for the laboratory enabled Dr. Vandenheuvel not only to subscribe, but also to obtain the extra issues back to 1950. Thus, in my one and only direct personal contact with Dr.Kaufmann, he showed not only common sense but a degree of good business acumen of which the editors of our current journals might well take note.

For the benefit of part of the audience I should like to draw to their attention that in Munster, under the capable direction of Dr. H.K. Mangold, there is today an "H.P. Kaufmann-Institut," the "Institut für Biochemie und Technologie" of the "Bundesanstalt für Fettforschung."

A little sadly, I must close this short eulogy on H.P. Kaufmann with a thought of one of his countrymen. A noted scientist and poet wrote in *Minerology and Geology* that:

> "The history of science is science itself; the history of the individual, the individual."
>
> — *Goethe*

We must expect that most of the published works of an individual such as H.P. Kaufmann, like those of his contemporaries, and eventually of ourselves, will be made obsolete by progress, and indeed in a recent book (1) there is only one reference to work by H.P. Kaufmann. It occurred to me to make a comparison with a North American scientist from the same period and arbitrarily I picked R.W. Riemenschneider. In the same book, he is not mentioned at all. A book might not be a fair test, although in the 1947 edition of Markley (2), these two scientists were cited an equal number of times. I am pleased to report that the *Science Citation Index* for 1977 and for 1978 shows that both scientists are still being cited in the literature. It is very appropriate to recall the inspiration of H.P. Kaufmann as an organizer and teacher, but on the technical level:

Good Reasons must, of Force, give Place to Better

Shakespeare: Julius Caesar: Act IV

I should like, therefore, to demonstrate how rapid this replacement process can be with a personal example from a period when fatty acid technology seemed to be advancing at a rate unbelievable even by today's standards.

About 1957 we slowly and laboriously prepared pure ethyl oleate with all the usual crystallizations of acid, etc., starting with filbert nut oil, and finally conducting a stately and delicate high vacuum distillation of ethyl esters through a Stedman column. The product met all the specifications for pure oleate given in *Fatty Acids, Their Chemistry and Physical Properties*, by K.S. Markley, first edition (2), and of *Fatty Acids*, by A.W. Ralston (3), and of the then current literature. In 1958, we purchased a Podbielniak spinning band column apparatus for vacuum distillation and some pure ethyl oleate was to be cleaned up. It was a shattering experience to see the first distillate turn white and solid in the receiver, and to learn that the "pure" ethyl oleate contained about 5% of ethyl palmitate. By 1959, government science in Canada had progressed to the stage I had left behind at the Imperial College in 1956. The Halifax Laboratory's first gas chromatograph, a Podbielniak "Chromacon" was installed, tested, cursed, modified, and eventually used to test the ethyl oleate. The marvelous new polyester liquid phases just introduced by Orr and Callen (4) and others (5), were available for this test. In theory, the ethyl oleate, pure by spinning band column standard, could contain equal quantities of ethyl stearate and ethyl linoleate. This combination would give the correct iodine value and not modify the refractive index and other properties seriously. It was, in fact, shown that quite low levels (ca. 0.3%) of these two acids were present, but 99+% purity for the oleate was not to be sneered at in those days. To pursue the matter further, the filbert oil had been originally selected for its high content of a supposedly pure (6) oleic (*cis*-9-octadecenoic) acid. A novel (1961) ozonolysis technique showed that this was likely the case, with little or no *cis*-vaccenic (*cis*-11-octadecenoic) acid present (7), but somehow the pace of introduction of new developments in lipid technology seemed to be slowing down. It was not until 1965 that we were able to confirm by open-tubular gas chromatography that the oleic acid was, in fact, virtually 100% *cis*-9-octadecenoic acid isomer.

The decade encompassing 1955 to 1965 was a remarkable one for lipid chemists. From personal experience I can testify that at any AOCS meeting one could be sure of hearing of new advances on some point or technique, or of a change in methodology, worth starting up on the first day back at work in Halifax.

One of the most unusual aspects of the dramatic changes in lipid technology in this remarkable decade from 1955 to 1965 was the reduction in the scale of operations and sample size needed to execute most analyses of fats and oils:

1957	Stedman Column	1,000 g
1958	Podbielniak spinning band column	10 g

1959	Packed Column GLC/ Katherometer	0.001 g
1960	Open-tubular GLC/ Argon or FID	0.00001 g

In the GLC aspect, one must observe that open-tubular GLC was forecast (8) as early as 1958, practically demonstrated for methyl esters of fatty acids (9) by Lovelock in 1961, as well as used in the early sixties by a number of workers in Italy and elsewhere, but effectively, not brought to fruition until L.S. Ettre published a book on the subject (10) in 1965 and the Perkin-Elmer Corporation introduced the Model 226 gas chromatograph with metal pancake open-tubular columns (11).

In this same decade, a radical change took place from silicic acid column chromatography, with some paper chromatography, to silicic acid thin layer chromatography. Probably the appearance of the definitive book (12) by Stahl was the catalyst, and it is again important to note that this popularity stemmed not only from the efficiency of the analyses but also to the reduction in scale of materials needed. This was of the same order as in gas chromatography, so that sample sizes diminished from 0.1-1 g to 0.0001-0.01 g.

One of the problems with all societies is their inability to foresee progress. Every political party claims that it is the only group with the foresight to see the trend of future needs and developments, and to have the expertise to divert current resources into the right channels to meet these presumed needs. History proves that they are usually wrong on both counts! Our administrators and committees are in the same position and I should like to review some of the major problems resulting from progress and to consider our potential for changing a very few things for the benefit of society and of lipid scientists in particular.

A decade ago small institutions were even more acutely envious than usual of large institutions since the latter were installing mammoth computers with remote terminals and the capability for processing of data on-line or at a later time. The microchip revolution has given us dedicated minicomputers with the same capability, and at a reduced cost in both inflated and real dollars. My concern is that we will have an increase in material published which has been generated by such machines and apparently has not gone through the head of any knowledgeable party prior to publication. A very recent issue of a respected lipid journal included fatty acid data for bovine erythrocyte phospholipids. The gas chromatographic output was processed by a data processing system specified by make and model, and peaks were identified by "co-chromatography with known standards of methyl ester mixtures." According to this paper, the animals in question did not have any arachidonic acid (5,8,11,14-eicosatetraenoic) in their erythrocyte phospholipids, but apparently they did have 6 to 8% of 11,14,17-eicosatrienoic acid. This type of confusion of fatty acids coincident in certain gas chromatographic analyses is not new, and as long ago as 1968 an author (13) was faced with a biochemical conundrum: "The considerable labelling in arachidonate from linolenic acid-1-C^{14} has no ready explanation. How 18:3 (9,12,15) could become 20:4 (5,8,11,14) cannot be readily explained." Probably simple chain elongation was responsible in the latter case and the radioactivity was not in the arachidonic acid but in the coincident 11,14,17-eicosatrienoic acid.

By an ironic coincidence, a year before the appearance of this recent paper erroneously reporting 5,8,11,14-20:4 acid as 11,14,17-20:3 acid, a commercial firm published an entire page (14) on the subject, including a re-publication of their *1974* figure illustrating the problem. It is true that under certain circumstances, more efficient columns, such as open-tubular columns, will usually give the desired

The late H.P. Kaufmann was one of the key figures in the formation of the International Society for Fat Research (ISF). The H.P. Kaufmann Memorial Lecture is presented at each ISF World Congress in his honor.

TABLE I.

Comparisons of Retention Data for Methyl 11,14,17-Eicosatrienoate and 5,8,11,24-Eicosatetraenoate Predicted from That for Methyl 9,12,15-Octadecatrienoate (19)

ECL of 9,12,15-18:3	ECL of 11,14,17-20:3	ECL of 5,8,11,14-20:4	Liquid phase of approx. corresponding polarity
19.50	21.40	21.27	EGA,BDS
20.00	21.90	21.91	EGS,DEGS
20.49	22.39	22.55	Silar 10C, OV-275

separation (4,14-16), but, in fact, the problem was forecast in 1963 from packed column data (17,18) and specifically pointed out again (5) in 1969. Tabulations of retention data (such as in Table I) show how to adjust column polarity (19) or operating conditions (18) to achieve the separation of these two acids on either the packed columns commonly in use or on open-tubular columns (15,16,20).

There is generally a wider problem in even-carbon chain length overlap in gas chromatography (Fig. 1) and it is a striking commentary on the general attitude that more polar columns must always be better, that the potential confusion between 11,14,17-20:3 and 5,8,11,14-20:4 is compounded on the more polar columns by the C_{20}-C_{22} overlap. In fact, in a recent paper (16), the problem was not coincidence of 11,14,17-20:3 and 5,8,11,14-20:4, but of the 11,14,17-20:3 with 9-22:1. Basically, if the analyst is not interested in specifically identifying *trans* fatty acids, the older and less polar liquid phases (Fig. 2) may be the simplest and best for gas chromatography and reduce the possibility of erroneous data being generated for fatty acids which should be well known.

The new wall-coated open-tubular column of fused silica (Fig. 2) may offer specific advantages over steel in being much more inert towards polyunsaturated fatty acids,

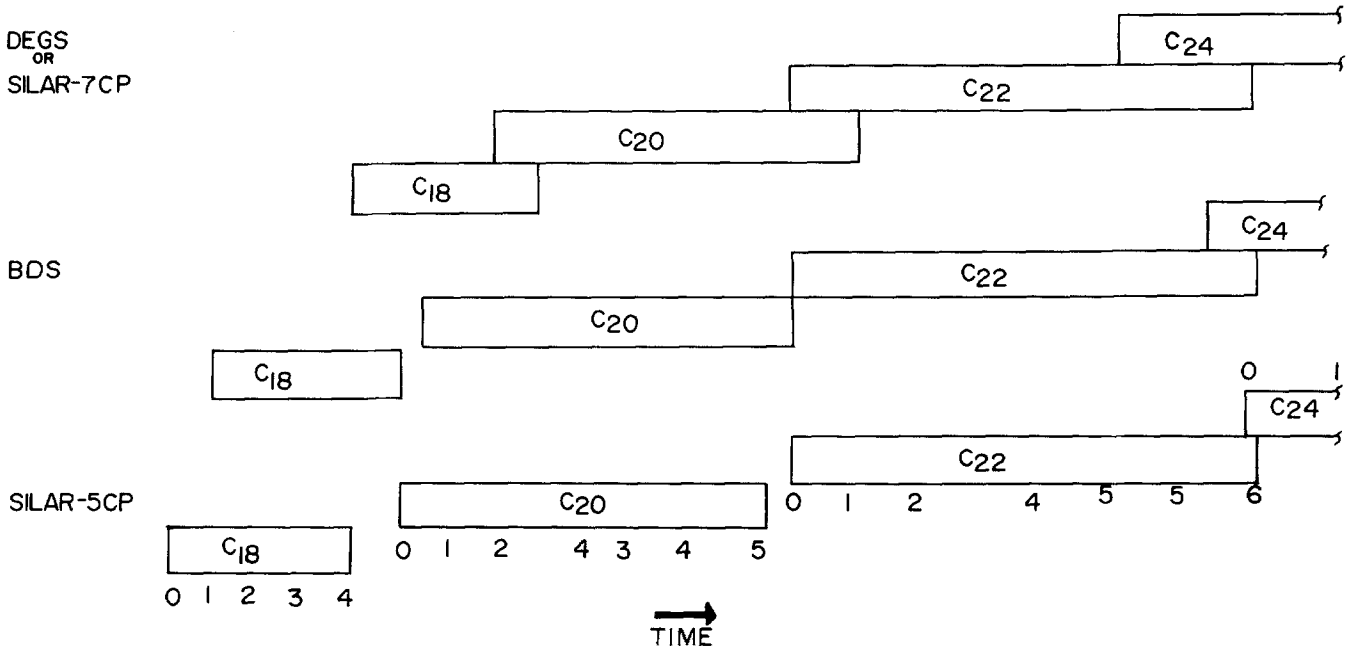


FIG. 1. Extent of overlap for even-carbon chain lengths of common fatty acids in gas chromatographic analyses on wall-coated stainless steel columns (15). The lower numbers refer to ethylenic bonds. The polarities are considerably greater with the same phases in packed columns. Reproduced from *Fette. Seifen. Anstrichmittel*, by permission.

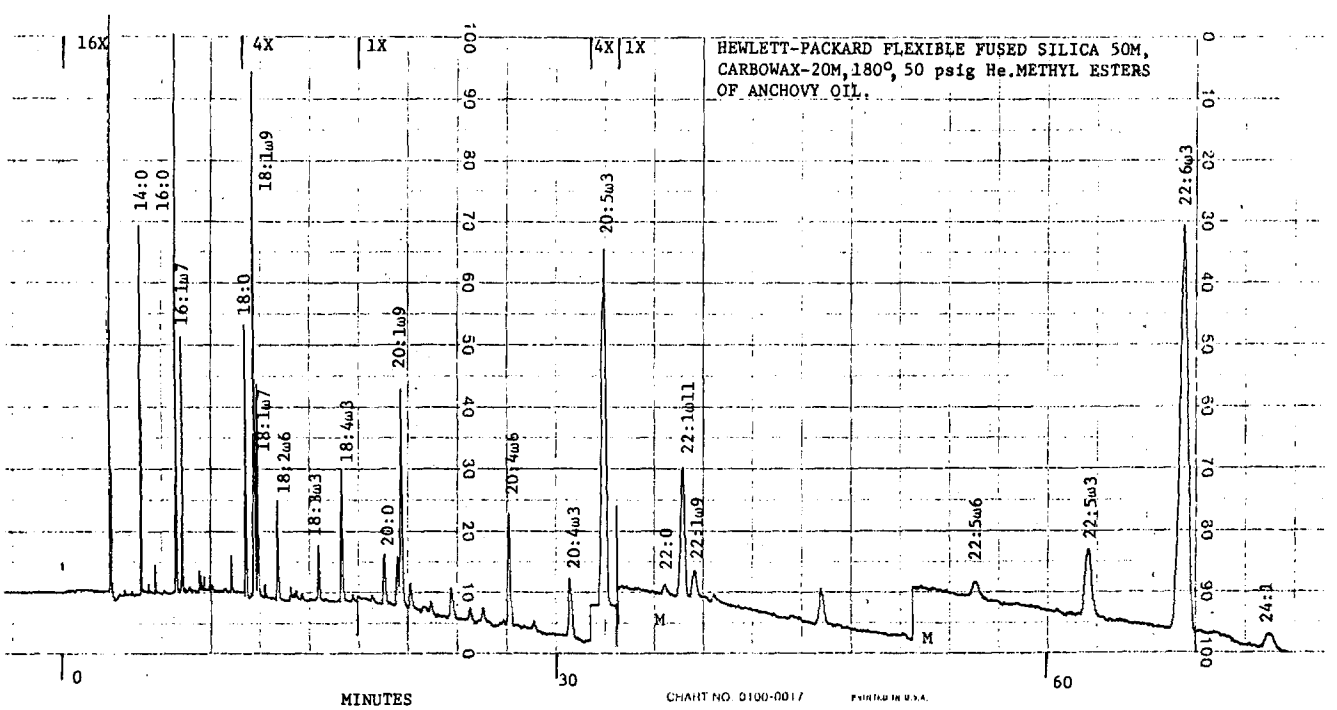


FIG. 2. Methyl esters of fatty acids of anchovy oil analyzed on a 50 m wall-coated open-tubular column of fused silica (Hewlett-Packard) with Carbowax-20 M as the liquid phase. Temperature, 180°; He pressure, 50 psig. Attenuation marked at top, and baseline adjustments (M) made for display purposes.

where losses are known to occur (15). Certainly, my personal experience indicates that the thermostable polyimide coating confers remarkable strength and safe-handling characteristics compared to glass open-tubular columns. The simplicity of installing these columns, their efficiency, and the excellent reproducibility (Table II) of Carbowax-20M as the liquid phase suggests that these columns could, in the future, become standard columns for analytical gas chromatography of methyl esters of fatty acids.

Is this type of ignorance an unavoidable result of the information explosion? Like many in this audience, I am also a victim of the latter problem. For example, I record all reprints on punch cards. As Figure 3 shows, the information explosion saturated my particular assimilation capabilities about 1973. How are young (or old) scientists going to publish their results without occasionally "reinventing the wheel" (21)? It is most important that the refereeing process be tightened up to avoid microprocessor or computer-based errors slipping into the literature. Once in hardcover book form, data acquires a veracity approaching sanctity, but these books have also undergone a radical change. In 1947, K.S. Markley could sum up almost the entire corpus of knowledge of fatty acids in one volume of 668 pages (2), while the contemporary volume by A.W. Ralston required 986 pages (3). By 1961, the second edition of Markley (22) had reached 3,865 pages in five volumes, and I expect that this heroic effort was in fact falling behind the then current rate of publication. At about the same time, T.P. Hilditch covered existing knowledge of the animal and vegetable sources of fats in only 664 pages (23). Currently you find most reference books, such as the *Handbook of Lipid Research* (1), put out on a multi-author basis. Unfortunately, even this process does not narrow down the sections enough to permit truly *critical* evaluation of published data.

If we look again at the tables in contemporary publications, one of the newer aspects is the plethora of statistical data, now readily generated by the aforementioned microchip data processor. I am quite prepared to accept that we need standard deviations, and comparison tests such as Student's T-test, but once again I question if some of the current scientists are not overcome with "precision," equating it with "accuracy", and misunderstand the concept of "validity." Precision and accuracy should be well understood and were defined by *Analytical Chemistry* (24) as:

> "*Accuracy* normally refers to the difference (error or bias) between the mean, $\bar{x}$, of the set of results and the value $\bar{x}$, which is *accepted* as the true or correct value for the quantity measured," and, "*Precision* relates to the reproducibility of measurements within a set."

TABLE II.

Reproducibility of ECL Values on Carbowax-20M Open-tubular Columns

Fatty acid	Fused silica[a] (Halifax 1980)	Stainless steel[b] (Flanzy 1976)
18:1ω9	18.18	18.20
18:2ω6	18.62	18.63
18:3ω3	19.23	19.25
20:4ω6	21.07	21.06
20:5ω3	21.68	21.67
22:6ω3	23.92	23.90

[a]R.G. Ackman, unpublished results.
[b]Ref. 20.

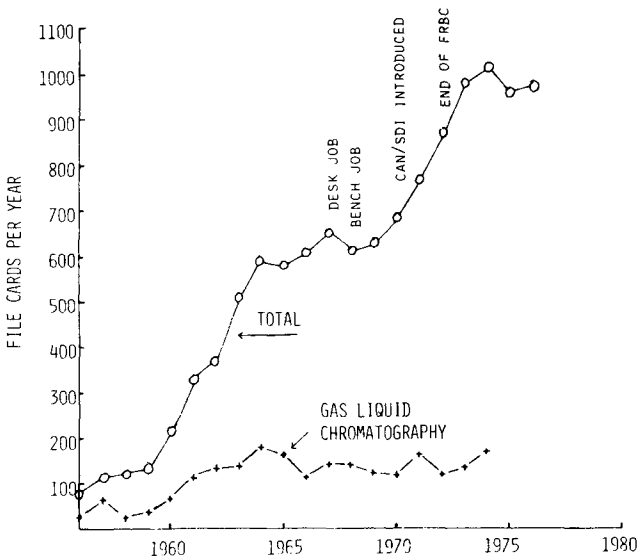


FIG. 3. Reprints (and later photocopies) filed per year by author. Note long-term effect of removal from bench for one year in 1967-68. CAN/SDI refers to computerized searching of *Chemical Abstracts*' tapes by National Science Library, Ottawa. FRBC refers to Fisheries Research Board of Canada, replaced in 1972 by Federal Department of Fisheries as management for Halifax Laboratory.

I must admit that the automation of analytical apparatus such as gas chromatography or atomic absorption spectrometers can produce very good precision. However, how this relates to accuracy is another matter. One could chose from innumerable published or unpublished sets of data, but a recent careful analysis of cod flesh lipids (25) is worthy of close study. This included an examination of the much-quoted Bligh and Dyer chloroform-methanol lipid extraction method (26), and the authors showed that considerable free fatty acid was being discarded in the aqueous-methanol phase. Modifying the method to retain the homophase $CHCl_3$-MeOH-H_2O increased lipid recoveries for cod muscle from 0.400 g to 0.494 g total lipid from 100 g of frozen fish. These authors have obviously increased *accuracy*. In their gas chromatography (Table III), the authors illustrate the often-ignored phenomenon that the *precision* of peak area measurements decreases with increasing retention times.

I should now like to pass on to "validity." This *concept* should include the relevance of the sample being analyzed to both the problem requiring the analyses and the sample, as well as the method of analysis.

Perhaps to promote "validity" every author submitting a paper on his method should be compelled to apply it to a natural sample from the bottom of a swamp as well as to pure standards. This would reinforce the old concept of "significant figures" which has been forgotten in the blizzard of digits displayed by the cheapest hand-held calculator.

Some of the published biochemical indexes for rat blood or organs are based on repetitive analyses of pooled samples, others on averages of individual analyses. Which is the more valid approach? Generally, the approach which can give the most reproducible results might be the more valid. In any event, it is desirable to include the statistical analysis as a basic part of the experimental design rather than as an afterthought.

The distribution of isomers of C_{22} monoethylenic

fatty acids in marine oils after partial hydrogenation was known (27,28) to be somewhat asymmetrical, that is, the dominant (~90%) 22:1Δ11 was accompanied by unequal proportions of the adjacent isomers 22:1Δ10 and 22:1Δ12. Several possible explanations for the asymmetry exist, such as defective analytical methodology, selectivity during hydrogenation, etc. In Figure 4, it is shown that a Danish crude fish oil had much more Δ13 isomer than Δ9, but that owing to the rigorous conditions of hydrogenation used in Europe, there was little obvious impact in the *cis* or *trans* 22:1 isomers in the product (28). In a Canadian herring oil, due to the milder conditions, the smaller excess of Δ13 over Δ9 can be followed into both the *cis* and *trans* products (29). If the objective of the derived information is for comparison of isomers in nutrition, then all studies mentioned (27-29) are equally valid, whereas if information on the hydrogenation process is required, only the complete analyses starting with crude oil are really "valid."

The use of a saturated fatty acid as an internal standard for the extraction of a polyunsaturated fatty acid may have been the only course open to analysts at one time, but the logical course of using a rather similar polyunsaturated fatty acid as the internal standard has been amply demonstrated in a recent "Note on Methodology" (30) which deserves to be put in a prominent place in every book on the analysis of lipids. Di-homo-γ-linolenic acid (8,11,14-eicosatrienoic) acid not only mimics arachidonic (5,8,11,-14-eicosatetraenoic) acid in physical properties such as solubility, or susceptibility to oxidation, but the two also fall conveniently close in gas chromatography of the methyl

TABLE III.

Repetitive Analysis by Capillary GLC of Three of the Fatty Acid Components of Cod Flesh Lipid

Run	1	2	3	4	5	6
18:0	14.5	14.5	14.6	14.7	14.7	14.5
20:5	32.9	31.0	31.8	30.9	32.6	31.9
22:6	103.5	99.8	97.6	97.2	98.5	108.1
Run	**7**	**8**	**9**	**10**	**χ**	**σ**
18:0	14.5	15.4	14.7	14.5	14.66	0.28
20:5	24.5	32.7	29.5	32.5	31.03	2.52
22:6	105.0	97.4	95.7	94.5	99.70	4.39

Area of peaks in mm^2 compared to a normalized standard of 100 mm^2. (Hardy et. al., 1979 [25]).

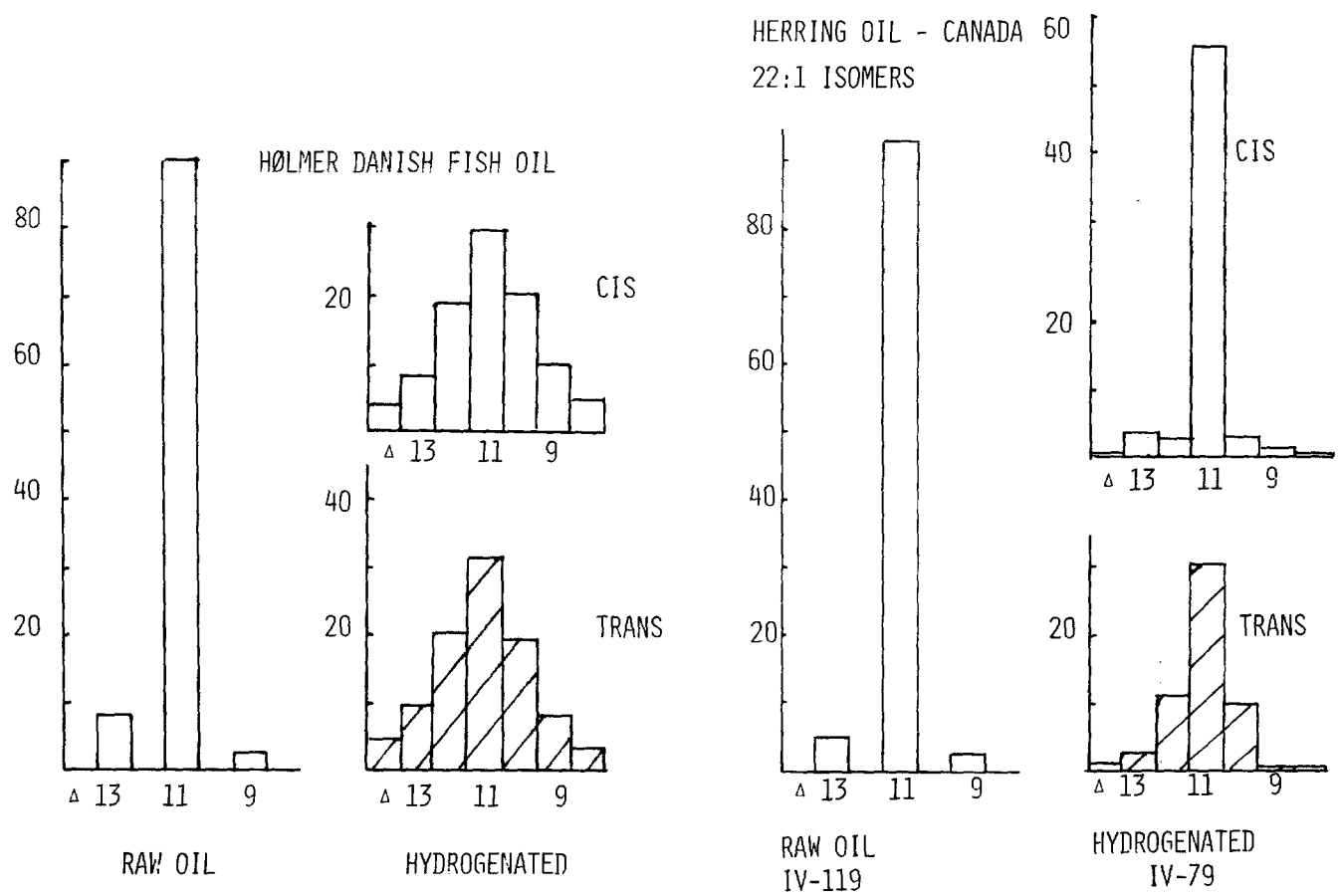


FIG. 4. Comparison of distribution of positional isomers of 22:1 fatty acids in samples of European (28) and Canadian (29) partially hydrogenated marine oils with same oils in unhydrogenated form.

esters. Thus, an esthetically satisfying modification to a method improves accuracy and precision, and at the same time contributes to the overall validity of the analysis.

It is opportune before leaving "validity" to return to the need for control over the sample. In many cases this is involuntary and limited, or poorly documented, and the lipid class extraction can be a problem area. In one case, the analyses of rat hearts, quite high proportions of free fatty acid had been found in lipids extracted by the conventional systems of chloroform-methanol. This was thought to be possibly important in evaluating the safety of dietary fats. Then a novel technique in which the tissue was first squashed between blocks of Dry Ice, and then extracted, demonstrated that the excessive free fatty acid was mostly an artifact derived from very rapid hydrolysis of lipid during extraction (31). The free fatty acid level was then less than when similar samples were extracted with chloroform-methanol at 0° and were more or less in proportion to the triglyceride present. The development of free fatty acid in tissue is not normally as fast, but surprisingly little has been done in this field (32). It is therefore worrisome to see many papers in which parts of animals or whole animals are held in frozen storage for extended periods before lipid class analysis.

If I may change the subject again to refer back to the title, one of the major areas where potential exists for improvement in lipid analyses is in reducing the scale of many operations. This usually has the effect of economizing on solvents and thus saving a great deal of money. Table IV gives the totals of assorted solvents for methods for cholesterol analyses. Two quite recent studies (33,34) require twenty-five times as much solvent as a simpler method (35) which must still be subjected to multi-laboratory evaluation. An excellent comparison of food extraction methodology by seven methods has been published (36) as part of the need for bringing in new methodology, and suggests the use of chloroform-methanol at 20 times sample weight for total lipid, principally to promote sterol recovery. Possibly the simpler sterol analysis (35) as a separate step would be a more economical approach. The replacement of inefficient multiple extractions with as few as one or two extractions with effective solvent systems (37) is a worthwhile objective which conserves solvents as well as time and effort.

The idea of executing a good deal of one's work in a screw-capped or glass-stoppered centrifuge tube appealed to me personally as long ago as 1967 (38). Since I have only intermittent freedom for research and cannot tie up valuable bench space I have found it convenient to use 5-ml centrifuge tubes for techniques as diverse as urea complexing (39) or ozonolyses (40,41). Although the sizes of the samples manipulated in these tubes are fully compatible with gas chromatography or plate thin layer chromatography, I should like to draw your attention to a further development of TLC, the combination of a silica-plus-glass frit coated on a quartz rod which can be passed through the flame of an ionization detector, the Iatroscan. This device, shown schematically in Figure 5, has been fully described elsewhere (42). A typical separation is shown in Figure 6, but application to lipids on this continent is just beginning (43,44). The sensitivity is at least two orders of magnitude better than ordinary plate TLC and the opportunities for burning off the more mobile materials and redeveloping offer a unique tool for lipid research (44). Tanks are smaller, solvent loss is minimal, and each rod can be used many times with consequent improvement in reproducibility. The question of reducing solvent loss in any of the techniques mentioned above is not only an important economic aspect of lipid research today, but generally contributes to healthier working conditions. It is regrettable that the international standard methods which are occupying the time and energy of many of us often appear to have changed little in the last fifty years.

The subject of publication has always been of interest to me, and as Dr. Kaufmann was for many years editor of the much-respected *Fette. Seifen. Anstrichmittel,* it is fitting that I close the Kaufmann memorial lecture with some remarks on potential improvements in publication of lipid material. On a lighter note I suggest that no titles be permitted to start with either "A simple apparatus . . ." or "A simple method . . ." My own contribution to this genre (45) was in fact a gross misuse of the word. Again, I feel sorry for foreign language readers and translators when I run across farcical terms in the literature. Some, such as "pussycat printers" and "daisy wheels," are furtunately rare in the lipid literature. One is still permitted to use trivial names such as palmitic and stearic since these

TABLE IV.

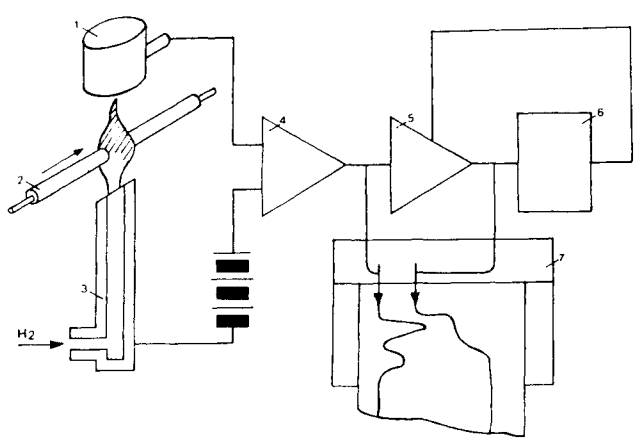
Solvent Volumes for Sterol Determinations

	Punwar 1975[a]	Sheppard et al., 1977[b]	Kovacs et al., 1979[c]
MEOH	100	100	–
ETOH	100	2	4
$CHCL_3$	235	200	–
C_6H_6	100	–	20
Pet E.	120	140	–
Diet E.	–	∿50	–
Total (ml)	655	492	24

[a]For cholesterol only (33).

[b]Combined sterol/fatty acid procedure (34).

[c]For sterols only (35).



1. Collector Electrode
2. Chromarod
3. Burner
4. Current Amplifier
5. Integral Amplifier
6. Integral Reset Circuit
7. Recorder

FIG. 5. Schematic of the organization of Iatroscan Chromarod/FID apparatus. The hydrogen flame is static, and the rods are automatically indexed in position by a rack system and moved in sequence through the flame. Courtesy of Newman-Howells Associates, Wolvesey Palace, Winchester, Hampshire S023 9NB, England.

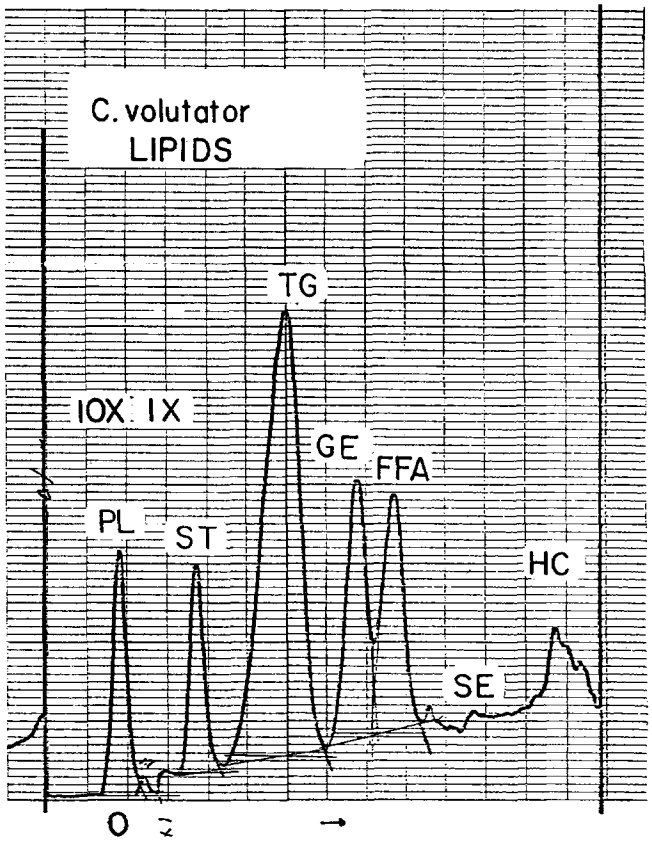


FIG. 6. Recording of Chromarod separation of lipids of *Corophium volutator* (a small burrowing marine amphipod). Double development with first solvent of petroleum ether/benzene/formic acid in volume proportions 80:20:1 and second solvent of petroleum ether/diethyl ester/formic acid in volume proportions at 96:4:1. Note attenuations (X). Abbreviations are: PL=phospholipids, ST=sterols, TG=triglycerides, GE=glyceryl ethers, FFA=free fatty acids, HC=hydrocarbons, SE=sterol esters, O=origin. Arrow shows direction of development. Reproduced from *Proc. N.S. Inst. Sci.*, by permission.

are unambiguous, but I should like to make a special plea for accuracy in referring to methyl octadecenoate and similar materials where isomeric forms may be present. If an author is using packed column gas chromatography it is invidious to expect a reader to accept a chromatographic peak as "methyl oleate" when substantial proportions of a *cis*-11-octadecenoic (*cis*-vaccenic) acid have long been known (46) to accompany the better-known *cis*-9-octadecenoic (oleic) acid. In view of alternative pathways to these two octadecenoic isomers:

$$\left.\begin{array}{l} 16{:}0 \rightarrow 18{:}0 \rightarrow 18{:}1\omega9 \\ \quad\downarrow \\ 16{:}1\omega7 \rightarrow 18{:}1\omega7 \end{array}\right\} \begin{array}{l} \textit{cis}\text{-9-Octadecenoic} \\ \text{Methyl octadecenoate} \\ \textit{cis}\text{-11-Octadecenoic,} \end{array}$$

it is particularly galling to see space wasted in lipid and biochemical journals on involved arguments as to how radioactive labeling can appear in the "oleate" product, when it is really in the *cis*-11-ocatdecenoic acid product. Of rather less concern is the fact that this fatty acid can be chain-elongated in parallel with the "$\omega9$" or "$\omega6$" acids, so either the *cis*-11-octadecenoic ($18{:}1\omega7$) or its precursor *cis*-9-hexadecenoic (palmitoleic or $16{:}1\omega7$) acid can give rise to families of C_{20} and C_{22} unsaturated fatty acid with "$\omega7$" structures (47). You will notice that I have freely used a shorthand fatty acid notation mixed into the Geneva nomenclature and I feel that it is up to the author to choose the most appropriate system from among $\omega\eta$, η-x, or Δy to clarify his context.

My one and only attempt to publish in *Lipids* using the officially sanctioned and recommended fatty acid nomenclature (48), previously published in *Lipids*, was rudely commented on in the refereeing process. I think that this attitude to regimentation augers well for lipid chemists realizing their potential for improving both lipid analyses and quality of publications. I am sure that H.P. Kaufmann would have been delighted to see the variety of good work currently appearing smoothly and rapidly in *Fette. Seifen. Anstrichmittel, Lipids, Journal of the American Oil Chemists' Society, Chemistry and Physics of Lipids, Journal of Lipid Research, Revue Francaise Corps Gras, Rivista Italiana Sostanze Grasse and Yuhagaku*, to mention only a few outlets familiar to most present.

REFERENCES

1. "Fatty Acids and Glycerides," Handbook of Lipid Research, edited by A. Kuksis, Vol. 1, Plenum Press, New York, 1978.
2. Markley, K.S., "Fatty Acids, Their Chemistry and Physical Properties," Interscience, New York, 1947.
3. Ralston, A.W., "Fatty Acids and their Derivatives," Chapman and Hall, London, 1948.
4. Orr, C.H., and J.E. Callen, J. Am. Chem. Soc. 80:249 (1958).
5. Ackman, R.G., in "Methods in Enzymology," Vol. XIV:329, edited by J.M. Lowenstein, Academic Press, New York, 1969.
6. Chisholm, M.J., and C.Y. Hopkins, Can. J. Chem. 31:1131 (1953).
7. Ackman, R.G., M.E. Retson, L.R. Gallay and F.A. Vandenheuvel, Can. J. Chem. 39:1956 (1961).
8. Golay, M.J.E., in "Gas Chromatography," edited by V.J. Coates, H.J. Noebels and I.S. Fagerson, Academic Press, New York, 1958.
9. Lovelock, J.E., Anal. Chem. 33:162 (1961).
10. Ettre, L.D., "Open Tubular Columns in Gas Chromatography," Plenum Press, New York, 1965.
11. Ackman, R.G., JAOCS 43:483 (1966).
12. Stahl, E., "Thin-layer Chromatography, A Laboratory Handbook", 2nd Edition, Allen and Unwin, London, 1969.
13. Rothstein, M., and P. Gotz, Arch. Biochem. Biophys. 126:131 (1968).
14. Anon., Gas-Chrom. Newsletter. Applied Science Laboratories, 19(4), 4 (1978).
15. Ackman, R.G., and C.A. Eaton, Fette Seifen Anstrichm. 80:21 (1978).
16. Slover, H.T., and E. Lanza, JAOCS 56:933 (1979).
17. Ackman, R.G., Ibid. 40:564 (1963).
18. Ackman, R.G., J. Gas Chromatogr. 1(4):11 (1963).
19. Jamieson, G.R., J. Chromatogr. Sci. 13:491 (1975).
20. Flanzy, J., M. Boudon, C. Leger and J. Pihet, Ibid. 14:17 (1976).
21. Ettre, L.S., and J.E. Purcell, Ibid. 17:584 (1979).
22. Markley, K.S., "Fatty Acids, Their Chemistry, Properties-Production and Uses," 2nd Edition, in 5 parts, John Wiley and Sons, New York, 1968.
23. Hilditch, T.P., "The Chemical Composition of Natural Fats," 3rd Edition, John Wiley and Sons, New York, 1956.
24. Anon., "Instructions to Authors," Anal. Chem. 50:191 (1978).
25. Hardy, R., A.S. McGill and F.D. Gunstone, J. Sci. Food Agric. 30:999 (1979).
26. Bligh, E.G., and W.J. Dyer. Can. J. Biochem. Physiol. 37:911 (1959).
27. Conacher, H.B.S., B.D. Page and R.K. Chadra, JAOCS 49:520 (1972).
28. Hølmer, G., in "Sammenlignende untersogelser af uhaerdede og haerdede danske fiskeolier, specielt med henblik a C_{20}-og C_{22}-monoeninholdet," Tech. Univ. Denmark, Lyngby 26p. and appendices, 1976.
29. Sebedio, J-L., M.F. Langman, C.A. Eaton and R.G. Ackman, JAOCS (submitted for publication).
30. Gerber, J.G., J.S. Barnes and A.S. Nies, J. Lipid Res. 20:912 (1979).
31. Kramer, J.K.G., and H.W. Hulan, Ibid. 19:103 (1978).
32. Fairbairn, D., J. Biol. Chem. 157:645 (1945).
33. Punwar, J.K., J. Assoc. Off. Anal. Chem. 58:804 (1975).

34. Sheppard, A.J., D.R. Newkirk, W.D. Hubbard and T. Osgood, Ibid. 60:1302 (1977).
35. Kovacs, M.I.P., W.E. Anderson and R.G. Ackman, J. Food Sci. 44:1299 (1979).
36. Hubbard, W.D., A.J. Sheppard, D.R. Newkirk, A.R. Prosser and T. Osgood, JAOCS 54:81 (1977).
37. Dutta, J., A. Biswas, S. Saha and C. Deb., J. Chromatogr. 124:29 (1976).
38. Castell, J.D., and R.G. Ackman, Can. J. Chem. 45:1405 (1967).
39. Ackman, R.G., and S.N. Hooper, Comp. Biochem. Physiol. 24:549 (1968).
40. Ackman, R.G., Lipids 12:293 (1977).
41. Sebedio, J-L., and R.G. Ackman, Can. J. Chem. 56:2480 (1978).
42. Ackman, R.G., Methods in Enzymology, Academic Press, New York (in press).
43. Sipos, J.C., and R.G. Ackman, J. Chromatogr. Sci. 16:443 (1978).
44. Ackman, R.G., and A.D. Woyewoda, Ibid. 17:514 (1979).
45. Ackman, R.G., M.A. Bannerman and F.A. Vandenheuvel, J. Chromatogr. 8:44 (1962).
46. Brockerhoff, H., and R.G. Ackman, J. Lipid Res. 8:661 (1967).
47. Pullerkat, R.K., and H. Reha, J. Chromatogr. Sci. 14:25 (1976).
48. Anon., Lipids 12:455 (1977).

People

Kreutzer joins EMI

George M. Kreutzer has been named to the newly created post of marketing representative for EMI Corporation of Des Plaines, Illinois, to help introduce new processes developed by EMI, to help adapt existing designs to new applications and to assist companies using EMI processes to develop new markets and new products.

Kreutzer previously was a group vice-president for PVO International. At EMI, he will work with domestic firms on development of foreign production facilities and to provide technical assistance to public and private interests in developing nations. □

Acidchem hires Gallagher

James V. Gallagher, formerly with Akzo in Littleborough, England, has been named operations manager for the new Acidchem (M) Sdn Bhd fatty acid plant in Prai, Penang, Malaysia. The firm previously has announced appointment of Roy de Vries as general manager.

The firm, part of the Palmco Group, plans to market throughout the world fatty acids made from palm, palm kernel and coconut oils. □

Hammond receives award

Earl G. Hammond, professor of biochemistry at Iowa State University, has been named recipient of the 1980 Pfizer Award in Cheese and Cultured Products research. Dr. Hammond received the award during the American Dairy Science Association's annual meeting earlier this year. □

Appointments

Firmenich Inc. has announced the appointment of **Adolph Leon** as financial director of the Western Hemisphere and **Thomas R. Hummer** as supervisor of the fragrance section of the quality control department. ... **R. Douglas Varvil** has been appointed national technical representative and **Frederick W. Seeholzer** has been appointed bulk sales manager—eastern region at Durkee Industrial Foods Group/SCM Corporation. ... **Rita Batens** has been named public relations manager for the American Soybean Association office in Brussels, Belgium. ... The American Society for Testing and Materials has changed **William T. Cavanaugh's** title to president from chief executive officer. □

Deaths

B.B. O'Malley

AOCS has been informed of the December 1979 death of Benedict B. O'Malley. Dr. O'Malley was a professor of biochemistry and pharmacology at Jersey City State College in Jersey City, New Jersey. He had received his doctorate in biochemistry from Fordham University in 1950. □

Martin A. Beck

Martin A. Beck, a member of the AOCS since 1932, died Sept. 14, 1980, in Louisville, Kentucky. He was 88.

Mr. Beck had retired in 1957 after 40 years service with what was then The Glidden Co. and its predecessor organizations in Louisville. When he joined AOCS in 1932, Mr. Beck was superintendent of the soap plant for